



**INSTITUTO LATINO-AMERICANO DE
TECNOLOGIA, INFRAESTRUTURA E
TERRITÓRIO**

**PROGRAMA DE PÓS-GRADUAÇÃO
INTERDISCIPLINAR EM ENERGIA E
SUSTENTABILIDADE**

**MINERALIZAÇÃO DE CO₂ EM PÓS RECICLADOS DE RESÍDUOS DE
CONSTRUÇÃO E DEMOLIÇÃO:
USO DE CARBONO E EMPREGO COMO MATERIAL CIMENTÍCIO SUPLEMENTAR**

KATHLEEN DALL BELLO DE SOUZA RISSON

Foz do Iguaçu
2025



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Tese de doutorado apresentado ao Programa de Pós-Graduação Interdisciplinar em Energia e Sustentabilidade do Instituto Latino-Americano de Tecnologia, Infraestrutura e Território da Universidade Federal da Integração Latino-Americana, como requisito para a obtenção do título de Doutora em Energia e Sustentabilidade.

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Orientadora: Prof^ª. Dr^ª. Edna Possan

Coorientadora: Prof^ª. Dr^ª. Kátya Regina de Freitas Zara

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*O sucesso nasce do querer, da
determinação e persistência em se
chegar a um objetivo. Mesmo não
atingindo o alvo, quem busca e vence
obstáculos, no mínimo fará coisas
admiráveis.*

José de Alencar

RISSON, Kathleen Dall Bello de Souza. **Mineralização de CO₂ em pós reciclados de resíduos de construção e demolição: uso de carbono e emprego como material cimentício suplementar**. 2025. 208f. Tese (Doutorado em Energia e Sustentabilidade) – Universidade Federal da Integração Latino-Americana, Foz do Iguaçu, PR.

RESUMO

Este trabalho aplicou a mineralização por carbonatação acelerada (gás-sólido) em pós reciclados de resíduos da construção e demolição (RCD), com o objetivo de fixar CO₂ e utilizá-los como materiais cimentícios suplementares (MCS) na fabricação de cimento. Para isso, foi desenvolvido um conjunto de cinco estudos experimentais envolvendo as principais variáveis que afetam esse processo. Inicialmente, estudou-se a moagem das partículas de RCD, avaliando as características físicas após a cominuição. O processamento por 30 minutos com 0,5% de aditivo propilenoglicol reduziu o d₅₀ e aumentou a área superficial BET, sendo adotado como padrão por demandar menor tempo de moagem e teor de aditivo, e produzir partículas com características físicas similares às do cimento Portland (CP). Na sequência, foi conduzida a avaliação de procedimentos para determinação da absorção de água dos pós, comparando-se a análise termogravimétrica (TGA) com outros métodos da literatura. A TGA apresentou potencial de aplicação, com menor variabilidade nos resultados e menor interferência do operador. No entanto, o tempo de saturação deve ser inferior a 24 h para minimizar os efeitos da pré-hidratação das partículas residuais anidras do cimento. O terceiro estudo explorou os efeitos de diferentes origens de pós de RCD (mista e concreto), com três faixas granulométricas, submetidas a diferentes tempos de exposição (2 h, 8 h, 24 h, 48 h e 168 h) ao CO₂ (15% a 23 ± 2 °C), avaliando-se o potencial de uso como MCS em argamassas com substituição de 25% do CP. Constatou-se que, por meio da mineralização, foi possível fixar entre 0,8 e 29,51 kg.CO₂/t, com maior eficácia em pós reciclados de concreto (RCP). No quarto experimento, foram avaliados apenas os RCP, analisando-se a fixação de carbono e o desempenho como MCS. Utilizando CO₂ a 15% de concentração, foram testados teores de umidade entre 0% e 24% e temperaturas de ensaio de 20, 40 e 60 °C, com tempo fixo de exposição de 0,5 h. Para pós com 12% de umidade e temperatura de 40 °C, avaliou-se a fixação de carbono nos tempos de 1 h, 1,5 h, 2 h e 24 h. O arranjo com 12% de umidade e temperaturas de 40 °C e 60 °C por 0,5 h de exposição ao CO₂ permitiu substituir 25% do cimento, reduzindo o índice de carbono (kg CO₂/m³/MPa) em 9% e 8%, respectivamente, quando comparado ao RCP não mineralizado. Por fim, no último estudo, avaliou-se o desempenho de pós reciclados de concreto de múltiplas fontes, observando-se a influência na fixação de carbono e no desempenho com substituições de 10%, 25% e 40% do CP. Os pós com 12% de umidade foram submetidos à exposição de 15 a 120 minutos a 15% de CO₂ e 60 °C. Constatou-se a fixação de CO₂ entre 7 e 25 kg.CO₂/t, valores influenciados pela granulometria, pela origem dos pós e pelo tempo de exposição ao CO₂, sendo observada a maior fixação de CO₂ nos pós produzidos com CP V e maiores porosidades (maior relação água/cimento). De modo geral, constatou-se que o potencial de fixação de CO₂ em pós reciclados de concreto é superior aos de origem mista, com maior fixação em partículas menores e mais porosas. O processo de mineralização é fortemente influenciado pelas variáveis de contorno: umidade, temperatura e tempo de exposição ao CO₂. A fixação de CO₂ em RCP representa uma estratégia que promove a economia circular, podendo ser considerada em negociações de créditos de carbono.

Palavras-chave: Carbonatação gás-sólido; Resíduos da Construção Civil; Economia Circular; Captura, utilização e armazenamento de carbono (CCUS).

RISSON, Kathleen Dall Bello de Souza. Mineralización de CO₂ en polvos reciclados de residuos de construcción y demolición: uso de carbono y aplicación como material cementante suplementario. 2025. 208f. Tesis (Doctorado en Energía y Sostenibilidad) – Universidad Federal de la Integración Latinoamericana, Foz do Iguaçu, PR.

RESUMEN

Este trabajo aplicó la mineralización por carbonatación acelerada (gas-sólido) en polvos reciclados de residuos de construcción y demolición (RCD), con el objetivo de fijar CO₂ y utilizarlos como materiales cementicios suplementarios (MCS) en la fabricación de cemento. Para ello, se desarrolló un conjunto de cinco estudios experimentales que involucraron las principales variables que afectan este proceso. Inicialmente, se estudió la molienda de las partículas de RCD, evaluando las características físicas después de la conminución. El procesamiento durante 30 minutos con 0,5% de aditivo propilenglicol redujo el d₅₀ y aumentó el área superficial BET, siendo adoptado como estándar por requerir menor tiempo de molienda y contenido de aditivo, y producir partículas con características físicas similares a las del cemento Portland (CP). A continuación, se llevó a cabo la evaluación de procedimientos para la determinación de la absorción de agua de los polvos, comparando el análisis termogravimétrico (TGA) con otros métodos de la literatura. El TGA presentó potencial de aplicación, con menor variabilidad en los resultados y menor interferencia del operador. Sin embargo, el tiempo de saturación debe ser inferior a 24 h para minimizar los efectos de la prehidratación de las partículas residuales anhidras del cemento. El tercer estudio exploró los efectos de diferentes orígenes de polvos de RCD (mixto y concreto), con tres rangos granulométricos, sometidos a diferentes tiempos de exposición (2 h, 8 h, 24 h, 48 h y 168 h) al CO₂ (15% a 23 ± 2 °C), evaluando el potencial de uso como MCS en morteros con sustitución del 25% del CP. Se constató que, mediante la mineralización, fue posible fijar entre 0,8 y 29,51 kg.CO₂/t, con mayor eficacia en polvos reciclados de concreto (PRC). En el cuarto experimento, se evaluaron solo los PRC, analizando la fijación de carbono y el desempeño como MCS. Utilizando CO₂ al 15% de concentración, se probaron contenidos de humedad entre 0% y 24% y temperaturas de ensayo de 20, 40 y 60 °C, con un tiempo fijo de exposición de 0,5 h. Para polvos con 12% de humedad y temperatura de 40 °C, se evaluó la fijación de carbono en los tiempos de 1 h, 1,5 h, 2 h y 24 h. El arreglo con 12% de humedad y temperaturas de 40 °C y 60 °C por 0,5 h de exposición al CO₂ permitió sustituir el 25% del cemento, reduciendo el índice de carbono (kg CO₂/m³/MPa) en 9% y 8%, respectivamente, en comparación con el PRC no mineralizado. Finalmente, en el último estudio, se evaluó el desempeño de polvos reciclados de concreto de múltiples fuentes, observándose la influencia en la fijación de carbono y en el desempeño con sustituciones del 10%, 25% y 40% del CP. Los polvos con 12% de humedad fueron sometidos a exposición de 15 a 120 minutos a 15% de CO₂ y 60 °C. Se constató la fijación de CO₂ entre 7 y 25 kg.CO₂/t, valores influenciados por la granulometría, el origen de los polvos y el tiempo de exposición al CO₂, observándose la mayor fijación de CO₂ en los polvos producidos con CP V y mayores porosidades (mayor relación agua/cemento). En general, se constató que el potencial de fijación de CO₂ en polvos reciclados de concreto es superior al de origen mixto, con mayor fijación en partículas más pequeñas y porosas. El proceso de mineralización está fuertemente influenciado por las variables de contorno: humedad, temperatura y tiempo de exposición al CO₂. La fijación de CO₂ en PRC representa una estrategia que promueve la economía circular, pudiendo ser considerada en negociaciones de créditos de carbono.

Palabras clave: Carbonatación gas-sólido; Residuos de la construcción civil; Economía circular; Captura, utilización y almacenamiento de carbono (CCUS).

RISSON, Kathleen Dall Bello de Souza. **CO₂ mineralization in recycled construction and demolition waste powders: carbon utilization and use as a supplementary cementitious material**. 2025. 208 f. Doctoral thesis (PhD in Energy and Sustainability) – Federal University of Latin American Integration, Foz do Iguaçu, PR.

ABSTRACT

This work applied mineralization by accelerated carbonation (gas-solid) in recycled powders from construction and demolition waste (CDW), with the objective of fixing CO₂ and using them as supplementary cementitious materials (SCM) in cement manufacturing. For this, a set of five experimental studies was developed involving the main variables that affect this process. Initially, the grinding of CDW particles was studied, evaluating the physical characteristics after comminution. Processing for 30 minutes with 0.5% of propylene glycol additive reduced d₅₀ and increased the BET surface area, being adopted as a standard for requiring less grinding time and additive content, and producing particles with physical characteristics similar to Portland cement (PC). Next, the evaluation of procedures for determining the water absorption of the powders was conducted, comparing the thermogravimetric analysis (TGA) with other methods from the literature. The TGA showed potential for application, with less variability in results and less operator interference. However, the saturation time should be less than 24 h to minimize the effects of pre-hydration of the residual anhydrous cement particles. The third study explored the effects of different origins of CDW powders (mixed and concrete), with three particle size ranges, subjected to different exposure times (2 h, 8 h, 24 h, 48 h and 168 h) to CO₂ (15% at 23 ± 2 °C), evaluating the potential use as SCM in mortars with 25% replacement of PC. It was found that, through mineralization, it was possible to fix between 0.8 and 29.51 kg.CO₂/t, with greater efficiency in recycled concrete powders (RCP). In the fourth experiment, only RCP were evaluated, analyzing carbon fixation and performance as SCM. Using CO₂ at 15% concentration, moisture contents between 0% and 24% and test temperatures of 20, 40 and 60 °C were tested, with a fixed exposure time of 0.5 h. For powders with 12% moisture and temperature of 40 °C, carbon fixation was evaluated at times of 1 h, 1.5 h, 2 h and 24 h. The arrangement with 12% moisture and temperatures of 40 °C and 60 °C for 0.5 h exposure to CO₂ allowed replacing 25% of the cement, reducing the carbon index (kg CO₂/m³/MPa) by 9% and 8%, respectively, when compared to non-mineralized RCP. Finally, in the last study, the performance of recycled concrete powders from multiple sources was evaluated, observing the influence on carbon fixation and performance with replacements of 10%, 25% and 40% of PC. The powders with 12% moisture were subjected to exposure from 15 to 120 minutes at 15% CO₂ and 60 °C. CO₂ fixation between 7 and 25 kg.CO₂/t was found, values influenced by particle size, origin of the powders and exposure time to CO₂, with greater CO₂ fixation observed in powders produced with CP V and higher porosities (higher water/cement ratio). In general, it was found that the CO₂ fixation potential in recycled concrete powders is higher than those of mixed origin, with greater fixation in smaller and more porous particles. The mineralization process is strongly influenced by boundary variables: moisture, temperature and CO₂ exposure time. CO₂ fixation in RCP represents a strategy that promotes the circular economy and can be considered in carbon credit negotiations.

Keywords: Gas-solid carbonation; Construction waste; Circular economy; Carbon capture, utilization and storage (CCUS).

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LISTA DE ABREVIATURA E SIGLAS

ABNT	Associação Brasileira de Normas Técnicas
ACV	Avaliação do Ciclo de Vida
a/c	Água/Cimento
a/s	Água/Sólido
AFt	Alumina, óxido férrico, tri-sulfato/ Etringita
AFm	Alumina, óxido férrico, mono-sulfato/ Monossulfoaluminato
Al	Alumínio
Al ₂ O ₃	Óxido de alumínio
ARCI	Agregado Reciclado Cimentício
ARCO	Agregado Reciclado de Concreto
ARM	Agregado Reciclado Misto
ASTM	American Society for Testing and Materials/ Sociedade Americana de Testes e Materiais
BECCS	Bioenergy with Carbon Capture and Storage/ Bioenergia com Captura e Armazenamento de Carbono
BET	Brunauer, Emmett, Teller
BI	Binder Index/ Índice de ligante
Ca	Cálcio
Ca (OH) ₂	Hidróxido de cálcio/ Portlandita
Ca/Si	Cálcio/Silício
CaCO ₃	Carbonato de cálcio
CaO	Óxido de cálcio
CCS	Carbon Capture and Storage/ Captura e Armazenamento de Carbono
CCUS	Carbon Capture, Utilization and Storage/ Captura, Utilização e Armazenamento de Carbono
CH ₄	Metano
CI	Carbon Index/ Índice de carbono
CLC	Chemical looping Combustion/ Combustão em Looping Químico
CO ₂	Dióxido de carbono
CP	Cimento Portland
CPI	Cimento Portland comum

CP II F	Cimento Portland composto com filer
CP IV	Cimento Portland pozolânico
CP V	Cimento Portland de alta resistência inicial
C-S-H	Silicato de cálcio hidratado
C ₂ S	Silicato dicálcico/ Alita
C ₃ S	Silicato tricálcico/ Belita
d ₁₀	Diâmetro pelo qual passam 10% das partículas
d ₅₀	Diâmetro pelo qual passam 50% das partículas
d ₉₀	Diâmetro pelo qual passam 90% das partículas
DAC	Direct Air Capture/ Captura Direta de Ar
DRX	Difração de raios-X
DTG	Termogravimetria derivada
EC	Economia circular
EDS	Energy Dispersive Spectroscopy/ Espectroscopia de Dispersão de Energia
FA	Fly Ash/ Cinza volante
f _c	Resistência à compressão
FTIR	Espectroscopia de Infravermelho por Transformada de Fourier
GEE	Gases de Efeito Estufa
G/S	Gás-Sólido
GS	Ground granulated blast furnace slag/ Escória de alto-forno granulada moída
H ₂	Hidrogênio
H ₂ CO ₃	Ácido carbônico
H ₂ O	Água
IPCC	Intergovernmental Panel on Climate Change/ Painel Intergovernamental das Alterações Climáticas
L/S	Líquido-Sólido
LC ³	Limestone Calcined Clay Cement/ Cimento de argila calcinada
MC	Mechanochemical Carbonation/ Carbonatação mecanoquímica
MCS	Materiais Cimentícios Suplementares
MEV	Microscopia Eletrônica de Varredura
Mg	Magnésio
MgO	Óxido de Magnésio

NaCl	Cloreto de sódio
NaHCO ₃	Bicarbonato de sódio
NaOH	Hidróxido de sódio
NBR	Norma Brasileira Regulamentadora
N ₂ O	Óxido nitroso
NH ₄ Cl	Cloreto de amônio
pH	Potencial Hidrogeniônico
RCC	Resíduos da Construção Civil
RCD	Resíduo de Construção e Demolição
RCF	Recycled Concrete Fines/ Finos de concreto reciclado
RCP	Recycled Concrete Powder/ Pó de concreto reciclado
SF	Silica Fume/ Sílica Ativa
Si	Silício
SiO ₂	Dióxido de silício/ Sílica
T	Temperatura
TG	Termogravimetria
TGA	Análise termogravimétrica
UR	Umidade Relativa do ambiente

LISTA DE SÍMBOLOS E UNIDADES

~	Aproximadamente
Ø	Diâmetro
Δ	Delta
°C	Graus Celsius
>	Maior que
±	Mais ou menos
<	Menor que
µm	Micrômetro
Ω	Ômega/ Ohm
%	Porcentagem
atm	Unidade de pressão atmosférica
bar	Bar
cm ³	Centímetro cúbico
CO ₂ -eq	Carbon dioxide equivalent/ Equivalente de dióxido de carbono
g	Gramas
G	Giga
h	Horas
kg	Quilograma
kW	Quilowatt
L	Litro
M	Mega
m ²	Metro quadrado
m ³	Metro cúbico
mm	Milímetro
min	Minuto
mol	mol
Nº	Número
Pa	Pascal
ppm	Partes por milhão
rpm	Rotações por minuto

t	Tonelada
US\$	United States dólar/ Dólar dos Estados Unidos

GLOSSÁRIO

Alcalinidade: refere-se à capacidade da água de resistir à acidificação. Tal é conhecido como o efeito de tampão da água ou a capacidade da água de resistir a uma alteração no pH quando é adicionado um ácido.

Absorção: processo pelo qual uma substância, como um sólido ou líquido, absorve outra substância, como um líquido ou gás, através de minúsculos poros ou espaços entre suas moléculas.

Adsorção: processo pelo qual um material atrai moléculas, como dióxido de carbono, para sua superfície para que ele possa ser capturado e/ ou armazenado.

Armazenamento de carbono (CO₂): estocagem do CO₂ por meios permanentes e de longo prazo, geralmente em locais geológicos, para impedir o retorno à atmosfera.

Captura de carbono (CO₂): remoção do dióxido de carbono (CO₂) resultante de operações industriais, usinas de energia e outras fontes.

Carbonatação de materiais à base de cimento: trata-se de uma reação química em que o dióxido de carbono (CO₂) penetra no material cimentício e reage com os compostos alcalinos presentes, principalmente o hidróxido de cálcio (Ca(OH)₂) e os silicatos de cálcio hidratados (C-S-H). Como resultado, formam-se carbonato de cálcio (CaCO₃) e outros carbonatos, alterando a microestrutura e a alcalinidade do sistema.

Circularidade: conceito econômico de reutilizar ou regenerar produtos e recursos em toda a cadeia de valor para reduzir o desperdício.

Crédito de Carbono: títulos com valor monetário atrelados a uma certa quantidade de Gases de Efeito Estufa (GEE) não emitida (redução) ou removida (remoção) para/da atmosfera, podendo ser comercializados no Mercado de Carbono.

Fixação de carbono (CO₂): imobilização de CO₂ por sua reação com outro material para produzir um composto estável.

Fração fina de RCD: frações finas de resíduos de construção e demolição triturados como alternativas à areia natural (0,15 mm < Ø < 4,75 mm).

Mercado de carbono: criado com o objetivo de incentivar a descarbonização. Empresas que não conseguem reduzir as emissões do seu sistema produtivo, podem comprar créditos de carbono de projetos que promovam a redução de emissões ou a remoção de gases de efeito estufa da atmosfera.

Mineral: substância natural, sólida e cristalina, com uma composição química definida e uma estrutura atômica ordenada. Formado por processos geológicos e podem ser encontrados em rochas, solos e até na água.

Mineralização de CO₂ ou carbonatação mineral: processo no qual o CO₂ reage com certos tipos de formações rochosas porosas para formar minerais estáveis. Essas reações acontecem naturalmente ao longo de milhares de anos, mas podem ser aceleradas com processos industriais.

Mitigação (das mudanças climáticas): uma intervenção humana para reduzir emissões ou aumentar os sumidouros de gases de efeito estufa.

Neutralidade de carbono: objetivo global, alcançado quando as emissões de CO₂ são compensadas globalmente pelas remoções de CO₂ durante um período específico.

Pós reciclados de RCD: fração de RCD processada passante na peneira de 0,15 mm ($\emptyset < 0,15$ mm).

Reator: equipamento projetado para conter e controlar reações químicas em escala industrial. É projetado para otimizar as condições da reação, como temperatura e pressão, para garantir a produção de produtos desejados.

Sequestro de carbono: processo que ocorre de modo natural de remoção do CO₂ da atmosfera e armazenamento em florestas, solo e oceanos. Pode ser estimulado por tecnologias de captura e armazenamento de carbono.

Sustentabilidade: processo dinâmico que garante a persistência dos sistemas naturais e humanos de forma equitativa.

Transporte de carbono: processo de mover o CO₂ capturado por meio de um gasoduto ou por outros meios (por exemplo, navio) de sua fonte até um local de armazenamento adequado.

Carbono neutro:

Utilização de carbono (CO₂): uso do CO₂ para produzir combustíveis, rações, produtos químicos, materiais de construção ou outros produtos valiosos.

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1 INTRODUÇÃO

Atividades humanas que fazem uso de recursos naturais face à emissão intensa de gases de Efeito Estufa (GEE) têm contribuindo para o aquecimento global e mudanças climáticas. Segundo relatório do IPCC (2018) a humanidade deveria evitar um aumento em 1,5 °C na temperatura em relação aos níveis pré-industriais (1850-1900) até 2030, para conter impactos irreversíveis, como a extinção de ecossistemas, insegurança alimentar e hídrica, o descongelamento de geleiras e aumento dos níveis do mar, entre outras situações que podem inviabilizar a adaptação humana. Em 2024 o planeta terra superou esse patamar indesejado, atingindo 1,6 °C (Copernicus, 2025).

No Sexto Relatório de Avaliação (AR6), o Painel Intergovernamental das Alterações Climáticas relatou que as emissões em 2019 foram de aproximadamente 59 GtCO₂-eq¹, cerca de 12% superior às de 2010 e 54% superior às de 1990. Logo, para limitar o aquecimento a 2 °C em 2050, medidas mais ambiciosas precisam ser tomadas, a fim de garantir um futuro sustentável e habitável para todos. Neste sentido, além da necessidade de políticas públicas voltadas para os setores altamente emissivos, é essencial o uso de técnicas que permitam a captura e armazenamento de carbono (IPCC, 2023).

A indústria do cimento tem um papel fundamental no combate às mudanças climáticas, pois seu processo produtivo é responsável por aproximadamente 5 a 9% das emissões globais de CO₂ (Kaptan; Cunha; Aguiar, 2024; Marmier, 2023). O processo de produção do cimento requer moagem e calcinação de uma mistura de argila e calcário. A queima dos combustíveis fósseis é responsável por cerca de 40% do total das emissões de CO₂ enquanto a decomposição do calcário durante a calcinação é responsável pelo restante (Imbabi; Carrigan; McKenna, 2012; SNIC, 2019).

Aumentar a eficiência energética não é suficiente para impactar significativamente na redução das emissões, a calcinação do calcário também deve ser minimizada. Em vista disso, além da utilização de combustíveis renováveis, o uso de Materiais Cimentícios Suplementares (MCS), associado às tecnologias de Captura, Utilização e Armazenamento de Carbono, no inglês, *Carbon Capture, Utilization and Storage* (CCUS) (Antunes *et al.*, 2022; Miller *et al.*, 2018; Scrivener; John; Gartner, 2018), são consideradas estratégias fundamentais para a neutralidade de carbono na indústria do cimento e do concreto (GCCA, 2021; PCA, 2021).

¹ Medida internacionalmente aceita que expressa a quantidade de gases de efeito estufa em termos equivalentes da quantidade de dióxido de carbono.

Com relação aos MCS, incertezas relacionadas à disponibilidade e limitações normativas e geográficas, estimulam a busca por materiais alternativos, tais com o uso de cinzas de biomassas e argilas calcinadas (Dias *et al.*, 2022; Juenger; Snellings; Bernal, 2019; Lothenbach; Scrivener; Hooton, 2011; SNIC, 2019). Outro material que vem sendo estudado por diversos autores (Jin *et al.*, 2024; Li *et al.*, 2022; Rocha; Toledo Filho, 2023; Wang, C. *et al.*, 2024), são os pós obtidos a partir da reciclagem de resíduos da construção civil classe A, tais como componentes cerâmicos, argamassa e concreto, em agregados reciclados (BRASIL, 2002).

Os resíduos da construção civil também chamados de Resíduos de Construção e Demolição (RCD) representam cerca de 30 a 50% do total de resíduos sólidos gerados globalmente (Gálvez-Martos *et al.*, 2018; Gao *et al.*, 2023; Huang *et al.*, 2018; Liu, J. *et al.*, 2019; López Ruiz; Roca Ramón; Gassó Domingo, 2020), e sua quantidade aumenta proporcionalmente à tendência de urbanização global (Zhang, C. *et al.*, 2022). China, Estados Unidos e União Europeia são os três principais geradores deste resíduo (Kabirifar *et al.*, 2020). No Brasil, em 2023, foram gerados cerca de 44,46 milhões de toneladas de RCD (ABREMA, 2024) e estima-se que mundialmente o valor chegue a 10 bilhões de toneladas por ano (Wu *et al.*, 2019).

Com a conscientização sobre sustentabilidade e gestão dos recursos, a implementação da Economia Circular (EC) surgiu como um modelo potencial para minimizar o impacto negativo dos RCD no meio ambiente (Purchase *et al.*, 2022). Em conjunto com estratégias de redução e reutilização, relacionadas à fase de pré-construção e projeto (Atta; Bakhom, 2024), a reciclagem e uso de agregados reciclados em argamassas e concretos são as principais abordagens para a gestão eficaz do RCD (Kabirifar *et al.*, 2020; Purchase *et al.*, 2022), e que promovem a circularidade no setor da construção civil (Bonoli; Zanni; Serrano-Bernardo, 2021; Vásquez-Cabrera; Montes; Llatas, 2025).

O uso de agregados reciclados de RCD para a produção de argamassas e concretos já é regulamentado em vários países (Tam; Soomro; Evangelista, 2018) e formas de aplicação são investigadas por diversos autores (Kisku *et al.*, 2017; Nedeljković *et al.*, 2021; Sivamani; Renganathan, 2021). Recentemente iniciaram-se os estudos associados à produção e emprego de pós de RCD (material particulado com dimensões inferiores a 0,15 mm), como MCS. Estima-se que a fração de pó gerada durante o processo de reciclagem do RCD esteja entre 5 a 20% (Lu *et al.*, 2018; Oliveira; Dezen; Possan, 2020; Ulsen *et al.*, 2013; Wang *et al.*, 2022), podendo ser elevada mediante tratamentos físicos (cominuição) como a moagem da fração da areia (Bogas; Carriço; Pereira, 2019; Liu *et al.*, 2024;

Ma *et al.*, 2019; Wang *et al.*, 2020).

Devido à alta absorção de água e baixa reatividade (Shi *et al.*, 2016), soluções técnicas (tratamentos), a fim de potencializar o uso dos pós como MCS ao cimento Portland, vêm sendo avaliadas na literatura, tais como a mecanoativação (Costa; Gonçalves, 2022; Fediuk *et al.*, 2018; Vashistha *et al.*, 2023), ativação térmica (Baggio; Possan; Andrade, 2024; Bu *et al.*, 2023), tratamentos químicos com ácido tânico (Proença *et al.*, 2024; Wang *et al.*, 2022) e com CO₂, por meio da carbonatação mineral (Hu; He; Shi, 2024; Ye *et al.*, 2024; Zhang, T. *et al.*, 2023).

Além da valorização dos pós de RCD (Chai *et al.*, 2022), a carbonatação mineral também conhecida como mineralização de CO₂, pode ser usada para fixar CO₂ em larga escala, sendo uma alternativa ao armazenamento geológico, em oceanos e aquíferos (Olajire, 2013). O processo envolve a reação do CO₂ com minerais contendo cálcio e/ou magnésio formando carbonatos estáveis (Fernández Bertos *et al.*, 2004; Zhang, T. *et al.*, 2023) e pode ocorrer, naturalmente, por longos períodos por meio do intemperismo, mas também pode ser acelerado em reatores (Liu, W. *et al.*, 2021). Duas vias podem ser adotadas para a carbonatação mineral, direta ou indireta. Na carbonatação direta tem-se a rota seca (gás-sólido), geralmente com uma relação água/sólido (a/s) inferior a 0,2 (Lim *et al.*, 2010) e a rota aquosa (gás-líquido), onde os minerais ou pastas, respectivamente, reagem com o CO₂, enquanto na carbonatação indireta ocorre a extração do Ca ou Mg em condições alcalinas (Liu, W. *et al.*, 2021).

Embora o método de carbonatação a seco (gás-sólido) apresenta uma reação química mais lenta e com maiores dificuldades de controle de umidade (Yadav; Mehra, 2021), a configuração experimental é mais simples (Zajac, M. *et al.*, 2022) e resultados promissores têm sido observados na literatura. Um exemplo é o trabalho de Lu *et al.* (2018), que submeteram pós provenientes da moagem de pasta de cimento (64,5% CaO, 21,9% SiO₂ e 5,3% Al₂O₃) à mineralização de CO₂ em câmara de carbonatação (temperatura (T) de 20 ± 1 °C, umidade relativa (UR) de 60 ± 5% e concentração de CO₂ de 99%), até a completa conversão em carbonatos. A substituição de 20% do cimento Portland por esse material resultou em uma resistência à compressão 12% superior à da pasta de referência.

No caso de Kaliyavaradhan, Ling e Mo (2020), que expuseram a lama de concreto (CaO~52,81%) nas condições 20 °C (T), 65% (UR) e 20% de CO₂ por 72 horas, além da fixação de CO₂ de 20,4%, ou seja, 204,35 kg/tonelada de material, os autores verificaram que com a substituição de 20% da massa de cimento Portland em argamassas, o índice de desempenho com o cimento foi de 84,85% (aos 28 dias), ou seja, valor superior ao requerido

para uso dos materiais como MCS que é de 75% pela ASTM 311M (ASTM, 2022).

Também é relatado o emprego de pós provenientes de pastas de cimento hidratadas após a mineralização de CO₂ pela rota gás-sólido, em substituição de até 30% da massa do cimento Portland. Zhang, J. *et al.* (2023), conduziram a mineralização de CO₂ em pós reciclados de pasta de cimento (65,4% CaO, 21,0% SiO₂ e 5,4% Al₂O₃) por 7 dias. O ensaio teve como condições 20 ± 2 °C (T), 60 ± 5% (UR) e 20 ± 2% de CO₂. Os resultados revelaram um ganho de resistência à compressão aos 28 dias em relação à pasta de referência de 18%, com redução nos tempos de pega inicial e final em cerca de 25%. Em condições similares de mineralização (20 ± 2 °C (T), 70 ± 5 % (UR), 20% de CO₂ e exposição por 3 dias), Wu *et al.* (2021) observaram um ganho de 12,6% na resistência à compressão aos 28 dias, neste caso com aplicação em argamassas.

Neste contexto, observou-se que a maioria dos trabalhos empregou pós reciclados provenientes de matrizes cimentícias produzidas de forma controlada em laboratório, sem exposição ao CO₂ atmosférico ou a condições ambientais. Considerando que a tecnologia de CCUS ainda não é globalmente explorada, estando mais avançada na América do Norte, China, Índia e Europa (GCCA, 2021), esse estudo quantifica o potencial fixação de CO₂ de pós de RCD submetidos à mineralização de CO₂, via carbonatação gás-sólido, avaliando também seu emprego como MCS e a possível contribuição destas estratégias de CCUS na mitigação das emissões de carbono na indústria cimenteira.

1.1 QUESTÕES DE PESQUISA

Levando em consideração que em todas as fases do ciclo de vida do cimento-concreto é gerada uma quantidade considerável de CO₂ e de resíduos, tem-se como questões de pesquisa:

- i) Pós reciclados de resíduos de construção e demolição (RCD), após submetidos à mineralização de CO₂ por carbonatação acelerada, apresentam potencial para serem utilizados como materiais cimentícios suplementares (MCS) na composição de cimentos menos emissivos?
- ii) A variação de parâmetros do material (granulometria e origem dos pós reciclados de RCD) e operacionais (temperatura, umidade e tempo de exposição), influencia na eficiência da mineralização de CO₂ e no desempenho dos pós reciclados como MCS?

1.2 OBJETIVOS

O objetivo geral deste estudo é avaliar a mineralização de CO₂ em pós reciclados de RCD, considerando o emprego como material cimentício suplementar, a fim de mitigar as emissões de CO₂ e contribuir com a economia circular na indústria da construção.

Em paralelo foram estabelecidos objetivos específicos, a saber:

- (i) Avaliar processos de cominuição e de absorção de água para a obtenção de pós reciclados de RCD para uso como MSC;
- (ii) Analisar a fixação de CO₂ em pós de RCD de diferentes origens (misto e concreto) e granulometrias;
- (iii) Avaliar o efeito da umidade, da temperatura e do tempo de exposição ao CO₂ na fixação de CO₂ em pós de concreto;
- (iv) Avaliar a influência do tipo de cimento e da relação a/c do concreto de origem dos pós reciclados de RCD na fixação de CO₂;
- (v) Avaliar o uso dos pós reciclados de concreto com diferentes propriedades físico-químicas quando submetidos à mineralização de CO₂ como material cimentício suplementar.

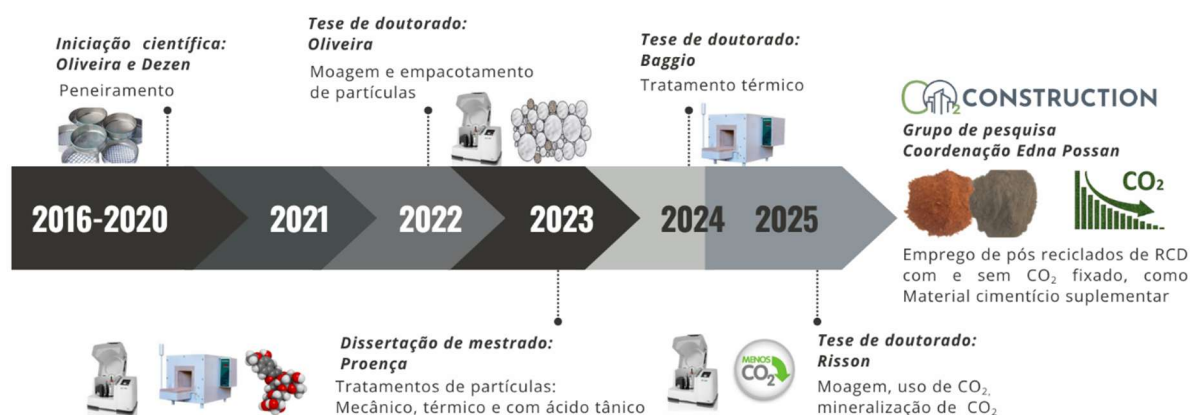
1.3 ORIGINALIDADE

A presente tese tem como originalidade a mineralização de CO₂ em pós reciclados de RCD, com diferentes origens e composições químicas, obtidos de três fontes: usinas de reciclagem, concreteiras e produzidos em laboratório (propriedades conhecidas). No Brasil, até o momento, a mineralização de CO₂ em pós reciclados de RCD e avaliação da sua aplicação como MCS ainda não foi explorado, indicativo de que esse estudo é pioneiro na esfera nacional. Além disso, o trabalho avança como uma proposta de otimização do processo via gás- sólido ao investigar, de forma sistemática variáveis críticas, como a granulometria dos pós, o tempo de exposição ao CO₂, a temperatura e a presença de água, visando maximizar a fixação de CO₂ e viabilizar o emprego como MCS. Na esfera internacional, o estudo destaca-se por empregar como MCS pós reciclados de concretos de múltiplas fontes após o processo de mineralização de CO₂, quantificando o potencial dessa tecnologia para a redução e remoção de emissões, com vistas à sua inserção no mercado de carbono.

1.4 HISTÓRICO DA PESQUISA

As pesquisas na área de pós reciclados de resíduos de construção e demolição (RCD), vinculadas ao grupo de pesquisa CO₂-Construction² da Universidade Federal da Integração Latino-Americana (UNILA), tiveram início em 2016 (Figura 1), a partir das atividades desenvolvidas por alunos de iniciação científica. Com a primeira publicação em 2020, intitulada *Uso da fração fina do resíduo de concreto como substituto ao cimento Portland* (Oliveira; Dezen; Possan, 2020). Este trabalho inaugurou uma linha de pesquisa inédita, voltada à valorização de pós reciclados de RCD e sua incorporação em matrizes cimentícias, estabelecendo um ponto de partida para uma área pouco explorada no cenário acadêmico nacional.

Figura 1 - Histórico da pesquisa vinculadas ao grupo de pesquisa CO₂-Construction



Os estudos foram aprofundados na tese de doutorado *Aproveitamento Da Fração Fina De Resíduo De Concreto Como Substituto Ao Cimento Portland* (Oliveira, 2022), defendida em 2022, que investigou a associação entre a moagem dos resíduos e a técnica de empacotamento de partículas. Este estudo aprofundou as bases científicas para a substituição do cimento Portland por pós reciclados, reforçando o potencial técnico e ambiental dessa abordagem, e gerando publicações em periódicos (Oliveira; *et al.*, 2025; Oliveira *et al.*, 2024).

Na sequência, além da moagem, outros tratamentos foram estudados na dissertação de mestrado intitulada *Pós de RCD tratados por processos físico-químicos para a composição de matrizes cimentícias de baixo carbono* (Proença, 2023), e que resultou na publicação *Pó Ativado por Tratamento Mecânico, Térmico e com Ácido Tânico: Uma Opção para a Circularidade na Construção* (Proença *et al.*, 2024).

² <https://www.co2construction.com/>

Em 2024, outra tese de doutorado foi dedicada à caracterização e tratamento de pós de RCD: *Ativação termomecânica de pó de resíduo de construção e demolição como material cimentício suplementar* (Baggio, 2024). Este estudo originou a publicação *Caracterização físico-química de pó de resíduos de construção e demolição com ativação termomecânica para uso como material cimentício suplementar* (Baggio; Possan; Andrade, 2024), ampliando o entendimento sobre a utilização dos resíduos como materiais cimentícios suplementares.

No ano de 2025, o grupo de pesquisa apresenta esta tese com uma abordagem integrada, articulando os conhecimentos acumulados ao longo dos anos sobre moagem, eficiência energética dos processos e balanço de emissões de CO₂ com a temática de uso e captura de carbono em materiais à base de cimento via mineralização de CO₂. A pesquisa reforça o compromisso do grupo com a transição para uma construção civil de baixo carbono, temática de grande relevância na atual agenda global de sustentabilidade, sendo pioneira no Brasil e posicionando o grupo CO₂-Construction na vanguarda da utilização de resíduos da construção como materiais cimentícios inovadores, com e sem fixação de CO₂, com elevado potencial de transformação para a indústria da construção no país.

Em 2024, o grupo recebeu reconhecimento nacional, com a premiação no 2º Desafio ENTAC, promovido pela Associação Nacional de Tecnologia do Ambiente Construído (ANTAC), no eixo *Processo da Produção do Ambiente Construído*. O projeto vencedor, intitulado “*Cimento Bioativado com Pó Reciclado de RCD com CO₂ Fixado (PCR BIO FIX)*”. Atualmente, os estudos em pós reciclados com o uso e fixação de CO₂ estão em pleno desenvolvimento pelo grupo de pesquisa internacional CO₂-Construction.

1.5 ESTRUTURA DA TESE

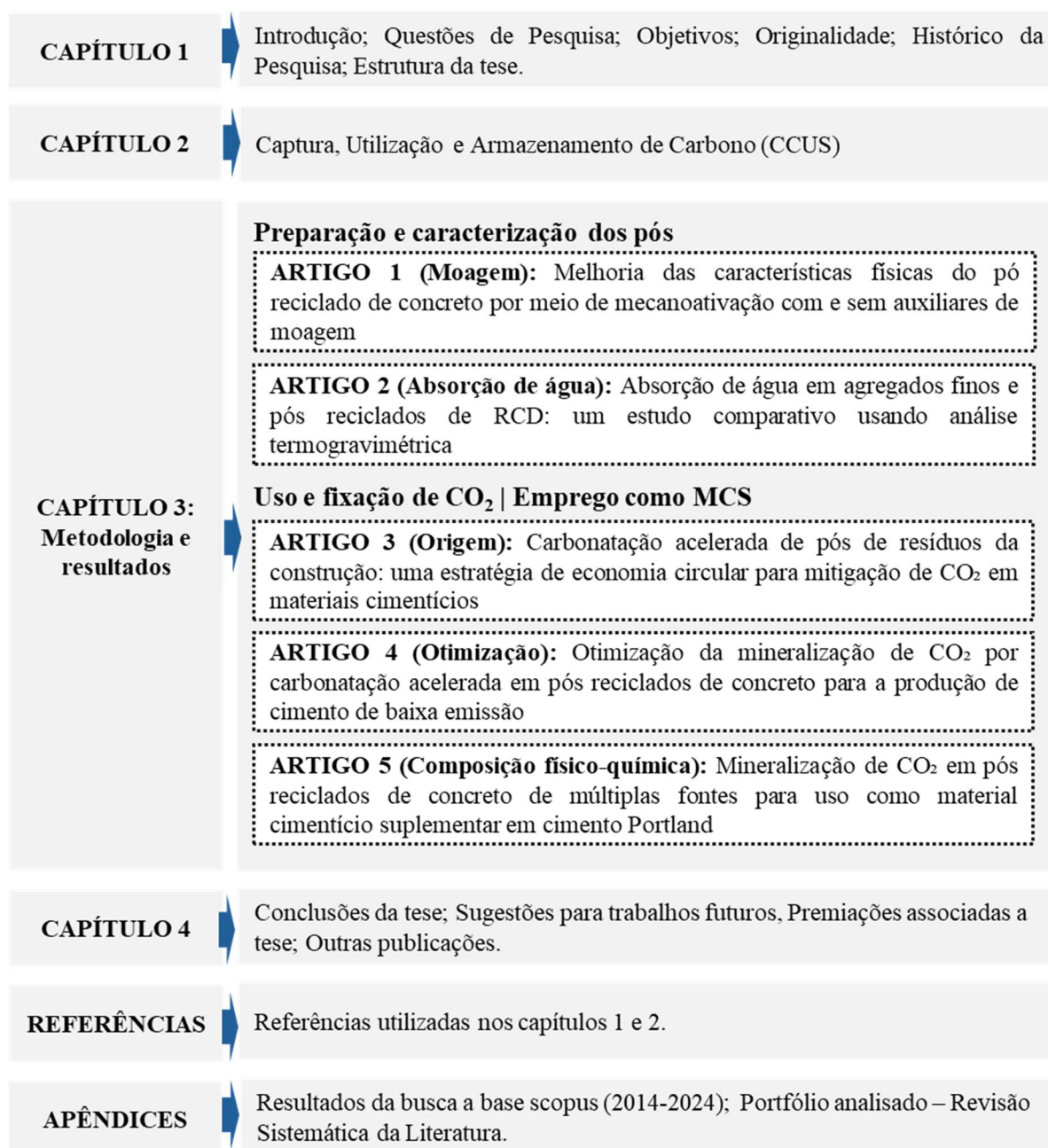
Esta tese foi estruturada em modelo de artigos, composta por um capítulo introdutório, revisão bibliográfica, metodologia e resultados, conclusões, referências e apêndices, conforme ilustrado na Figura 2.

No *capítulo 1*, tem-se a introdução, contemplando as questões de pesquisa, os objetivos, a originalidade da tese, o histórico da pesquisa e como a mesma foi estruturada.

O *capítulo 2* contempla a revisão bibliográfica sobre as tecnologias de CCUS, com ênfase na mineralização de CO₂ em materiais cimentícios. São abordados os mecanismos de difusão e fixação de CO₂ nessas matrizes, culminando na apresentação de uma Revisão

Sistemática da Literatura (RSL), que envolve a mineralização de CO₂ como uma estratégia promissora para a fixação de CO₂ em pós e finos reciclados de RCD, além de seu desempenho quando usados em pastas ou argamassas.

Figura 2- Fluxograma da tese



Fonte: Autora (2025).

No *capítulo 3*, são apresentados na íntegra os cinco artigos que compõem a tese, sendo dois relacionados ao preparo e caracterização das partículas e os demais ao uso e fixação de CO₂ (CCUS) com emprego dos pós reciclados de RCD como MCS.

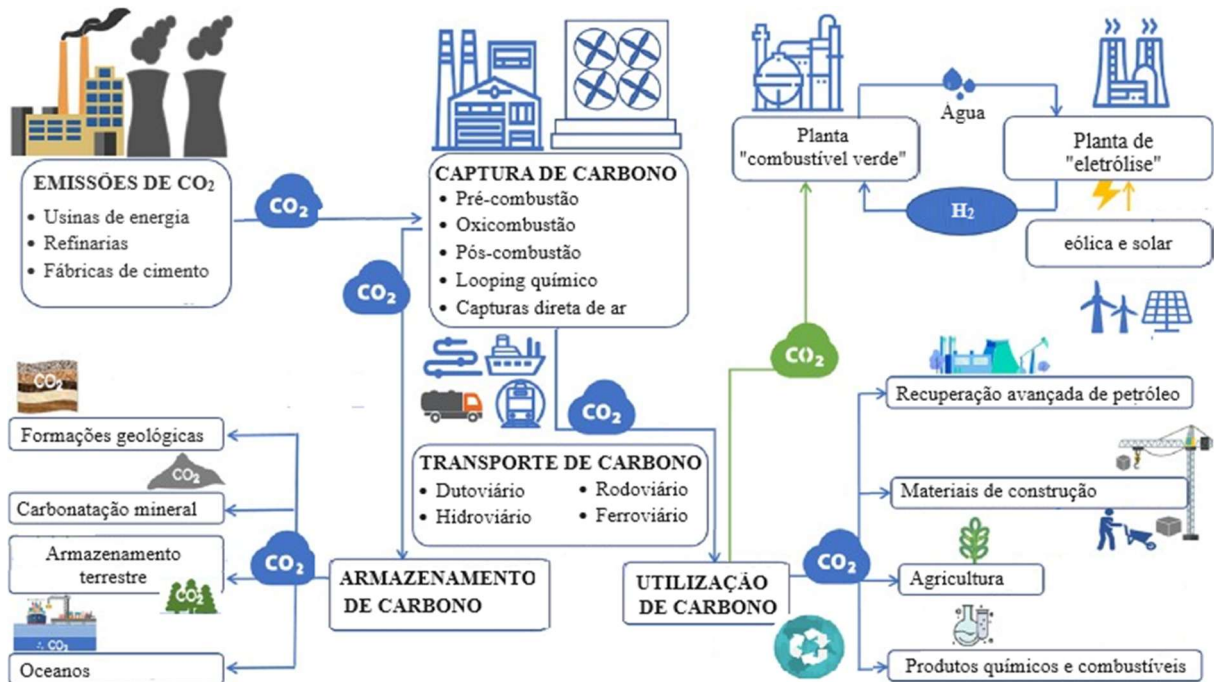
As conclusões da tese são indicadas no *capítulo 4*, além de ser apresentado sugestões para trabalhos futuros, as premiações e publicações associadas a tese e/ou desenvolvida no período. Seguido são listados as referências e os apêndices.

Observa-se que, nos artigos 1, 2 e 3, foi utilizado o mesmo lote de pós reciclados de concreto (RCP), enquanto, nos artigos 4 e 5, foi empregado um novo lote, também proveniente de concretos reciclados. Essa distinção justifica as variações observadas na composição química, especialmente nos teores de CaO.

2 CAPTURA, UTILIZAÇÃO E ARMAZENAMENTO DE CARBONO (CCUS)

As tecnologias de Captura, Utilização e Armazenamento de Carbono (CCUS) são geralmente aplicadas às fontes altamente emissoras de CO₂ como usinas de geração de energia térmica, refinarias, fábricas de cimento, entre outros, para capturar o CO₂ e armazená-lo de forma segura e permanente em reservatórios geológicos ou utilizá-los para a geração de energia ou no desenvolvimento de novos produtos (L'Orange Seigo; Dohle; Siegrist, 2014; Raza *et al.*, 2019), conforme ilustrado na Figura 3.

Figura 3- Visão geral das tecnologias que envolvem a CCUS



Fonte: Traduzido de Zhao *et al.* (2023).

A captura de CO₂ pode ser realizada conforme cinco mecanismos: (i) a captura pré-combustão, que ocorre antes da queima do combustível fóssil, onde normalmente converte-se o combustível em um gás com remoção do CO₂ antes da combustão (por exemplo a transformação do metano (CH₄) em hidrogênio (H₂)); (ii) a captura baseada na oxidação, que envolve a queima de combustíveis fósseis em um ambiente rico em oxigênio, resultando em CO₂ e vapor de água. O gás resultante é resfriado e condensado para remover o vapor de água, deixando um fluxo concentrado de CO₂ que pode ser armazenado; (iii) a captura pós-combustão, geralmente realizada em fontes que usam combustíveis fósseis ou de biomassa (usinas, siderúrgicas e cimenteiras), sendo essa considerada a tecnologia mais madura e com menor custo quando comparada às demais (CAS, 2022; Garcia *et al.*, 2022;

Madejski *et al.*, 2022; Olabi *et al.*, 2022; Raza *et al.*, 2019); (iv) a captura de combustão em looping químico (no inglês, *Chemical looping Combustion* - CLC) é baseado em decompor a reação entre o combustível e o ar em duas reações gás-sólido com a ajuda de um transportador de oxigênio (Liu; Lu; Wang, 2023; Zhao *et al.*, 2023) e (v) a captura direta de ar (no inglês, *Direct Air Capture* - DAC), considerada uma tecnologia futurista e ainda requer ampliação para determinar sua viabilidade econômica (Ma *et al.*, 2022).

Após a captura, o CO₂ é comprimido e transportado por dutos, navios, ferrovias ou caminhões e armazenado em formações geológicas profundas como reservatórios de petróleo e gás esgotados ou aquíferos salinos (Becattini *et al.*, 2022; L'Orange Seigo; Dohle; Siegrist, 2014). A quantidade de CO₂ e a distância entre o local de captura e de armazenamento são fundamentais para a escolha do modo de transporte (Liu; Lu; Wang, 2023). Estima-se que os custos para a captura e armazenamento de carbono variam de US\$ 15 a US\$ 130/tCO₂ (Statista, 2023), sendo que o transporte e armazenamento podem variar de US\$ 4 a US\$ 45/tCO₂ (Rubin; Davison; Herzog, 2015; Smith *et al.*, 2021).

Tendo em conta as limitações geográficas, os desafios tecnológicos, os custos, as incertezas com relação à capacidade e a eficiência do armazenamento do CO₂ em meios geológicos (Raza *et al.*, 2019; Zahasky; Krevor, 2020) e a necessidades de sistemas robustos de monitoramento (Flohr *et al.*, 2021), diversos autores, como Burkart *et al.* (2019); Hepburn *et al.* (2019); Osman *et al.* (2021) e Zajac *et al.* (2022), apontam que as tecnologias que envolvem a utilização do CO₂ no desenvolvimento de novos produtos pode ser considerada um trampolim para a implementação em larga escala da tecnologia CCUS.

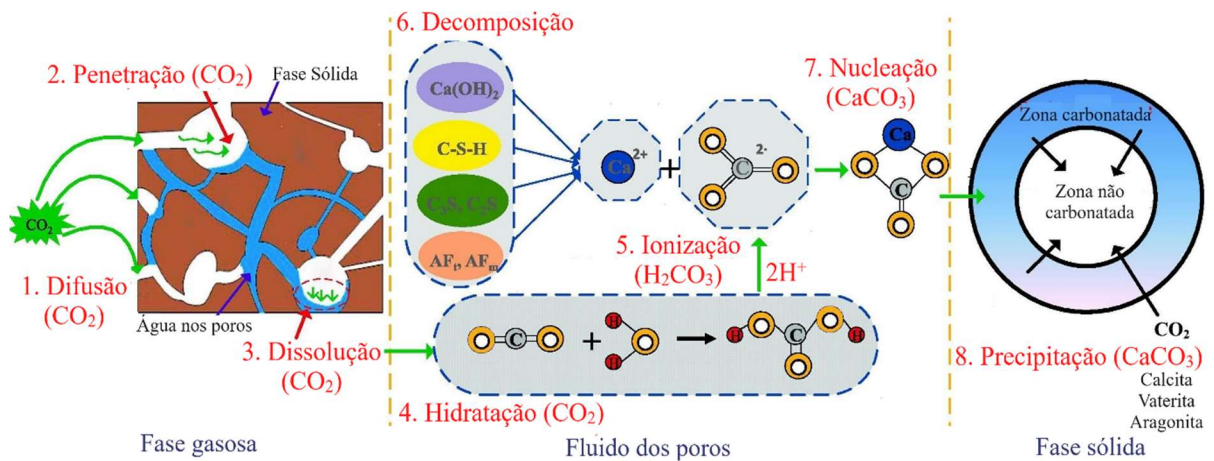
A utilização ou uso de CO₂ foi proposta em 1970, muito antes da mitigação do clima se tornar uma preocupação mundial (Aresta; Dibenedetto, 2007; Aresta; Forti, 2012), existindo várias rotas plausíveis de aplicações em produtos comerciais (combustíveis e produtos químicos), onde o CO₂ pode substituir o carbono fóssil, para produzir o mesmo produto ou produtos com função semelhante, seja por processos eletroquímicos, biológicos, catalíticos, de copolimerização ou mineralização (Godin *et al.*, 2021; Gupta *et al.*, 2024; Hepburn *et al.*, 2019).

Outras rotas de utilização não convencionais como a Bioenergia com Captura e Armazenamento de Carbono (BECCS, Bioenergy with Carbon Capture and Storage, em língua inglesa), intemperismo aprimorado, técnicas florestais, práticas de manejo da terra e biochar, também são relatados na literatura (Hepburn *et al.*, 2019).

2.1 MINERALIZAÇÃO DE CO₂ EM MATERIAIS CIMENTÍCIOS

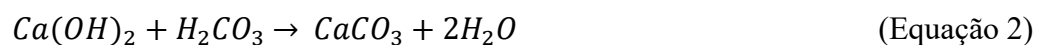
A carbonatação mineral ou mineralização de CO₂ é um processo físico-químico que ocorre através da reação entre o CO₂ atmosférico e os minerais contendo magnésio (Mg) e cálcio (Ca) para formar carbonatos estáveis e insolúveis (Hu; He; Shi, 2024; Olajire, 2013; Power *et al.*, 2013). Em materiais cimentícios, pode ser explicado por meio de um processo de difusão, dissolução e precipitação (Figura 4), gerando produtos à base de carbonato e gel de sílica (SiO₂) (Gunning; Hills; Carey, 2010; Li; Wu, 2022; Zhang *et al.*, 2023).

Figura 4- Ilustração dos processos físicos e químicos de mineralização de CO₂ em materiais cimentícios



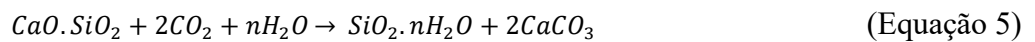
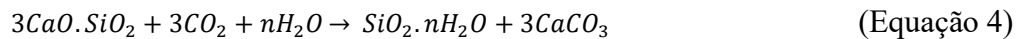
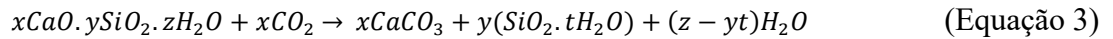
Fonte: Traduzido de Pu *et al.*(2021).

Inicialmente, o CO₂ se difunde pelos poros dos materiais cimentícios preenchidos parcialmente com água, e se dissolve produzindo o ácido carbônico (Equação 1), que reage com o hidróxido de cálcio (Ca(OH)₂; CH), precipitando na forma de carbonato de cálcio (CaCO₃) (Equação 2) (Johannesson; Utgenannt, 2001; Pu *et al.*, 2021; Yang *et al.*, 2003).

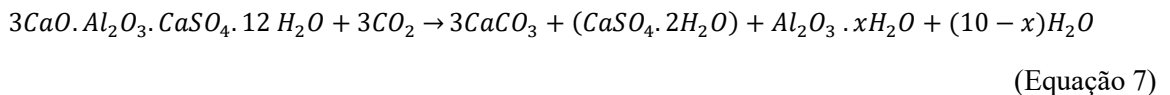
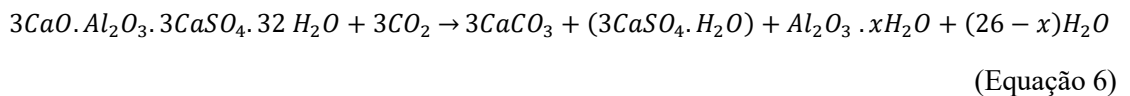


Além do hidróxido de cálcio, também podem ser carbonatados os silicatos de cálcio hidratado (xCaO·ySiO₂·zH₂O, C-S-H) (Equação 3) (Liu, X. *et al.*, 2022; Lu *et al.*, 2023) e as partículas de cimento não hidratado, como silicato tricálcico (3CaO·SiO₂, C₃S) (Equação 4) e silicato dicálcico (2CaO·SiO₂, C₂S) conforme Equação 5 (Luo *et al.*, 2024; Pu *et al.*, 2021;

Yang *et al.*, 2003). A sílica gel amorfa e o carbonato de cálcio são geralmente os produtos finais da carbonatação do C-S-H (Liu, B. *et al.*, 2021; Pu *et al.*, 2021, 2023), e a água (H₂O) é liberada, como produto da reação química (Arandigoyen *et al.*, 2006). Não há consenso sobre a dependência da taxa de carbonatação da relação Ca/Si, visto à complexidade do C-S-H (Liu, X. *et al.*, 2022).



Também é relatado que a etringita (AFt) e o monossulfoaluminato (AFm) podem ser carbonatados, respectivamente conforme Equações 6 e 7 (Chen *et al.*, 2020).



O carbonato de cálcio tende a se precipitar nos poros em diferentes morfologias, calcita rômica, aragonita tipo agulha e vaterita esférica. A calcita é a fase mais estável à temperatura ambiente (~25°C) em condições atmosféricas normais (~1 atm), enquanto a aragonita e a vaterita são polimorfos metaestáveis que se transformam prontamente na fase estável a calcita (Peter *et al.*, 2008; Šavija; Luković, 2016). Nesse processo, se os fatores cinéticos predominam, o CaCO₃ precipita como aragonita ou como vaterita, ambos os polimorfos se converterão finalmente em calcita, o polimorfo mais estável (Arandigoyen *et al.*, 2006; Han *et al.*, 2005; Kitamura, 1989).

Conforme o polimorfo formado, há mudanças na porosidade e estrutura dos poros de matrizes cimentícias. Por exemplo, como o volume molar da calcita (36,93 cm³) é cerca de 11% maior que o volume do hidróxido de cálcio (33,20 cm³), há preenchimento dos vazios e densificação da microestrutura do material cimentício (Arandigoyen *et al.*, 2006),

melhorando o desempenho mecânico (Zajac, Maciej *et al.*, 2022). Caso ocorra a descalcificação do C-S-H, a porosidade pode aumentar (Liu, L. *et al.*, 2019; Zhan *et al.*, 2021).

Durante a vida útil do material cimentício, a carbonatação mineral pode ocorrer em processo natural. No entanto, devido à baixa concentração atmosférica de CO₂, atualmente em 425,38 partes por milhão ou 0,425% em volume (Global Monitoring Laboratory, 2024) e ao coeficiente de difusão de CO₂ em materiais à base de cimento (10^{-10} a 10^{-12} m²/s), o processo poderia demorar décadas (Campos Neto *et al.*, 2023; Georget; Prévost; Huet, 2018; Namouniara; Turcry; Aït-Mokhtar, 2016).

Para acelerar as reações são utilizados reatores onde se aumenta a concentração de CO₂. Duas rotas são possíveis: a cura carbônica, uma técnica que envolve a exposição dos materiais cimentícios ao CO₂, seja no estado fresco ou em idades iniciais, sendo este último processo geralmente utilizado em pré-fabricados (Fortunato; Parsekian; Neves Junior, 2022; Liang *et al.*, 2024) e a mineralização de CO₂ em agregados reciclados (Liu, B. *et al.*, 2021; Pu *et al.*, 2021; Tam *et al.*, 2020). Recentemente, observa-se um crescente interesse no uso de CO₂ em pós provenientes de fornos de cimento, lama de concreto, pastas de cimento hidratadas e pós reciclados de concreto (Zhang, T. *et al.*, 2023).

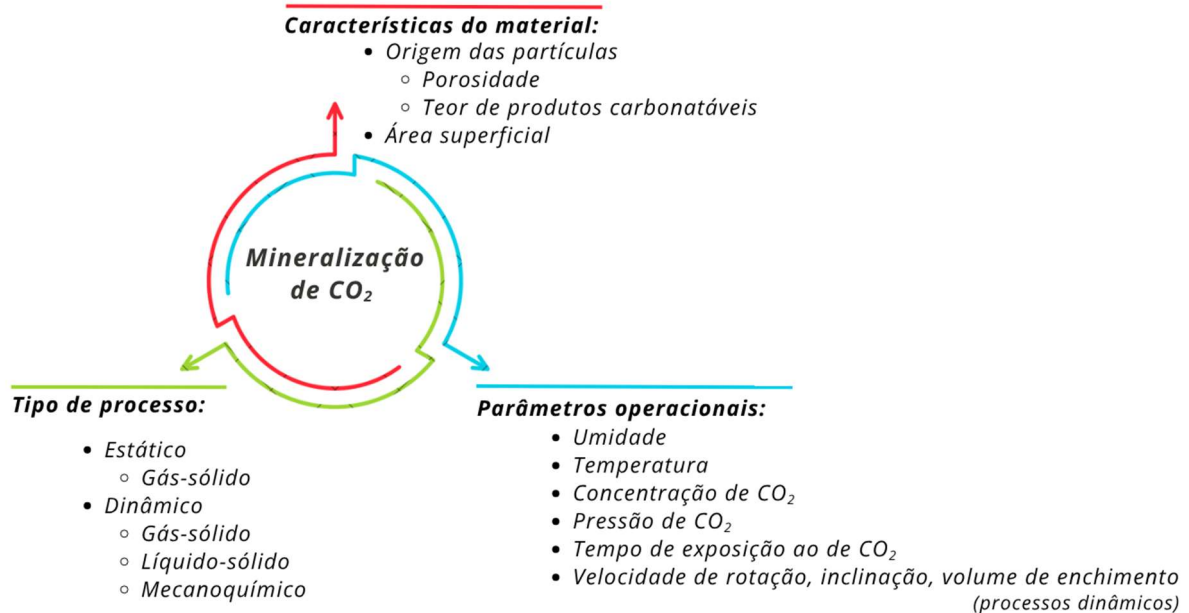
No contexto da mineralização de CO₂, múltiplos fatores atuam de forma interdependente na fixação de CO₂ em materiais cimentícios (Ben Ghacham *et al.*, 2017; Fernández Bertos *et al.*, 2004; Thonemann *et al.*, 2022; Wang; Xiao; Duan, 2022; Zajac *et al.*, 2021; Zhang, T. *et al.*, 2023) a saber (Figura 5): as características do material, o tipo de processo aplicado e os parâmetros operacionais.

A origem do material pode influenciar a mineralização de CO₂ de duas formas: pela quantidade de cálcio (CaO) disponível para reagir com o CO₂ (Li *et al.*, 2023; Sereng, 2020) e pela porosidade capilar do material. Em materiais cimentícios, o cálcio está presente principalmente sob duas formas: hidróxido de cálcio (Ca(OH)₂) e silicato de cálcio hidratado (C-S-H) (Zajac *et al.*, 2021; Zhang, T. *et al.*, 2023). A presença de adições minerais, como sílica ativa e escória de alto-forno e o tipo de cimento empregado, podem afetar a quantidade de Ca(OH)₂ disponível para a mineralização de CO₂, bem como a porosidade do material, impactando a profundidade e velocidade da reação química (Felix; Possan, 2018; Jiang *et al.*, 2024).

Com relação ao tamanho das partículas, quanto maior a área de superfície específica disponível para reagir com o CO₂, maior a fixação de CO₂. No entanto, devido a maior compacidade, a difusão do gás pode ser afetada (Dong *et al.*, 2019; Fernández Bertos *et*

al., 2004; Jang *et al.*, 2016), e nesse sentido o uso de reatores dinâmicos pode favorecer o acesso do CO₂ às partículas (Dos Reis *et al.*, 2020, 2021; Sereng, 2020).

Figura 5- Fatores que influenciam na mineralização de CO₂ em materiais cimentícios particulados



Fonte: Autora (2025).

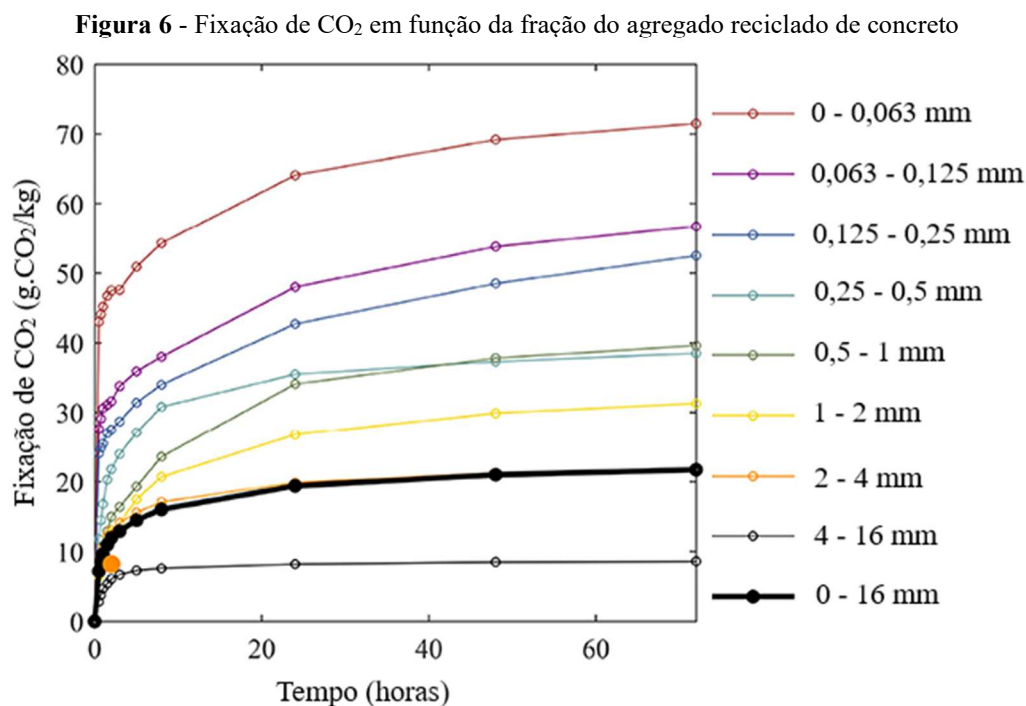
No que tange aos processos, diferentes reatores foram estudados para otimizar as reações gás-sólido, incluindo sistemas em pilhas com ou sem agitação, reatores de leito fixo ou fluidizado e tambores rotativos (Wehrung *et al.*, 2025). Entre as principais vantagens dessa rota, destacam-se as altas taxas de reação próximas à superfície e a possibilidade de operar com menor intensidade energética, desde que se tolere a reação parcial dos elementos alcalinos, o que pode, no entanto, limitar sua aplicação em larga escala (Walker; Bell; Rippy, 2024; Wang, 2024).

A rota considerada mais eficiente para a conversão de resíduos em carbonatos é rota aquosa, que ocorre na água seja na ausência ou presença de produtos químicos tais como ácidos, bases ou sais, que podem ser usados para acelerar o processo de reação, tais como NaCl e NaHCO₃ (O'Connor *et al.*, 2005), entre outros (Yadav; Mehra, 2021). No entanto, essa rota ainda não é viável economicamente, em razão do alto custo operacional e do baixo valor de mercado do carbono. Além disso, a adição de produtos químicos não recuperáveis deve ser evitada em estudos futuros (Veetil; Hitch, 2020). Outro processo de mineralização de CO₂ considerado inovador, é a chamada carbonatação mecanoquímica, que ocorre na presença de água e combina, simultaneamente, a injeção de CO₂ com a aplicação de força mecânica por

meio do impacto de alta energia de bolas. Esse processo promove a formação de carbonatos de cálcio reativos (Zhao *et al.*, 2024a; Zhao *et al.*, 2024b).

Para o processo gás-sólido, considerando as partículas com menor porosidade, a difusão do CO₂ é influenciada principalmente pelo teor de água; já em partículas mais porosas, a cinética da reação química passa a ser o fator predominante (Thiery *et al.*, 2013). Quando o teor de água é relativamente alto, a difusão de CO₂ é bloqueada pela água na estrutura dos poros, e quando o teor de água é baixo, o CO₂ não consegue ser dissolvido. Assim, deve-se obter um teor ótimo de água, que varia de 10 a 25% (Fang; Chang, 2015; Teramura; Isu; Inagaki, 2000) e uma umidade relativa de 50 a 70% (Zhang, T. *et al.*, 2023).

O tempo de exposição ao CO₂ atua de forma complementar na fixação de CO₂ (Dos Reis *et al.*, 2021). Todas as frações de agregados e pós reciclados de concreto apresentaram a mesma tendência: uma rápida fixação inicial de CO₂, que diminui com o aumento do tempo de exposição (Figura 6). Observa-se que o material mais fino apresentou uma fixação de CO₂ mais rápida e em maior quantidade, o que pode ser atribuído à maior área de superfície específica e à composição química mais rica em cimento hidratado, quando comparado às frações maiores, que tendem a ser mais ricas em sílica (Tiefenthaler *et al.*, 2021).



Fonte: Traduzido de Tiefenthaler *et al.* (2021).

Quanto à temperatura, observou-se em geral o uso entre 20 e 25 °C (Pu *et al.*, 2023), valores superiores são benéficos para a lixiviação do cálcio (Ca) das

partículas sólidas, mas retardam a solubilidade do CO₂ (Jang *et al.*, 2016). Além disso, a temperatura também influencia no formato do polimorfo formado, por exemplo, a 60 °C, a vaterita é a principal fase formada, sendo completamente convertida em calcita a 140 °C. Vale destacar que, acima de 100 °C, há uma aceleração na evaporação da água, o que pode comprometer as reações químicas durante o processo de mineralização do CO₂ (Wang; Noguchi; Nozaki, 2019; Wu, Y. *et al.*, 2022).

A concentração de CO₂ varia entre 5 a 100% (Zhang, T. *et al.*, 2023), sendo que 15% corresponde ao teor típico de CO₂ dos gases de exaustão do processo de fabricação de cimentos (Kaddah *et al.*, 2022; Sereng *et al.*, 2021; Torrenti *et al.*, 2022). Por sua vez para a pressão (Pa), 1 bar ($\approx 0,987$ atm) foi relatada como adequadamente eficiente (Dos Reis *et al.*, 2020), sendo recomendado o uso de até 5 bar, por questões práticas e econômicas (Fernández Bertos *et al.*, 2004; Gholizadeh-Vayghan *et al.*, 2020). A aplicação de um ambiente pressurizado é extremamente difícil para a industrialização devido aos altos custos operacionais e de segurança (Poon *et al.*, 2023).

A velocidade de rotação, inclinação e volume de enchimento, também devem ser levadas em consideração em processos dinâmicos (Brück *et al.*, 2018; Dos Reis *et al.*, 2020; Lombardi; Carnevale; Pecorini, 2016).

2.2 USO DE CO₂ EM PÓS E FRAÇÕES FINAS RECICLADAS DE RCD³ E APLICAÇÕES

2.2.1 Origem e produção dos pós e frações finas

Os Resíduos de Construção e Demolição (RCD) são aqueles provenientes das construções, reformas, reparos e demolições de obras de construção civil, e os resultantes da preparação e da escavação de terrenos, sendo os pertencentes a “Classe A”, possíveis de serem reutilizáveis ou recicláveis como agregados, tais como componentes cerâmicos (tijolos, blocos, telhas, placas de revestimento, entre outros), argamassa e concreto (BRASIL, 2002).

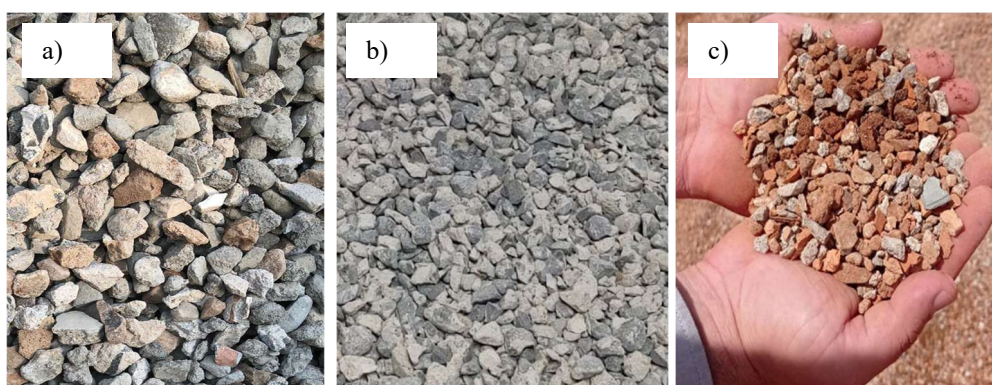
De acordo com Angulo e Ulsen (2023) os processos de reciclagem do RCD em agregados reciclados variam conforme a granulometria e a classificação requerida e, de

³ Neste estudo, considera-se como fração fina de RCD, o material que passa pela peneira de abertura 4,75 mm (nº 4) e fica retido na peneira de 0,15 mm (nº 100), ou seja, com diâmetro entre 0,15 mm e 4,75 mm ($0,15 \text{ mm} < \text{Ø} < 4,75 \text{ mm}$). O pó é definido como o material passante pela peneira de 0,15 mm (nº 100). Na literatura, os termos não são consensuais, sendo comum o uso da denominação fração fina também para os pós.

modo simplificado, é composto por cinco operações unitárias: (i) triagem, onde ocorre a separação do RCD classe A dos demais; ii) escalpe, consiste em remover a fração fina que pode estar em excesso devido à mistura com o solo; (iii) britagem, processo mecânico de fragmentação de separação de partículas, sendo comum a britagem primária e secundária; (iv) separação: remoção de materiais contaminantes como materiais magnéticos ou outras substâncias indesejáveis e (v) peneiramento e classificação: processo de separação com diferentes tamanhos.

Os agregados reciclados podem ser classificados, segundo a NBR 15116 (ABNT, 2021), em: (i) Agregado Reciclado Cimentício (ARCI), sendo aquele que contém predominantemente materiais como concretos, argamassas, blocos pré-moldados de concreto entre outros, podendo ter teores reduzidos de cerâmica vermelha (blocos, telhas de cerâmica vermelha, entre outros); (ii) Agregado Reciclado de Concreto (ARCO), constituído predominantemente por resíduos de concreto e (iii) Agregado Reciclado Misto (ARM) composto por materiais cimentícios e materiais cerâmicos, conforme visto na Figura 7.

Figura 7- Classificação dos agregados reciclados quanto à origem: a) Cimentício; b) Concreto e c) Misto



Fonte: Autora (2025).

O uso de agregados reciclados é reconhecido e consolidado na literatura (Fanijo *et al.*, 2023; Guo *et al.*, 2018; Joseph *et al.*, 2023; Tam; Soomro; Evangelista, 2018), com diversas aplicações na construção civil. Entre os avanços mais recentes, destacam-se o melhoramento nos processos de reciclagem para maior aproveitamento da fração fina ($0,15 \text{ mm} < \varnothing < 4,75 \text{ mm}$), e lavagem do agregado para a retirada do material menor que $0,15 \text{ mm}$ (Angulo; Ulsen, 2023; Sbardelotto *et al.*, 2024; Ulsen *et al.*, 2021).

A fração menor que $0,15 \text{ mm}$ (pós reciclados) apresenta baixa reatividade e, por isso, pode ser utilizada como carga inerte (Tang *et al.*, 2020; Xiao *et al.*, 2018). Quando submetida a tratamentos mecânicos, químicos ou térmicos, sua reatividade pode ser

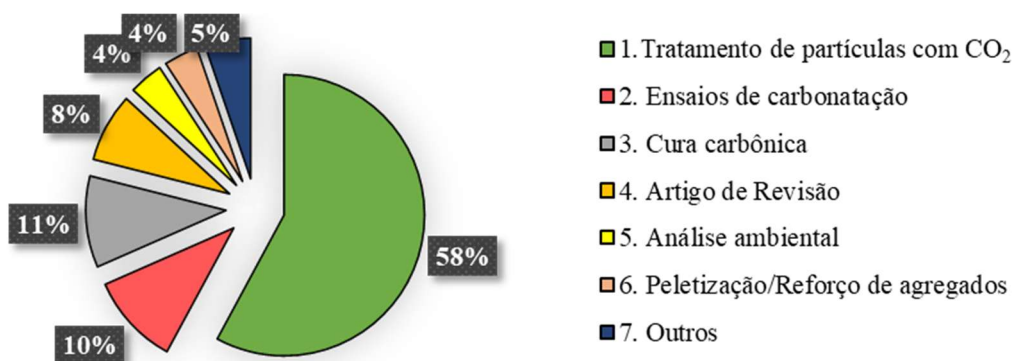
significativamente aumentada, viabilizando seu uso como MCS (Rocha; Toledo Filho, 2023; Schoon *et al.*, 2015; Snellings; Suraneni; Skibsted, 2023; Zhang, D. *et al.*, 2022). Porcentagens de substituição (<30%) e com um tamanho médio de partículas próximo ao do cimento Portland, obtiveram efeitos positivos sobre as propriedades mecânicas de materiais à base de cimento (Kaptan; Cunha; Aguiar, 2024; Oliveira; Dezen; Possan, 2020; Tang *et al.*, 2020; Xiao *et al.*, 2018; Yang; Liu; Ma, 2020). Em uma abordagem mais recente verificou-se, também, a mineralização por carbonatação acelerada, como forma de ao mesmo tempo usar o CO₂, e aumentar a reatividade de pós reciclados de origens cimentícias (Jiang, Y. *et al.*, 2023; Poon *et al.*, 2023; Zhang, T. *et al.*, 2023).

2.2.2 Sistematização dos estudos publicados na temática

A mineralização de CO₂ em pós reciclados de RCD não apenas se alinha com as estratégias de neutralidade de carbono, mas permite a integração entre o uso do CO₂ e o processamento dos resíduos da construção civil (Kravchenko; Besklubova, 2024). Sendo assim, com o intuito de analisar como os estudos vêm sendo conduzidos, as tendências ou perspectivas nessa área, por meio da metodologia multicritério de avaliação de artigos, a Methodi Ordinatio 2.0 (Pagani *et al.*, 2022), realizou-se uma Revisão Sistemática de Literatura considerando os artigos publicados na base Scopus.

Foram utilizadas as seguintes combinações de palavras-chaves e operadores booleanos: (("waste concrete powder" OR "recycled powder" OR "recycled concrete powder" OR "cement paste powder" OR "recycled concrete fines" OR "recycled fine powder") AND ("carbonation" OR "CO₂ treatment" OR "carbonation technology" OR "CO₂ uptake" OR "carbon capture utilization and storage" OR "accelerated carbonation" OR "carbonated" OR "Carbonation" OR "utilizing CO₂")). Com a delimitação de tempo entre 2014 a 2024, foram selecionados artigos científicos de periódicos, excluindo artigos de conferências e capítulos de livros. No total foram encontrados 76 artigos (Apêndice A), que após a leitura dos resumos, foram agrupados em 7 temas principais (Figura 8).

Figura 8- Classificação dos artigos para compor o portfólio



Fonte: Autora (2025).

Considerando que o escopo de trabalho se refere ao uso de pós como MCS e às emissões de carbono associados à mineralização de CO₂ nas partículas, selecionou-se para compor o Portfólio de análise os artigos das temáticas 1 e 5, totalizando 47 artigos (Apêndice B). Os artigos foram analisados em função da ordem de relevância calculada por meio da Equação 8 “InOrdinatio2.0”, considerando o número de citações dos artigos, obtidos pelo Google Scholar em 28 de junho de 2024; e assumido os valores para os indicadores: $\Delta = 10$, $\lambda = 10$ e $\Omega = 5$.

$$\text{InOrdinatio2.0} = \left\{ \left[\Delta * (IF) \right] - \left[\gamma * \left(\frac{\text{Ano da pesquisa} - \text{Ano de publicação}}{\text{vida média de citações}} \right) \right] \right\} + \Omega * \left\{ \frac{Ci}{(\text{Ano da pesquisa} + 1) - \text{Ano de publicação}} \right\}$$

(Equação 8)

Onde:

Δ : é o valor que varia de 0 a 10 que o pesquisador atribuirá à importância das métricas em sua pesquisa. Esse é o indicador que influencia a escolha pela submissão de artigos a periódicos, a preferência por citações, a pontuação em concursos, a avaliação da pós-graduação, a concessão de promoção de um pesquisador, entre outros fatores;

IF: são as métricas do periódico (CiteScore do ano passado ou estimativa do JCR no caso de um CiteScore inexistente). No entanto, apesar de essa métrica de periódico ser sugerida aqui, o pesquisador é capaz de adotar o que melhor se adapta ao seu portfólio, que é o principal objetivo desta versão do InOrdinatio.

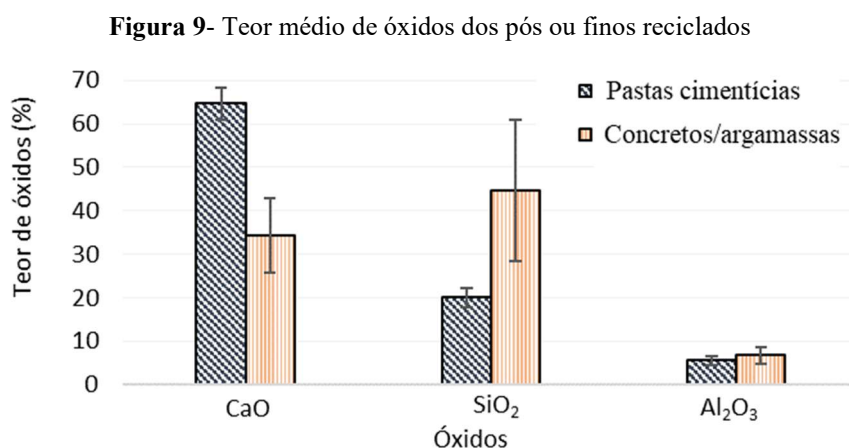
λ : é o valor, variando de 0 a 10, que o pesquisador atribui à importância da atualidade do portfólio em sua pesquisa. Na química, essa letra representa a meia-vida, e foi esse o fator utilizado para ajustar a relevância temporal (ou validade/atualidade) dos artigos. Esse critério é dividido por 7,6, valor baseado no cálculo da mediana da “Cited Half-Life” dos 12.142 periódicos da base de dados Web of Science, conforme a listagem do JCR 2021 (ano- base 2020) que apresenta esse indicador.

Ci: número total de citações encontradas no google Scholar.

Durante a análise do portfólio, foram considerados estudos envolvendo pós reciclados ($\emptyset < 0,15$ mm) e finos reciclados ($0,15$ mm $< \emptyset < 4,75$ mm), provenientes de pastas cimentícias hidratadas, resíduos de concreto ou argamassas.

2.2.2.1 Quanto à origem dos pós e/ou frações finas

Com relação à origem, a maioria dos estudos (71%) empregaram o CO₂ em pós ou frações finas provenientes da moagem de pastas de cimento após um período de hidratação superior a 90 dias. Como pode ser visto na Figura 9, geralmente apresentam valores médios de CaO superiores a 60%. Os demais são obtidos do processamento de concretos, apresentando o valor médio de CaO inferior a 45%, com predominância de SiO₂ devido à presença de agregados naturais (Nedeljković *et al.*, 2021; Rocha; Toledo Filho, 2023).



Fonte: Autora (2025).

Quanto aos teores de CaO e SiO₂, observa-se que a barra de erros nos óxidos provenientes da moagem de pastas de cimento hidratadas é inferior aos provenientes da moagem de concretos/argamassas, justificado pela heterogeneidade dos resíduos analisados. Feng *et al.* (2023), por exemplo, realizaram a moagem de concretos provenientes de pavimentos, Kaliyavaradhan, Li e Ling (2022) de uma usina de reciclagem, enquanto Qian *et al.* (2023) moeram argamassas.

Observou-se que, há uma preocupação em relacionar os resultados obtidos em pós provenientes da moagem de pastas hidratadas de cimento, com os obtidos da moagem de concretos. Por exemplo, Jiang *et al.* (2022) verificaram que apesar da elevada quantidade

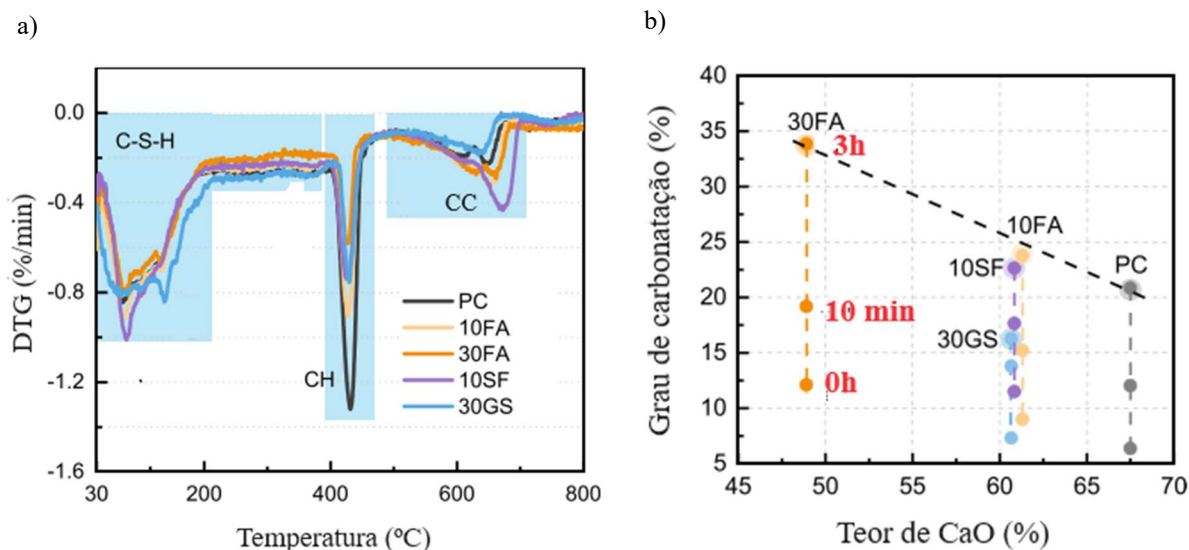
inicial de CaCO_3 na fração 0,60 mm originária de concreto, a fixação de CO_2 seguiu tendência similar para a obtidas de pastas, no entanto, o valor final foi inferior (~15%).

Por sua vez, Jiang, Y. et al. (2023) analisaram o desempenho de argamassas com a substituição de 100% da areia natural por finos reciclados com CO_2 mineralizados. A fração entre 0,3 e 2,36 mm foi obtida da moagem de pastas de cimento hidratadas e concretos, contendo respectivamente 65,70% e 27,50% de CaO, e a mineralização de CO_2 ocorreu em meio aquoso a 25 °C por 10 min, 3 e 6 h. De modo geral, a mineralização com CO_2 dos pós reciclados resultou em uma melhora na resistência à compressão das argamassas aos 28 dias, sendo esse efeito mais pronunciado nos pós provenientes de pastas de cimento. O aumento do tempo de mineralização, especialmente até 6 horas, intensificou essa melhora, com acréscimo na resistência em torno de 15%. Por esse motivo, os autores consideram que os finos obtidos da moagem de pastas de cimento hidratadas podem ser classificados como finos de concreto com elevada quantidade de pasta residual.

Para os pós provenientes de pastas de cimento hidratadas, verificou-se o uso de diferentes tipos de cimentos (CEM I 42,5R, CEM III 42,5, P042,5 e PII52,5, CP I) e relações água/cimento (0,20, 0,35, 0,45, 0,50, 0,55). Zajac *et al.* (2020) por exemplo, utilizaram pós reciclados de pastas hidratadas à base de cimento Portland CEM I (equivalente ao CP I) e cimento de alto-forno (CEM III, equivalente ao CP III). Os autores verificaram que pastas com maior teor de clínquer Portland após a mineralização de CO_2 apresentou um maior teor de carbonato de cálcio. Já aquelas com maior proporção de MCS (CEM III), resultaram em teores mais elevados de silício e alumínio, favorecendo a formação de gel de sílica-alumina.

No estudo de Jiang *et al.* (2024), os autores produziram uma pasta (PC) somente com cimento CEM I 52.5N (equivalente ao CP I, com adição mínima de gesso e resistência à compressão $\geq 52,5$ MPa aos 28 dias, outras quatro pastas contendo 10% e 30% de cinza volante (10FA e 30FA), 10% de sílica ativa (10SF) e 30% de escória de alto-forno granulada e moída (GS) e observaram que o teor de Ca (OH)₂ e a formação de C-S-H foi influenciada pela composição química e pelo teor de MCS presente nas pastas de cimento (Figura 10 a). Por sua vez, após a mineralização de CO_2 por 3 horas (Figura 10 b), observaram que o grau de carbonatação na fração entre 0,3 e 2,36 mm foi dependentes do MCS e da dosagem, e intrinsecamente associados à quantidade inicial de portlandita, à relação Ca/Si de C-S-H, à presença de hidratos de aluminato, bem como as características microestruturais.

Figura 10- Pós reciclados de pastas contendo MCS em substituição ao cimento CEM I 52.5N: a) DTG; b) Grau de carbonatação



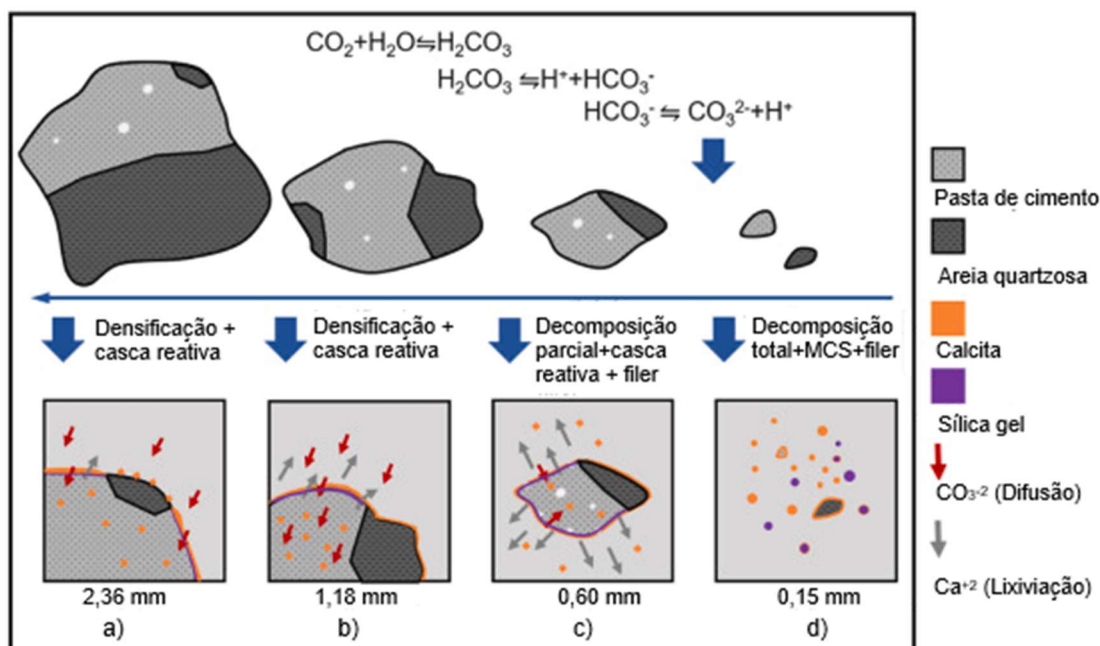
Legenda: Pós obtidos de pastas de cimento hidratadas sendo: PC (100% de CEM I 52.5N); 10FA (10% de cinza volante), 30FA (30% de cinza volante), 10SF (10% de sílica ativa) e 30GS (30% de escória de alto-forno granulada e moída).

Fonte: Traduzido de JIANG *et al.* (2024)

No geral, as investigações realizadas por Ghantous *et al.* (2016); Jiang *et al.*, (2024); Liu, Tang e Wang (2023) e Mehdizadeh *et al.* (2022) confirmaram que o uso de MCS como cinzas volantes, escórias granuladas de alto forno, sílica ativa, vidro residual, entre outros, na produção de pastas de cimento hidratadas, refletem em diferentes relações Ca/Si do gel C- S- H e microestruturas, e consequentemente em distintos comportamentos frente a mineralização de CO₂.

Com relação ao tamanho das partículas, Jiang *et al.* (2022) verificaram que as frações recicladas de concreto (0,6–1,18 mm e 1,18–2,36 mm), com a mineralização aquosa de CO₂ experimentaram dois efeitos positivos, i) melhoria das propriedades da superfície pela formação de uma casca reativa e ii) densificação significativa da microestrutura. Por sua vez, a fração mais fina (<0,15 mm) apresentaram efeitos diferentes, foram totalmente desintegradas e convertidas em um composto de carbonato de cálcio e sílica gel. Tal diferença foi atribuída à longa capacidade de manutenção da alcalinidade e extensa dissolução e lixiviação associadas à alta finura. A fração 0,15–0,6 mm foi considerada um tamanho de partícula de transição onde ocorreu a decomposição e densificação de modo equilibrado. A ilustração do processo de mineralização aquosa de CO₂ em frações recicladas de concreto de diferentes tamanhos pode ser vista na Figura 11.

Figura 11- Mineralização aquosa de CO₂ em frações recicladas de concreto: a) 2,36 mm; b) 1,18 mm; c) 0,60 mm e d) 0,15 mm.



Traduzido de: Jiang *et al.* (2022).

Silva *et al.* (2022) verificaram que a redução de partículas de 0,25 mm para 0,075 mm exerceu um aumento na fixação de CO₂ de cerca de 1,3%. Os autores ressaltam, contudo, que para aplicações em escala industrial, deve-se considerar o aumento dos custos de energia normalmente associados às etapas de moagem e peneiramento. Por outro lado, Teune e Schollbach (2024) afirmam que cada fração granulométrica contribui igualmente para a fixação de CO₂.

Diante do exposto, observou-se que os pós produzidos a partir de pastas de cimento hidratadas apresentam tendência de fixação de CO₂ semelhante à dos pós reciclados de concreto e, devido à menor quantidade de sílica, tendem a fixar uma maior quantidade de CO₂. Estudos com pastas contendo diferentes tipos de cimento, relações a/c e materiais cimentícios suplementares (como sílica ativa, escória de alto-forno e cinzas volantes) também foram abordados, evidenciando a preocupação dos autores com os desafios relacionados à heterogeneidade química dos pós reciclados de concreto, tanto em relação à fixação de CO₂ quanto ao seu desempenho como MCS.

2.2.2.2 Quanto ao método e parâmetros da mineralização de CO₂

Com relação ao método de mineralização de CO₂, 30 trabalhos (Apêndice B) utilizaram a câmara de carbonatação (G/S), sendo que 3 deles compararam com a carbonatação aquosa (L/S). Os parâmetros de mineralização de CO₂ não seguem uma tendência, o que dificulta a comparação entre os resultados, por exemplo, a temperatura variou de 20 a 180°C, sendo majoritariamente adotado 20°C, a umidade relativa (UR) de 5 a 100%, sendo comum entre 60 e 70% (UR). O valor da concentração de CO₂ para a maioria dos trabalhos foi de 20% de CO₂. Porém, alguns estudos adotaram menos de 5% (Wei *et al.*, 2022), e valores entre 25 e 80% de CO₂ (Silva *et al.*, 2022). Para o tempo de exposição ao CO₂, houve variação entre 5 minutos a 3 meses, enquanto para a pressão, nos trabalhos que informaram, a variação foi de 1 bar a 40 bar.

Um dos grandes desafios na carbonatação G/S é determinar a relação água/sólidos (a/s) ideal para garantir a dissolução e difusão do CO₂ nas partículas dos pós de RCD. Nesse sentido, Kaliyavaradhan, Li e Liang (2022) verificaram em seu estudo que a relação a/s=0,40 com a mineralização de CO₂ em pós reciclados de concreto por 90 h pode ser considerada uma condição ótima, entre a fixação de CO₂ e a energia consumida no processo. Por sua vez, Silva *et al.* (2022) estudaram o teor de umidade dos pós reciclados de pastas cimentícias contendo 5 a 30% de água. Com 1h de exposição e 80% de CO₂, os pós contendo 19% de teor de água tiveram uma fixação de CO₂ de 27%, para 6% de umidade e 29% de umidade os valores foram respectivamente de 6% e 10%.

Jin *et al.* (2024) investigaram 3 métodos de preparação dos pós reciclados de pastas de cimento hidratadas, a saber: seco, semi-seco com impregnação de água (a/s=0,1) e semi-seco com impregnação de uma solução contendo soda cáustica (0,1 mol L⁻¹ NaOH). Após a mineralização de CO₂, os autores verificaram um grau de carbonatação de 21% nos pós secos. O método semi-seco com soda cáustica demonstrou maior eficiência de carbonatação do que o método semi-seco com impregnação de água, com grau de carbonatação de 30% e 26% respectivamente. Os resultados corroboram com os achados de Kaliyavaradhan; Li; Ling (2022) que identificaram que a relação água/sólidos é um parâmetro crítico para a fixação de CO₂ durante a mineralização de pós reciclados de concreto.

Outro método verificado durante a RSL foi o uso da carbonatação mecanoquímica (no inglês, *Mechanochemical Carbonation* – MC) que envolve a mineralização de CO₂ em reatores dinâmicos na presença de água e esferas de aço. Em comparação com o método aquoso, os pós reciclados de pastas de cimento hidratadas apresentaram maior teor de

géis de sílica, atribuído ao aumento da área superficial resultante da moagem e da redução do tamanho das partículas, além de exibirem maior reatividade frente à mineralização de CO₂.

2.2.2.3 Quanto ao emprego dos pós com CO₂ mineralizados

O uso de pós com CO₂ mineralizado (parcialmente ou totalmente carbonatados) são usados em pastas e argamassas substituindo até 30% da massa do cimento Portland, sendo recomendado entre 10 e 20%, para combater os efeitos adversos sobre as propriedades no estado fresco (Pan *et al.*, 2023; Tang *et al.*, 2023; Wu *et al.*, 2021) e garantir a durabilidade quando usados em estruturas de concreto armado (Pan *et al.*, 2023).

A camada de sílica formada na partícula após a mineralização de CO₂ tem uma forte afinidade por água (H₂O), reduzindo tempo de pega e a fluidez, e conseqüentemente diminuindo o desempenho mecânico (Mao *et al.*, 2024; Ouyang *et al.*, 2020). O uso da Monoetanolamina (MEA) mostrou-se uma abordagem eficaz para mitigar o efeito adverso sobre as propriedades reológicas da pasta de cimento, ao invés do uso de superplastificante, ajuste de água, entre outras abordagens usadas na literatura, no entanto o custo-benefício precisa ser avaliado (Mao *et al.*, 2024).

Outro efeito o uso de pós reciclados após a mineralização do CO₂ é acelerar a cinética de hidratação em idades iniciais, os géis de sílica amorfa e sílica-alumina e a calcita presentes nas partículas facilitam a dissolução inicial dos íons e fornecem sítios de nucleação ativos adicionais para hidratos. Como resultado, a microestrutura é densificada pelos géis adicionais de C-S-H com altas relações Si/Ca e Al/Ca e fases carboaluminato, juntamente com o efeito de preenchimento inerte de partículas de calcita não reagidas (Peng *et al.*, 2023).

Em materiais como cimentos LC³ (Jin *et al.*, 2024), tijolos (Ouyang *et al.*, 2024) e concretos botânicos, um compósito prensado a quente de três componentes: pó de madeira, pó de concreto e pó de lignina kraft (Wei *et al.*, 2022) foram empregados até 50% de pós reciclados em substituição ao cimento Portland, com performance superior aos pós que não passaram pelo processo de mineralização de CO₂.

2.2.2.4 Quanto à avaliação da pegada de carbono

Uma das justificativas para a mineralização de CO₂ em pós reciclados de

RCD é a fixação permanentemente do CO₂ nas partículas. No entanto, durante a análise do portfólio (Apêndice B) observou-se que poucos autores quantificam a fixação de CO₂ por tonelada do material.

Mehdizadeh, Mo e Ling (2023) a 25 °C, pressão de injeção de 0,025 MPa, usando cloreto de amônio (NH₄Cl) na dosagem de 1 mol/L e um tempo de reação de 30 min, obtiveram em pós de pastas de cimento hidratadas, um grau de carbonatação de 91,4%, ou seja, 1 kg de pó poderia fixar 270 g de CO₂ (270 kg.CO₂/t).

Peng *et al.* (2023) estimaram que 1kg de pós de pastas de cimento hidratadas podem sequestrar aproximadamente 0,256 kg de CO₂ (256 kg.CO₂/t) após o tratamento de carbonatação L/S, 99,9% de CO₂, vazão de 0,5 L/min, 25°C por 120 minutos.

Bui *et al.* (2023), por meio da carbonatação aquosa com agitação (1400 rpm), vazão do gás 0,1 ml/min, taxas de fluxo de ar para a solução fornecida pela bomba de ar foi de 1 L/min e concentração de 5% de CO₂, verificaram a fixação de 11,75 g de CO₂/100 g de pó de pasta de cimento hidratada após 120 horas (117,5 kg.CO₂/t).

Com relação às emissões de CO₂ associadas à mineralização de CO₂, Tang *et al.* (2023) verificaram o Índice de Carbono (no inglês, *Carbon Index* -CI), que é a relação entre emissões de CO₂ pela resistência à compressão das argamassas. No entanto, foram levados em consideração dados da literatura acerca das emissões associadas à moagem em laboratório (0,188–0,2457 kg/kg de pó) e de fixação de CO₂ nas partículas (0,09-0,17 kg de CO₂/kg de pó). Os autores verificaram que com o uso de finos e pós carbonatados em diferentes teores pode-se reduzir em cerca de 2,0–10,8% das emissões de carbono por m³ de argamassa a depender do material usado e dos teores de substituição.

Liu, Tang e Wang (2023) com o uso de pós reciclados de pastas cimentícias contendo MCS (escória granulada moída de alto-forno, cinzas volantes e pó de vidro residual reciclado) verificaram que a mineralização de CO₂ em pós reciclados de pastas cimentícias reduziu o índice de carbono das argamassas de 8 kg.CO₂/m³/MPa para valores entre 5 e 6,3 kg.CO₂/m³/MPa, conforme teor de substituição do cimento e do MCS contido na pasta de origem.

Em uma análise mais ampla, considerando a metodologia de Avaliação do Ciclo de Vida (ACV), Kravhenko *et al.* (2024) verificaram que a mineralização de CO₂ em pós reciclados de concreto e uso como substituto parcial do cimento não teve um impacto significativo na redução das emissões de CO₂, e a análise de sensibilidade revela que a pegada de CO₂ é fortemente influenciada pelo energia elétrica gasta no processo, discordando dos resultados de Mo *et al.* (2023).

Kravchenko e Besklubova (2024) avaliaram as emissões de CO₂ levando em consideração o transporte dos agregados e pós reciclados até chaminés, e verificaram que há uma redução significativa no custo ambiental de mais de 51%, atribuída principalmente à diminuição da dependência de CO₂ de origem comercial.

Diante da ampla variabilidade nos materiais de origem e nos protocolos de mineralização de CO₂ adotados nos estudos revisados, a falta de uniformização compromete a comparação direta entre os resultados de fixação de CO₂. Essa heterogeneidade limita o avanço no entendimento técnico-científico e, principalmente, dificulta a consolidação de diretrizes para aplicação prática desses materiais no mercado de carbono. Assim, torna-se urgente o desenvolvimento de estudos que utilizem resíduos reais e representativos do setor da construção civil, com foco em metodologias reproduzíveis e adaptáveis a condições industriais.

2.2.2.5 Tendências e perspectivas

Diante da análise dos 47 artigos pode-se afirmar que otimizar os parâmetros para maximizar a fixação de CO₂ o em finos e pós reciclados de pastas cimentícias e avaliar o desempenho em pastas e argamassas são as principais tendências dos estudos.

Com relação às perspectivas, considera-se a necessidade de:

- i) ampliar os estudos laboratoriais usando pós reciclados provenientes da reciclagem de RCD, visando um protocolo unificado para a mineralização de CO₂;
- ii) investigar o uso de pós reciclados de concreto após o processo de mineralização de CO₂, em matrizes cimentícias frente ao desempenho e a durabilidade;
- iii) extrapolar o processo de mineralização de CO₂ em escala laboratorial para escala industrial, com uso de gases industriais;
- iv) analisar as emissões de CO₂ considerando as emissões associadas à mineralização de CO₂ em partículas recicladas de RCD.

3 METODOLOGIA E RESULTADOS

A pesquisa foi estruturada em cinco artigos. O primeiro aborda o protocolo de moagem dos RCD; o segundo utiliza a análise termogravimétrica (TGA) para avaliar a absorção de água das partículas dos pós reciclados de RCD, estudos que envolveram o preparo e a caracterização das partículas. Os três últimos artigos investigaram a fixação de CO₂ através da mineralização por carbonatação acelerada em pós reciclados de RCD, com ênfase em sua aplicação como MCS.

Os artigos 1, 2 e 3 foram desenvolvidos em paralelo usando o mesmo lote de RCD (tipo misto e de concreto), enquanto os Artigos 4 e 5 foram elaborados a partir dos resultados obtidos nos estudos anteriores para um novo lote, somente com RCD de concreto. Ressalta-se que, para cada artigo, devem ser analisados os protocolos de caracterização adotados, considerando suas especificidades metodológicas e materiais empregados.

3.1 ARTIGO 1 (MOAGEM)

O artigo 1 intitulado *Melhoria das características físicas do pó reciclado de concreto por meio de mecanoativação com e sem auxiliares de moagem* foi publicado na revista Ambiente Construído, da editora Scielo. O artigo teve como objetivo definir o protocolo de moagem de pós reciclados de concreto (RCP) em moinho de bolas planetário PM 100 (500 rpm) com base nas alterações do diâmetro médio das partículas (d₅₀) e na área superficial – BET (Brunauer, Emmett, Teller), contribuindo parcialmente para a resposta do objetivo (i) da tese.

Os RCPs foram moídos por 30, 60, 120, 180 e 240 minutos em jarro de ágata de 250 cm³, seguindo o protocolo: enchimento com 60 gramas de pó, 50 bolas de ágata com aproximadamente 10 mm de diâmetro, utilizando um aditivo no pó e reversão automática do sentido de rotação a cada 15 minutos com parada de 1 minuto para resfriamento. Foram testados três tipos de auxiliares de moagem (Gas): propilenoglicol (PG), sódio hexametáfosfato (SHMP) e trietanolamina (TEA), em diferentes níveis (0; 0,05; 0,1; 0,5 e 1%). O desempenho dos pós como MCS (substituição de 10%) foi avaliado em pastas quanto à consistência e resistência à compressão aos 7 dias. O estudo foi realizado em corpos de prova com dimensões reduzidas (Ø25 mm e h50 mm), conforme procedimentos de mistura e moldagem realizados por Oliveira (2022). De acordo com o teste estatístico realizado pela autora, essas alterações não

influenciaram significativamente a resistência à compressão, quando comparados aos resultados das argamassas moldadas conforme NBR 7215 (ABNT, 2019).

Considerando o menor tempo e o menor teor de auxiliar de moagem o processamento por 30 minutos com 0,5% de aditivo propilenoglicol (PG) reduziu o d50 de 20,99 para 8,85 μm e aumentou a área superficial de 6,23 para 7,50 m^2/g , sem alterar a cristalinidade e com impacto na demanda de água quando usado como MCS em pastas cimentícias. Os resultados em pastas indicaram que o uso dos auxiliares de moagem favoreceu o emprego dos pós reciclado de concreto como MSC, com influência estatística significativa na resistência à compressão.

O artigo tem como forma de citação: RISSON, Kathleen Dall Bello de Souza et al. Physical characteristics improvement of recycled concrete powder by mechanoactivation with and without grinding aid. **Ambiente Construído**, v. 25, p. e139436, 2025, e está apresentado na íntegra a seguir.

Physical characteristics improvement of recycled concrete powder by mechanoactivation with and without grinding aid

Melhoria das características físicas do pó reciclado de concreto por meio de mecanoativação com e sem auxiliares de moagem

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Abstract

This study aimed to evaluate the effect of mechanoactivation with and without grinding aids (GAs) on the physical characteristics of recycled concrete powder (RCP) for use as supplementary cementitious material (MCS). RCP was subjected to the comminution process in a planetary ball mill for 30, 60, 120, 180, and 240 minutes, using three types of grinding aids (propylene glycol (PG), sodium hexametaphosphate (SHMP) and triethanolamine (TEA)), at different levels (0, 0.05, 0.1, 0.5 and 1%). The powders' performance as SCM (10% substitution) was evaluated in pastes on consistency and compressive strength at 7 days. Considering the shortest milling time and the lowest content of milling aid, mechanoactivation for 30 minutes with 0.5% propylene glycol (PG) additive reduced the 50 from 20.99 to 8.85 μm and increased the BET from 6.23 to 7.50 m^2/g , without altering crystallinity. The results in pastes indicated that the use of grinding aids favored the use of recycled concrete powder as MSC, with a statistically significant score in particularity resistance.

Keywords: *Chemical additives. Grindability index. Milling. Supplementary cementitious materials.*

Resumo

Este estudo teve como objetivo avaliar o efeito da mecanoativação com e sem auxiliares de moagem (GAs) nas características físicas do pó de concreto reciclado (RCP) para uso como material cimentício suplementar (MCS). O RCP foi submetido ao processo de mecanoativação em moinho de bolas planetário por 30, 60, 120, 180 e 240 minutos, utilizando três tipos de GAs (propilenoglicol (PG), sódio hexametáfosfato (SHMP) e trietanolamina (TEA)), em diferentes níveis (0, 0.05, 0.1, 0.5 e 1%). O desempenho dos pós como SCM (substituição de 10%) foi avaliado em pastas quanto à consistência e resistência à compressão aos 7 dias. Considerando o menor tempo de moagem e o menor teor de auxiliar de moagem, a mecanoativação por 30 minutos com 0.5% de aditivo propilenoglicol (PG) reduziu o d_{50} de 20.99 para 8.85 μm e aumentou a SSA de 6.23 para 7.50 m^2/g , sem alterar a cristalinidade. Os resultados em pastas indicaram que o uso dos auxiliares de moagem favoreceu o emprego do pó reciclado de concreto como MSC, com influência estatística significativa na resistência à compressão.

Palavras-chave: *Aditivos químicos. Índice de moabilidade. Moagem. Materiais cimentícios suplementares.*

Introduction

Around 100 billion tons of construction and demolition waste (CDW) are generated globally. It is estimated that by 2060, the demand for materials such as steel, concrete, and cement, the main contributors to greenhouse gas emissions, will be double that of today (UNEP, 2022), indicating that the construction industry needs immediate measures to decarbonize the sector by 2050 (GCCA, 2021). To boost sustainability in the construction chain, it is essential to adopt the circular economy model, making it possible to reduce the consumption of natural resources, thus minimizing waste and greenhouse gas emissions (Benachio; Freitas; Tavares, 2020).

In this perspective, CDW may be processed and used as coarse and fine aggregate in producing mortars and concretes, with its use regulated in several countries (Kenai, 2018) and Brazil (ABNT, 2021). However, fine particles are generated (<0.15 mm) during the production process of these aggregates (Martin *et al.*, 2023; Quattrone; Angulo; John, 2014). Around 15 to 19% of the processed material corresponds to CDW dust (Oliveira; Dezen; Possan, 2020; Zhang *et al.*; 2022), which, as it has no commercial value, becomes a liability for recycling companies (Lampris; Lupo; Cheeseman, 2009).

The RCP contains CaCO_3 , SiO_2 , AFt (ettringite), AFm (monosulfate), CH (portlandite), C-S-H (hydrated calcium silicate) and non-hydrated cement particles (anhydrous) (Oliveira *et al.*, 2023; Wang *et al.*; 2024), it has low reactivity and high Absorption (Duan *et al.*, 2020a; Mehdizadeh *et al.*, 2021), acting as a filler material (Tan *et al.*, 2020), where the particles fill the space between the cement grains, modifying their granular packing, which implies a change in the initial porosity of the paste (Irassar *et al.*, 2015), and which, depending on the proportion and granulometry, can densify the microstructure, resulting in better mechanical performance (Awoyera; Adesina; Gobinath, 2019; Oliveira *et al.*, 2024).

In recent years, the literature has shown that recycled concrete powder (RCP) may be used as supplementary cementitious materials (SCMs) (Aquino Rocha; Toledo Filho, 2023; Martin *et al.*, 2023; Tang *et al.*, 2020; Tokareva; Kaassamani; Waldmann, 2023), which, in addition to acting as a raw material with added value, may reduce the emissions associated with Portland cement production (Scrivener; John; Gartner, 2018).

Some treatments such as thermal activation (Chen *et al.*, 2024; Vashistha *et al.*, 2023), carbonation (Mehdizadeh *et al.*, 2021; Ouyang *et al.*, 2020; Qin; Gao, 2019), and mechanoactivation are used to improve the characteristics of these particles. Mechanoactivation occurs by comminution (grinding), which can modify the crystalline structure, provided by the higher SiO_2 content and lower CaO content, reducing the particle size and increasing the specific surface area (SSA), culminating in greater reactivity of the RCP due to the exposure of the anhydrous Portland cement grains (Bu *et al.*, 2023; Oliveira *et al.*, 2023; Zhang *et al.*, 2022).

Although particle size decreases with increasing grinding time, there is an exponential reduction in the grinding efficiency of recycled CDW powders with increasing time (Aquino Rocha; Toledo Filho, 2023; Sun *et al.*, 2021; Xu *et al.*, 2021), justified by the tendency to aggregation due to Van der Waals forces (interparticle attraction). The magnitude of these forces increases during dry mechanoactivation (Bernardes, 2006) as the electric charges on the fractured surfaces are particularly negative in C_3S and C_2S crystals and positive in C_3A and C_4AF crystals (Aïtcin; Mindess, 2011).

In this way, to improve the efficiency of milling processes, producing greater fineness with lower energy consumption and consequently lower carbon dioxide emissions (Prziwara; Kwade, 2020; Zan; Ishak, 2023), the forces of repulsion between the particles must outweigh the forces of attraction (De Castro; Pandolfelli, 2009). Grinding aids may be used during this process, the most common being amines, glycols, alcohols, and phenols (Engelsen, 2009; Katsioti *et al.*, 2009; Toprak; Altun; Benzer, 2018).

High-polarity grinding aid groups (-COOR, $-\text{NH}_2$, -OH, $-\text{SO}_3$) are generally composed of long-chain, electrically charged organic molecules that prevent agglomeration, causing a propensity for adsorption on electrostatic surfaces formed by breaking the covalent bonds of Al-O, Si-O, and Ca-O (Collepari, 2005; Toprak; Altun; Benzer, 2018). The effect of using a grinding aid on a clinker is shown in Figure 1.

The concentration range of grinding aids in cement production is between 0.01 and 0.1%, and up to 0.5% may be used (Jeknavorian; Barry; Serafin, 1998; Teoreanu; Guslicov, 1999). The most commonly used grinding aids are propylene glycol (PG), triethanolamine (TEA), triethanolamine acetate, and polyglycol phenol ether (Assaad; Vachon, 2021; Engelsen, 2009; Katsioti *et al.*, 2009; Toprak; Altun; Benzer, 2018). Sodium hexametaphosphate (SHMP) is also used as a particle dispersant (Rodrigues; Evangelista; Brito, 2013).

In the search for process efficiency (increased production capacity, reduced specific energy consumption, or finer particles), several studies are carried out related to the grinding of raw materials such as quartz, feldspar, limestone and calcite, and the performance of the final product. (Portland cement) by controlling the specific surface area (SSA) using the Blaine method (Cayirli *et al.*, 2023; Katsioti *et al.*, 2009; Kaya *et al.*, 2023; Prziwara; Kwade,

2020). However, the grinding aid is commonly adopted in studies with recycled powder materials, without prior compatibility or efficiency studies.

In this sense, considering that, depending on the water/cement ratio, the content of the C3A phase ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$), as well as the amount of sulfates (the increase in the sulfate content reduces the adsorption of the additive if it is used during grinding) (Kapeluszna; Kotwica, 2022; Kujawa; Olewnik-Kruszkowska; Nowaczyk, 2021) and that the Blaine method does not take into account the difference between the particle size distribution curves of the powders (Assaad; Vachon, 2021; Delagrammatikas; Tsimas, 2004; Ferraris *et al.*, 2002), this study aimed to evaluate the effect of mechanoactivation in the presence of grinding aids on the physical characteristics (d50 and SSA-BET) of recycled concrete powder for use as a supplementary cementitious material.

Materials and methods

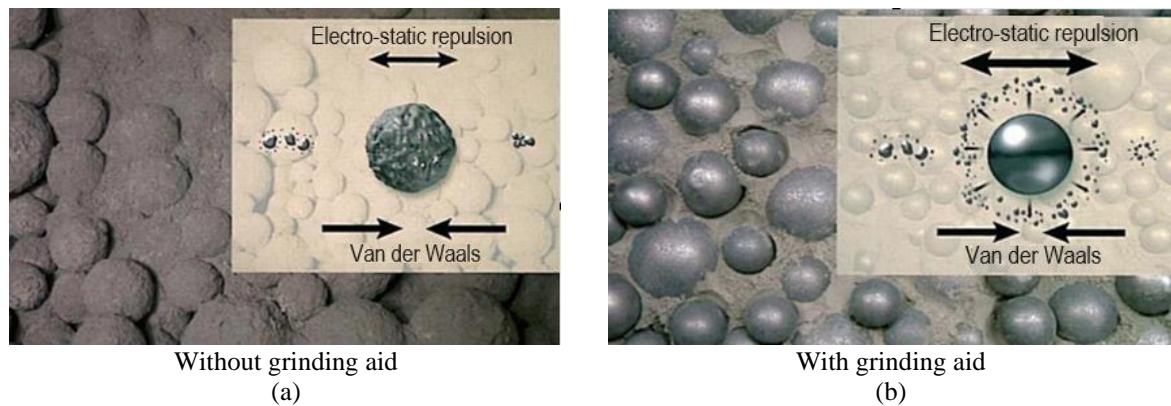
Materials

The recycled concrete powder (RCP) used in this study originated from the comminution process of concrete specimens with different compositions supplied by a concrete and precast plant. During this process, the Los Angeles Abrasion machine was filled with 30 kg of material and used 24 stainless steel balls with a 48 mm diameter and a mass ranging between 390 and 445 g, and a 30 rpm speed was set for two hours. The fraction obtained was sieved, and the particles that passed the Mesh 100 sieve (<0.15 mm) were called RCP. Table 1 shows the raw materials' chemical composition obtained by X-ray fluorescence spectrometry (XRF) using the Rigaku model ZSX Primus IV instrument equipped with a Rh tube.

It was found that the main oxide present was silica (SiO_2). Its origin is mainly associated with the natural aggregates present in the concrete and secondarily with calcium oxide (CaO), which is related to the binder (hardened cement paste, lime, gypsum, etc.) (Ulsen *et al.*, 2010; Vashistha *et al.*, 2023; Wang *et al.*, 2022). The chemical requirements were compatible with those established by NBR 12653 (ABNT, 2014) for use as a pozzolanic material.

Propylene glycol (PG), sodium hexametaphosphate (SHMP), and triethanolamine (TEA) are commonly used in the literature for grinding cement (Chipakwe *et al.*; Nthiga Njiru *et al.*, 2023), whose characteristics are shown in Table 2, were used as grinding aids (GAs).

Figure 1 - Clinker grinding principle



Source: Engelsens (2009).

Table 1 - RCP Oxide composition

Powder	Chemical composition (%)										
	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	SO ₃	Na ₂ O	MnO	P ₂ O ₅	LOI
RCP	47.20	21.36	8.56	6.94	3.28	2.14	0.62	1.36	0.13	0.22	7.84

Note: LOI: Loss on ignition; and <LQ: below the quantifiable limit.

Grinding study

The recycled concrete powder (RCP) was mechanoactivated in a PM 100 planetary ball mill (Retsch), equipped with a 250 cm³ agate grinding jar, rotating at 500 rpm following the protocol: filling with 60 grams of powder, 50 agate balls with approximately 10 mm diameter, using an additive on the powder (Prziwara *et al.*, 2018) and automated rotation direction reversal every 15 minutes with a 1-minute stop to cool down.

Propylene glycol was used firstly (1st stage) as grinding aid (GA) (Costa; Gonçalves, 2022) at 0.05 and 0.1% of the RCP mass, quantities provided in the literature for cement grinding (Chipakwe *et al.*, 2020; Nthiga Njiru *et al.*, 2023), and grinding was carried out at 60, 120, 180 and 240 minutes. However, powder agglomeration was observed as a reduction on the specific surface area (SSA) occurred.

For this reason, two other grinding aids were included (2nd stage): Sodium hexametaphosphate (Flores *et al.*, 2017) at a 12.5 g/l concentration and Triethanolamine (Katsioti *et al.*, 2009). The contents were increased to 0.5 and 1% of the RCP mass, and the grinding time was reduced to 30 and 60 minutes, according to the experimental design shown in Figure 2.

The RCPs from this study were characterized by mean particle size (d₅₀) and specific surface area (SSA). The powders' granulometry was assessed by analyzing the particle size distribution by laser diffraction using a granulometer (CILAS 1190) for the grain reading ranging between 0.04 and 2500 µm, in a liquid medium (distilled water), without the presence of a dispersant, and according to the Fraunhofer analysis method, with an approximately 25% obscuration. The methodology used to evaluate the Specific Surface Area (SSA) was nitrogen adsorption (BET - Brunauer-Emmett-Teller) in Quantachrome equipment, NOVA 3200 model. Before the test, the samples were degassed (using a vacuum at 40 °C for 16 hours) (Scrivener *et al.*, 2016).

To analyze the grinding process' efficiency, in the 2nd stage the RCP's grindability index (K) was calculated based on the specific surface area (cm²/g) generated and the energy consumed for 1 ton of material (kWh/t) ratio (Costa; Gonçalves, 2022; Katsioti *et al.*, 2009; Von Krüger, 2004), according to Equation 1. The planetary mill's power for this calculation was 750W.

$$K = \frac{SSA}{E} \quad \text{Eq. 1}$$

Where:

K: Grindability Index given in (cm²/g)/ (kWh/t);

SSA: specific surface area (BET) in cm²/g; and

E: Energy consumed in Wh to generate 1 ton of recycled concrete powder.

Table 2 - Grinding aids characteristics

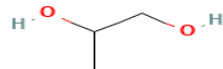
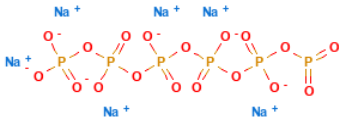
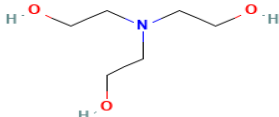
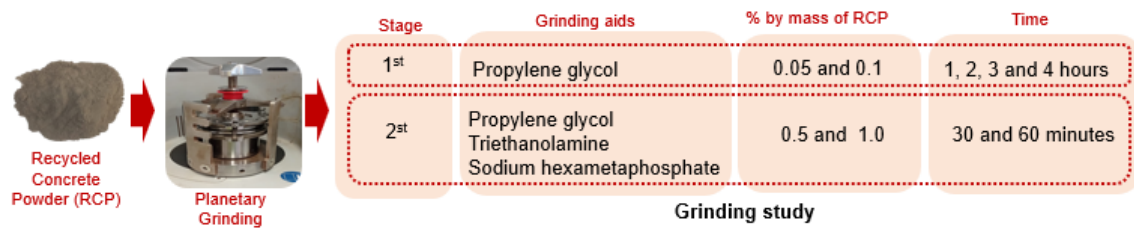
Type	Molecular Formula	Structure (2D)	Density (g/cm ³)	Ph (25 °C)	Water solubility (20 °C)	Reference
Propylene glycol (PG)	C ₃ H ₈ O ₂		1.04	7	Soluble	Manufacturer
Sodium hexametaphosphate (SHMP)	(NaPO ₃) ₆		2.20	6.5	Soluble	Manufacturer
Triethanolamine (TEA)	C ₆ H ₁₅ NO ₃		1.12	10.6	Soluble	Manufacturer

Figure 2 - Experimental design for the grinding study



Potential use of recycled mechanoactivated concrete powders as SCM

Given the time and additive content chosen in the grinding study, the following powders (Table 3) were selected for the study of their potential use as SCM.

Initially, the powders were characterized according to the tests described below and compared with CPV ARI, a high initial strength Portland cement equivalent to Portland cement type III (ASTM, 2016). CPV ARI was used because it is the one commercially available with the lowest addition content (<10% carbonate material) (ABNT, 2018). The specific mass was determined using a gas pycnometer (He), Ultrapyc 5000 model, from Anton Paar, with a 10 psi operating pressure.

The pH was assessed using a digital bench pH meter in a solution containing around 10 ± 0.1 g of each powder/CPVARI and 10 ± 0.1 g of distilled water, kept in a glass beaker and left to stand for one hour (shaken every 10 to 15 minutes) to stabilize the Ph (GEB, 2015).

Thermogravimetric analysis (TGA) and X-ray diffraction (XRD) verified the elemental and mineralogical composition. Thermogravimetric analysis was carried out using TA Instruments equipment, model SDT Q600, an alumina crucible with a sample mass of around 10 mg, a $30 \text{ ml} \cdot \text{min}^{-1}$ nitrogen flow, a $100 \text{ }^\circ\text{C}$ to $900 \text{ }^\circ\text{C}$ heating interval, and a $20 \text{ }^\circ\text{C} \cdot \text{min}^{-1}$ equipment heating rate.

X-ray diffraction (XRD) was used to identify the RCP mineralogical composition after grinding. The samples were analyzed in a Panalytical diffractometer with $\text{Cu K}\alpha \lambda = 1.54$ incident radiation operating at $40 \text{ kV} / 20 \text{ mA}$ and a fixed divergent slit. The measuring range was from 5° to 100° , with an angular step of 0.026° (2θ) and 247 seconds per step. The diffraction patterns and Rietveld refinement were analyzed with Panalytical X-Pert HighScore Plus software (version 4.9) with Crystallography Open Database and Inorganic Crystal Structure Database (ICSD) files used to quantify the mineralogical phases using the following refinement parameters: scale factor, zero shift error, unit cell and peak shape parameters (W, V and U) using the pseudo-Voigt function. Finally, using the spherical harmonic function, the preferred orientation was refined for compounds that tend to orient themselves along the same crystallographic plane.

Pastes were produced with a water/fines ratio of 0.48 and replacement of 10% of Portland cement - CPV ARI with recycled concrete powders to evaluate consistency and compressive strength at 7 days, following the recommendations of standard NBR 7215 (ABNT, 2019). The materials were mixed in a mechanical mixer with a 1600 rpm rotation speed for 2 minutes (Figure 3a), and the consistency was evaluated by the mini-slump test (Kantro, 1980) (Figure 3b). Six specimens with 25 mm (diameter) and 50 mm (height) were molded (Figure 3c), unmolded, and placed in submerged curing saturated in lime (3g/l) until the test ages. On the scheduled date, the specimens were ground and broken using a Contenco I-3025-B hydraulic press with a 100-ton capacity (Figure 3d).

Results and discussions

Grinding study

The mechanoactivation effect on RCP's d_{50} and SSA-BET with propylene glycol grinding aid in the 1st stage is seen in Figure 4.

As seen in Figure 4a, mechanoactivation reduced the average particle size of RCP ($d_{50}=20.99 \mu\text{m}$) as the grinding time increased, with values ranging from 5 to $12 \mu\text{m}$, with the greatest particle reduction observed between 1h and 2 h using 0.05% propylene glycol. Overall, a d_{50} close to or smaller than that of Portland cement ($17.83 \mu\text{m}$) has positive effects on the cementitious materials' mechanical properties (Aquino Rocha; Toledo Filho, 2023; Horsakulthai, 2021) and generally the finer it is, the more reactive it is, as the increase in specific surface area increases the reaction area and the availability of a high number of active atoms that easily bond to other atoms (Duan *et al.*, 2020b; Tang *et al.*, 2020). Figure 4b showed that although the grinding aid favored the SSA-BET,

even when milled for 4 hours, all values were lower than the RCP (6.23 m²/g), suggesting that the material agglomerated and the propylene glycol content used was inefficient.

Table 3 - Application of powders as SCM

Acronym	Description
RCP	Recycled concrete powder (Mesh 100)
RCP-30 min- No GAs	Recycled concrete powder mechanized for 30 minutes without grinding aids
RCP-30 min- 0.5% PG	Recycled concrete powder mechanized for 30 minutes with 0.5% Propylene glycol (PG)
RCP-30 min- 0.5% SHMP	Recycled concrete powder mechanized for 30 minutes with 0.5% Sodium hexametaphosphate (SHMP)
RCP-30 min- 0.5% TEA	Recycled concrete powder mechanized for 30 minutes with 0.5% Triethanolamine (TEA)

Figure 3 - Production and evaluation of pastes containing recycled concrete powders

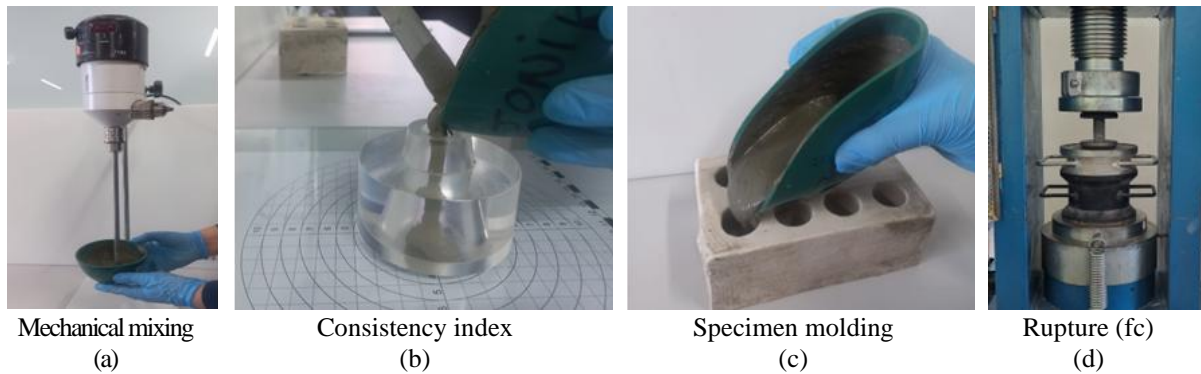
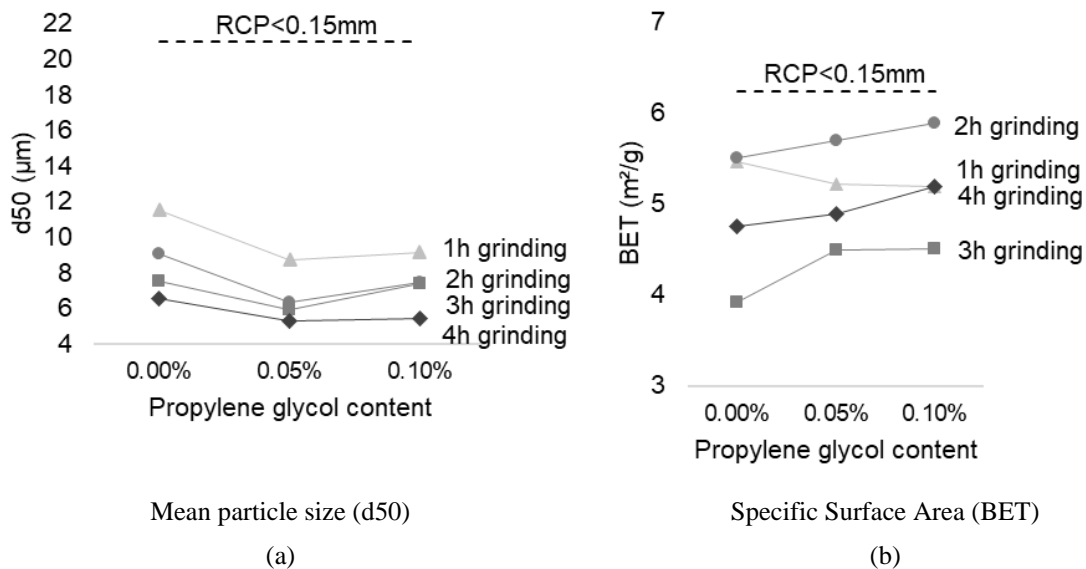
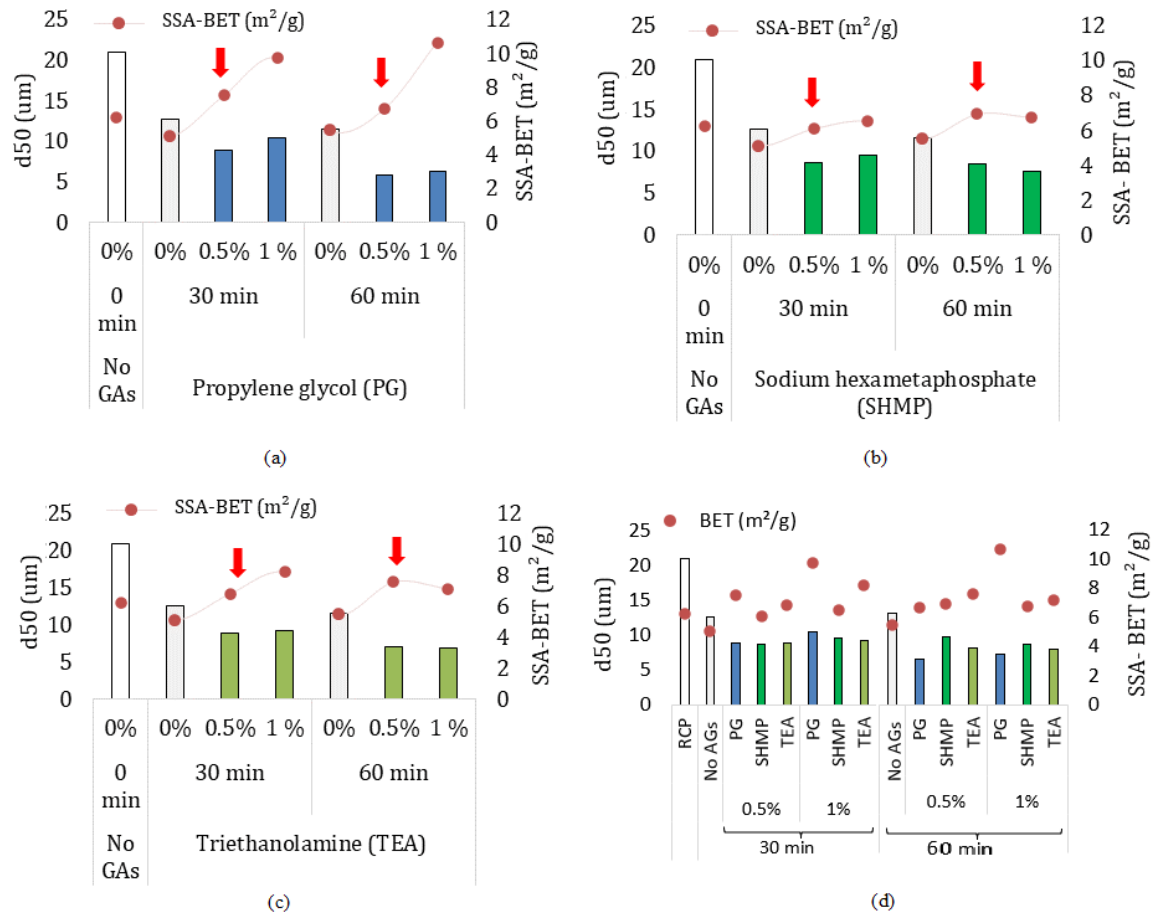


Figure 4 - Mechanoactivation effect on RCP in the 1st grinding stage



Therefore, to reduce energy consumption during grinding and ensure a higher SSA-BET than the RCP, higher levels of grinding aids (0.5 and 1%) and shorter grinding times (30 and 60 minutes) were evaluated. Two other grinding aids were also included (Sodium Hexametaphosphate and Triethalonamine), and the results for d50 and SSA-BET for the milled powders in the grinding study 2nd stage are shown in Figure 5 (5a, 5b, and 5c).

Figure 5 - Mechanoactivation effect on RCP in the 2nd grinding stage

Figures 5 (5a, 5b, and 5c) showed that for all the additives with a 0.5% GA content and grinding for 30 and 60 minutes, there was a simultaneous d50 reduction and an SSA-BET increase when compared to RCP (20.99 µm and 6.23 m²/g). There was no trend for the grinding aid content (Figure 5d), i.e., the d50 and SSA-BET characteristics depend on the combination of mechanoactivation time and GA content. It was noted that the contents and GAs used in the 2nd stage contributed to particle dispersion and greater efficiency in the mechanoactivation process, given the higher SSA-BET values compared to powders without GAs. To complement this analysis, Figure 6 shows the grindability indices for 1 ton of recycled concrete powder, considering different mechanoactivation times, contents, and GA types.

It can be seen that for the two mechanoactivation times with the use of grinding aids, the process was efficient, with values higher than 8.13 cm²/g/ (kWh/t) for the 30-minute period and 4.37 cm²/g/ (kWh/t) for the 60-minute period. Taking into account that the higher the grindability index, the greater the process efficiency, the best results were obtained with 1% PG/30 min < 1% TEA/30 min and 0.5% PG/30 min. Other scenarios may be evaluated to improve the powders' grindability index, such as using more balls or changing the mechanoactivation equipment. However, energy consumption must be considered as it directly impacts the RCP processing viability due to carbon emissions (Costa; Gonçalves, 2022).

In this sense, corroborating the findings of other authors (Aquino Rocha; Toledo Filho, 2023; Duan *et al.*, 2020a, 2020b; Xu *et al.*, 2021), it was considered that mechanoactivation for 30 minutes using 0.5% content for all grinding aids concomitantly guaranteed a lower d50 and a higher BET for RCP, with values close to CPV ARI (12.21 µm and 3.1 m²/g) and were therefore analyzed regarding their potential for use as SCMs.

Analysis of the potential for using RCP as SCM

The specific mass, pH, and a summary of the physical characteristics (d10, d50, d90, and SSA-BET) of the CPV ARI and the powders with 0.5% GA mechanoactivated for 30 minutes are shown in Table 4.

It was noted that the mechanoactivated powders had a specific mass ranging between 2.68 and 2.47 g.cm⁻³, values lower than the CPV ARI, consistent with the values found in the literature and used as SCMs (Kim *et al.*, 2023; Oliveira *et al.*, 2023). The pH in the aqueous solution ranged between 12.27 and 12.43, indicating their high degree of alkalinity.

The raw materials mineralogical composition was identified by TGA/ Differential Thermal Analysis (DTA) and XRD/Rietveld (Figure 7). TG/DTA (Figure 7a) showed that the CPV ARI loss of mass was attributed to gypsum (110-145 °C), while in the recycled concrete powders, the hydrated phases decomposition corresponds to the tobermorite dehydration (not necessarily in crystalline form) and ettringite (50-200 °C); calcium sulfate dihydrate dehydration (110-145 °C); calcium hydroxide dehydration (380-500 °C) and the characteristic peak of portlandite and calcium carbonate decomposition (600-800 °C) (Deng *et al.*, 2021; Dweck *et al.*, 2000; Nobre, 2016). The loss on ignition (LOI) of recycled concrete powder mechanoactivated for 30 minutes was 8.54%, within limits for cement containing mineral additions (ABNT, 2018). The highest residual mass was found when the SHMP additive was used, with 90.57%.

Analyzing the positions of the Bragg peaks of the crystalline phases used and the reliability factor of Rietveld refinement (Rwp) and Goodness of fit (GOF) (Figure 7b), it is observed that mechanoactivation without and with grinding aids did not enhance the particles' amorphism.

The Consistency index (CI) of the pastes (Figure 8 a) and the compressive strength of the pastes containing 10% replacement of CPV ARI by RCP, and by mechanoactivated powders for 30 minutes without and with grinding aids at 0.5% are shown in Figure 8b. The Z-Score was used to detect outliers, and the result presented is the average of 4 determinations. The data were analyzed using the SISVAR Software (Ferreira, 2011) and were subjected to the Tukey Test ($p < 0.05$).

Observing Figures 8 a and b, it can be seen that the replacement of CPV ARI with recycled concrete powders caused an increase in compressive strength at 7 days of 22% when using RCP and 5% when using RCP-30 min-No GAs. The modification of the particle size distribution due to the presence of RCP may have reduced the water demand by replacing the water in the voids, and this additional water reflected in the improvement of workability (CI= 46.6 mm) (Hawkins; Tennis; Detwiler, 2005), and compressive strength at 7 days (46.6 MPa), corroborating the findings of Chen *et al.* (2021), who found no adverse effects on the pore structure of the cement paste when replacing up to 10% of the cement with RCP.

Figure 6 - Grindability index of recycled concrete powders

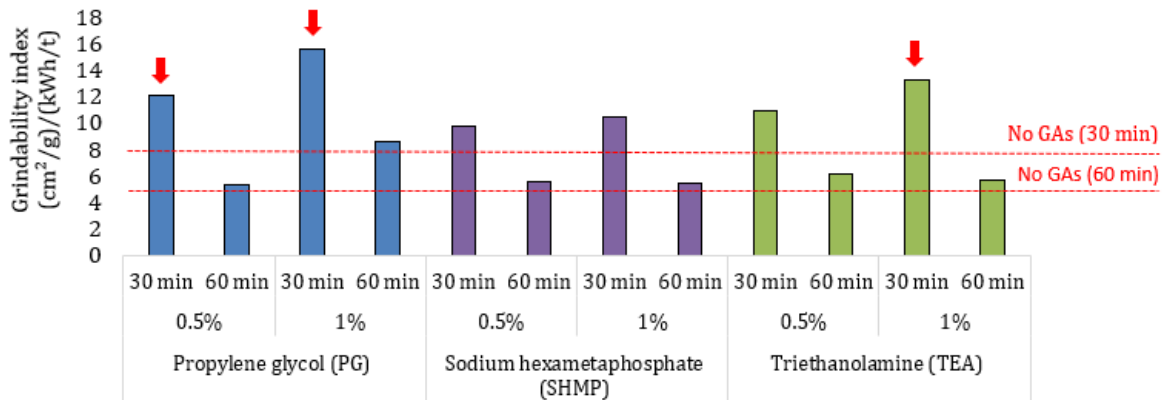


Table 4 - CPV ARI and recycled concrete powder characteristics

Materials	Specific mass (kg.m ⁻³)	pH	d10 (µm)	d50 (µm)	d90 (µm)	SSA - BET (m ² /g)
CPV ARI	3.09	12.00	1.03	12.21	36.77	3.10
RCP	2.68	12.45	1.50	20.99	75.11	6.23
RCP-30 min- No GAs	2.74	12.43	1.12	12.62	46.62	5.08
RCP-30 min- 0.5% PG	2.68	12.34	0.87	8.85	43.18	7.50
RCP-30 min- 0.5%SHMP	2.69	12.44	0.88	8.66	49.73	6.07
RCP-30 min- 0.5%TEA	2.69	12.27	0.87	8.89	46.78	6.81

Figure 7 - Materials' Mineralogical composition

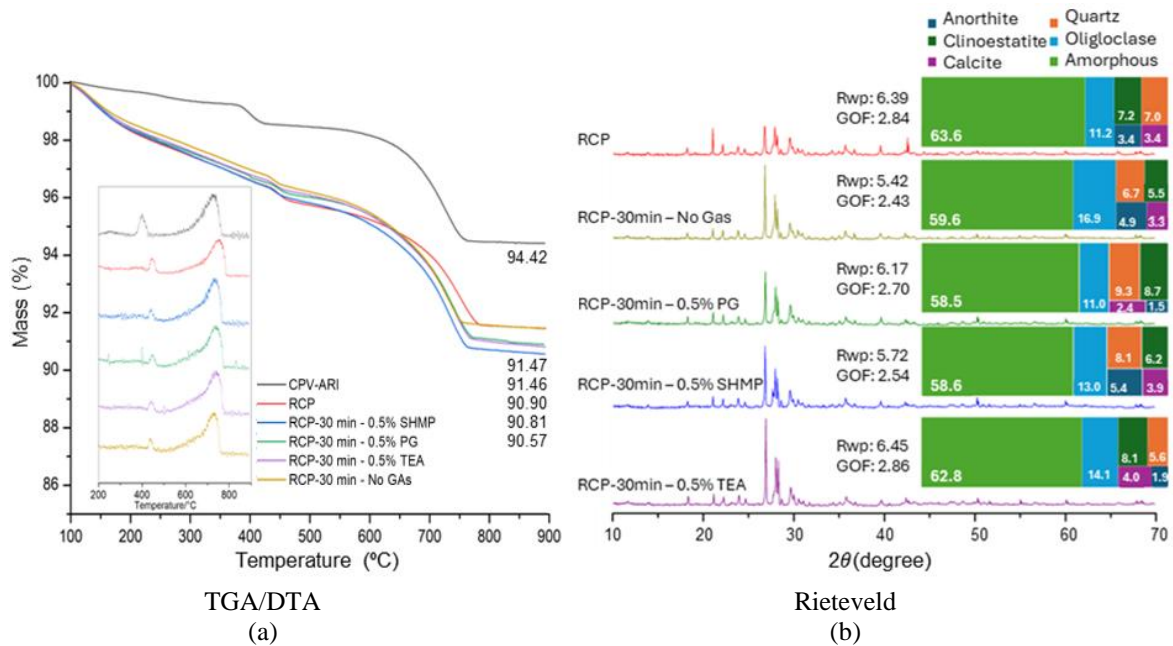
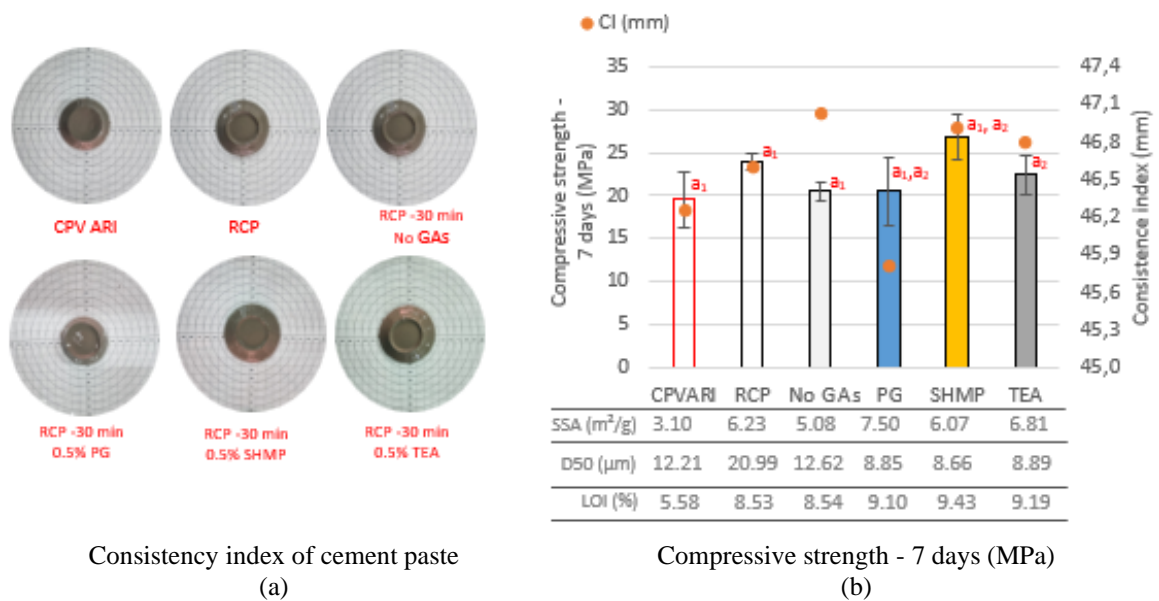


Figure 8 - Performance of mechanoactivated powders without and with grinding aids



When analyzing mechanoactivation in the planetary ball mill, it was observed that the paste containing RCP- 30min-No GAs presented an IC= 47 mm and a f_{c7} =20.49 MPa, a behavior that suggests that the dilution effect that occurs when the cement content is reduced, it has been maximized (Wang et al., 2024).

Taking into account that when RCP particles are finer than cement, due to water absorption, a decrease in the compressive strength of cementitious composites may occur (Zhang et al., 2023), it was verified from the Loss on ignition (LOI), which means the presence of grinding aids in the particles, may have compensated for the increase in fineness and specific surface area (SSA) in comparison to CPV ARI, providing an increase in the compressive strength of the pastes, when compared the reference (CPV ARI). It is worth highlighting that the paste containing RCP-30 min-0.5%PG was the grinding aid with the lowest LOI than others, and the particle with the highest SSA-BET (7.50 m²/g) had a direct impact on the consistency of the paste (CI=45.8 mm).

Statistically, the additives influenced the compressive strength of the pastes ($p < 0.05$), as indicated by the letters a1 and a2 (Figure 8b).

Finally, taking into account that the water/fine ratio was set at 0.48, adjusting water according to consistency can be a strategy to enhance the use of recycled concrete powders as supplementary cementitious materials, as already pointed out by John *et al.* (2019).

Conclusions

Based on the results, the following conclusions were drawn:

- (a) the use of a planetary ball mill combined with grinding aids contributed to the mechanoactivation of recycled concrete powders for use as supplementary cementitious material since, after 30 minutes, the Mean particle size (d50) was reduced, and the specific surface area (SSA) - BET increased simultaneously. However, there was no change in the material crystallinity;
- (b) the grindability index (K) as a function of the specific surface area (SSA) may be considered a way of evaluating the recycled concrete powders' mechanoactivation efficiency with grinding aids; the higher the k, the greater the efficiency of the process. Thus, the best results were obtained with 1% Propylene glycol (PG), followed by 1% Triethanolamine (TEA) and 0.5% Propylene glycol (PG), both lasting 30 minutes;
- (c) considering the shorter time and the smaller amount of grinding aid, grinding for 30 minutes with 0.5% Propylene glycol (PG) was suggested for the recycled concrete powder mechanoactivation since the RCP with a d50 of 20.99 and an SSA of 6.23 m²/g had a 42% d50 reduction and a 20% SSA increase; and
- (d) the results in pastes indicated that grinding aids favoured the use of recycled concrete powder as MSC, having a statistically significant influence on the compressive strength at 7 days. Using mechanoactivated RCP for 30 minutes and adding 0.5% SHMP showed an increase of around 3 MPa compared to RCP (<0.15 mm). However, for RCP-30min-0.5%PG, due to its high specific surface area (SSA) - BET, there was a reduction of around 3.5 MPa caused by the increase in water demand.

Suggested future studies:

- (a) carry out rheology studies on pastes containing recycled powders from mechano-activated concrete with different types and levels of grinding aids;
- (b) evaluate the effect of grinding aids on the setting of cements containing recycled concrete powders as additional mechano-activated cementitious materials; and
- (c) produce pastes with a standardized consistency index and use superplasticizing additives to compensate for the adverse effect of the finer particle size of recycled mechano-activated concrete powders, enhancing their use in higher levels as supplementary cementitious materials.

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3.2 ARTIGO 2 (ABSORÇÃO DE ÁGUA)

O artigo 2 denominado *Absorção de água em agregados finos e pós reciclados de RCD: um estudo comparativo usando análise termogravimétrica*, encontra-se em processo de avaliação por periódico científico. O objetivo do estudo foi propor um protocolo para a determinação da absorção de água de pós reciclados de resíduos da construção e demolição (RCD) utilizando análise termogravimétrica (TGA), dada a influência direta desse parâmetro na demanda de água e a ausência de métodos consolidados para esse tipo de material.

Para isso, analisou-se a absorção de água de agregados miúdos reciclados de RCD, dos tipos misto e de concreto, com duas faixas granulométricas distintas, a partir dos quais foram obtidos os respectivos pós, classificados em três granulometrias. Ao todo, foram avaliados dez materiais. A absorção de água dos agregados miúdos foi determinada conforme a NBR 16916 (2021), identificando a condição de Superfície Seca Saturada (SSD) pelo colapso da amostra no cone e posterior secagem a (100 ± 5) °C. Para os pós foi determinada com base no método proposto por Oliveira (2022), que consiste na pulverização de água até o colapso da amostra no cone de Kantro, seguida de secagem a 100 ± 5 °C. Ambos os procedimentos foram comparados ao método termogravimétrico (TGA), aplicado a amostras previamente mantidas em ambiente isotérmico (40 ± 5) °C por 3 horas. Padronizou-se o tempo de saturação em 24 horas.

Observou-se que a TGA apresentou menor variabilidade nos resultados, demonstrando potencial como técnica para avaliar a absorção de água tanto em agregados miúdos quanto em pós reciclados de RCD. Isso se deve ao fato de que a condição de SSD foi determinada graficamente após o ensaio, sem interferência de variáveis relacionadas à manipulação da amostra, o que reduz a influência do operador. Ressalta-se que a etapa de saturação deve ocorrer em menos de 24 horas para minimizar os efeitos da pré-hidratação de partículas residuais anidras de cimento.

A seguir, apresenta-se o artigo completo, conforme enviado ao periódico indexado na base Scopus, podendo sofrer modificações conforme a avaliação dos revisores para publicação.

WATER ABSORPTION IN RECYCLED CDW FINE AGGREGATES AND POWDERS: A COMPARATIVE STUDY USING THERMOGRAVIMETRIC ANALYSIS

Highlights

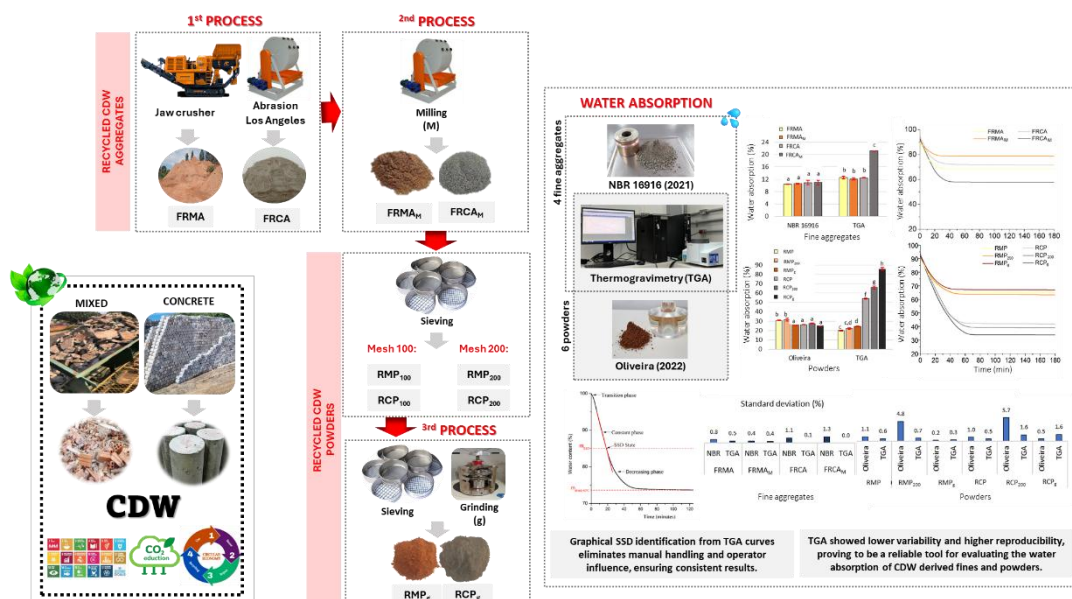
- Use of different methods to determine water absorption from fine aggregate and powder from CDW with different sources and particle size distributions.
- Recycled fine aggregates presented WA between 10% and 11% (NBR 16916) and 12% to 22% (thermogravimetry analysis method – TGA).
- Recycled powder from mixed sources presented WA between 25% and 32% and, from concrete, between 25% and 28% using Oliveira (2022) method.
- TGA method presented less variability in results and greater sensitivity to capture the smaller particles (0.15mm) influence in WA (fine and powder particle size).

Abstract

Water absorption determination (WA) of fine aggregates and powders from construction and demolition waste (CDW) is a recurring challenge in enabling the application of these materials, primarily due to their high porosity, varying chemical compositions, and high powder content (<0.075 mm). This paper aims to compare different methods for the determination of WA for 4 fine aggregates and 6 recycled powders from CDW, with varying sources and particle size distributions. For recycled fine aggregate, the method was evaluated from NBR 16916 (2021), with identification of the Saturated Surface Dry condition (SSD) by the cone sample collapse and drying until $(100 \pm 5)^\circ\text{C}$. For the powders, the method was evaluated by Oliveira (2022), which involves spraying water until the sample collapses in the Kantro cone and then drying it to $100^\circ\text{C} \pm 5^\circ\text{C}$. Both protocols were compared to the method with thermogravimetric analysis (TGA) application to samples submitted to an isothermal environment $(40 \pm 5)^\circ\text{C}$, for 3 hours. It was verified that TGA presented lower results variability and higher reproducibility compared to the conventional and adapted methods, highlighting its potential as a reliable tool for assessing the WA of CDW-derived fine aggregates and powders. The graphical identification of the SSD condition directly from the thermogravimetric curves eliminates manual handling and minimizes operator interference, ensuring consistent results. The sample saturation stage before testing must be completed within 24 hours to reduce the pre-hydration effects of anhydrous residual cement particles. The findings confirm that TGA is a promising thermoanalytical technique for quantitatively characterizing the water absorption behavior of recycled fines and powders, providing a physicochemically grounded and operator-independent approach that aligns with the principles of thermal analysis in materials science.

Keywords: Thermogravimetric analysis (TGA); Construction and demolition waste (CDW); Powders; Saturated Surface Dry (SSD).

Graphical abstract



1 INTRODUCTION

The construction industry employs approximately 7% of world population [1,2], being recognized for driving social and economic development in several countries. However, it faces significant challenges due to the demand for 30 to 50% of planet's natural resources [3,4] and the production of 10 to 30% of construction and demolition waste (CDW) [5,6]. In 2018, for example, the United States (US) produced 600 million tons of CDW [7], on European Union (EU) and China, in 2020, were generated respectively 0.807 [8] and 30.39 billion tons [9]. In Brazil, in 2019, approximately 7% of municipalities participating in the National Information System on Solid Waste Management (SINIR) generated just about 115 million tons [10].

These CDW are composed of a materials variety such as cementitious, ceramics, plastics, paper / cardboard, metals, glass, wood, plaster, paints, solvents, oils, among others [11], some of that could be recycled. If from cementitious and ceramic materials, it can be used as substitutes for natural raw materials, especially as coarse aggregates [12,13], fines aggregates [14,15] and supplementary cementitious materials (SCM) in cement [16,17], highlighting circularity [18–21] and promoting CO₂ emissions reduction depending on its application [22].

The effective application of CDW products (powder, fine, and coarse aggregate) depends on adequate characterization, especially regarding water absorption (WA), which is generally higher due to intrinsic CDW characteristics. However, there are still difficulties in measuring recycled CDW materials WA, especially post-processing (particle size < 0.15 mm). If WA is overestimated, the water excess influences the water to cement ratio (w/c), reducing concrete mechanical performance and durability [23,24], but if underestimated, the lack of water could lead to incomplete cement hydration reactions and reduced workability in fresh state, also compromising mechanical properties development [25,26]. The WA can be influenced by many factors such as amount of paste adhered to aggregate surface [27–31], the materials nature, particle size distribution [32,33], and the evaluation method applied [34–36].

For these reasons, several authors seek to improve procedures evaluation for this property, which are generally the same as those adopted for natural aggregates. Duan *et al.* (2022) [34] and Sosa *et al.* (2023) [37] documented that at least eight methods for WA measurement for recycled aggregate (RA), both coarse and fine, are available in the literature. The methods include techniques such as cone collapse, centrifugation, evaporimetry, airflow drying, conductivity, pycnometer, hydrostatic balance, and extrapolation. The water estimation of recycled fine aggregate was also observed by analyzing the decrease in mortars consistency [38] and the use of dispersant additives during tests to eliminate air bubbles between particles [32].

Conventional methods for determining the WA of fine and powdery recycled materials rely on empirical procedures and subjective visual assessments to identify the saturated surface-dry (SSD) condition. These approaches often result in high variability among operators and laboratories, particularly when applied to materials containing large fractions of particles smaller than 0.15 mm, which tend to agglomerate and retain water within their porous structure. Although some studies have proposed alternative procedures to improve reproducibility, most of them still depend on manual handling and empirical criteria, which limit their accuracy and reliability as a quantitative and reproducible technique to evaluate the water absorption of fines. Unlike conventional methods, TGA is based on physicochemical principles that allow the kinetic distinction between free and adsorbed water during controlled heating. This approach eliminates the subjective step of visually identifying the SSD condition and minimizes sample handling, thus improving both accuracy and repeatability. Moreover, the method provides objective graphical criteria for defining the hygroscopic equilibrium point of the material, offering more profound insight into the water–solid interaction in wonderful recycled particles. Consequently, this study introduces a new thermoanalytical framework for characterizing recycled cementitious materials, contributing to the advancement of standardized procedures for WA determination and for the quality control of fine recycled aggregates.

Therefore, this study aims to assess the applicability of TGA for determining the water absorption of fine and powdery recycled cementitious materials and to compare its performance with that of conventional and adapted methods currently used in literature. The research investigates the influence

of saturation time and particle size distribution on WA results, providing a comprehensive analysis of the reproducibility, sensitivity, and practical feasibility of the TGA approach.

Accordingly, this work compares the method presented in NBR 16916 [39], usually applied for recycled fine aggregate, and the procedure proposed by Oliveira's (2022), adapted for powder, with WA determination through TGA. The TGA technique has been investigated by several authors as a potential analytical tool, aiming to establish a faster and more reliable method for measuring the water absorption of recycled aggregates [36,40].

The outcomes are discussed in the context of developing a more reliable methodology for characterizing powders, with potential implications for mixture design as well as allowing greater control of desirable properties, enabling more effective dosages. These aspects will contribute to carbon reduction goals by providing better performance with optimized materials consumption, benefiting circularity aspects and, Environmental, Social, and Corporate Governance (ESG) goals.

2 MATERIALS AND METHODS

2.1 MATERIALS PROCESSING

It was evaluated the WA of 4 fine aggregates ($\phi < 4.75$ mm) and 6 powders ($\phi < 0.15$ mm), which were obtained in the recycling process of construction and demolition waste (CDW) from two different sources (mixed and full concrete), according to processes presented in Figure 1 and Table 1.

Figure 1- Processes for obtaining Fine Recycled Mixed Aggregates and Recycled Powder

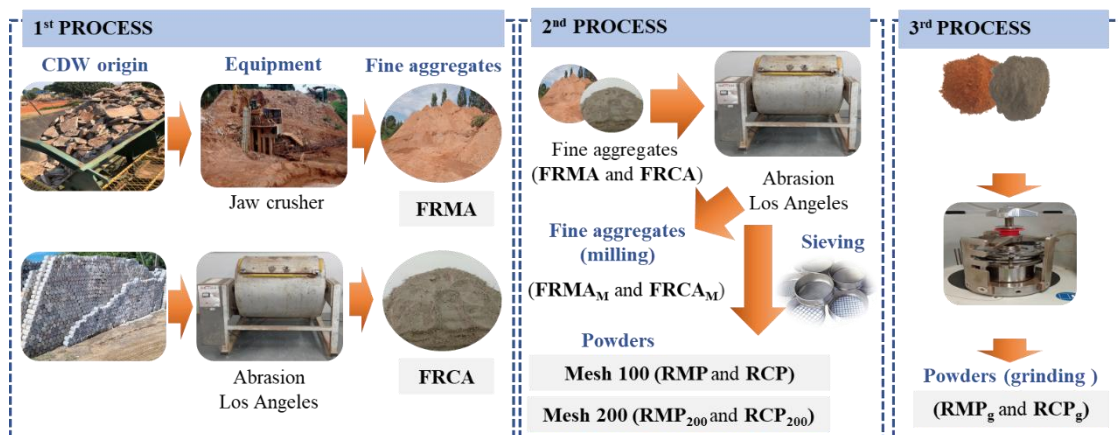


Table 1- Identification of recycled fine aggregate and powder used

CDW source	Designation	Description	Process*
Mixed	FRMA	Fine Recycled Mixed Aggregates	1 st
	FRMA _M	Fine Recycled Mixed Aggregates Milling	2 nd
	RMP	Recycled Mixed Powder sieved the mesh 100	2 nd
	RMP ₂₀₀	Recycled Mixed Powder sieved the mesh 200	2 nd
	RMP _g	Recycled Mixed Powder - grinding	3 rd
Concrete	FRCA	Fine recycled Concrete Aggregates	1 st
	FRCA _M	Fine recycled Concrete Aggregates Milling	2 nd
	RCP	Recycled Concrete Powder sieved the mesh 100	2 nd
	RCP ₂₀₀	Recycled Concrete Powder sieved the mesh 200	2 nd
	RCP _g	Recycled Concrete Powder - grinding	3 rd

* The processes are shown in Figure 1.

The first process consisted of collecting fine recycled mixed aggregates (ceramic and cementitious materials) in a CWD recycling plant, with processing of up to 3000 m³ per month (40% of nominal capacity). The material underwent screening, grinding in a jaw crusher and sieving process (Mesh 4 - $\phi < 4.75$ mm), until obtaining Fine Recycled Mixed Aggregates (FRMA). To the production of Fine Recycled Concrete Aggregates (FRCA), concrete specimens discharged after technological control testing were grounded in lab environment using a Los Angeles abrasion machine, with the fraction passing through the sieve being reserved for application (Mesh 4 - $\phi < 4.75$ mm).

In the 2nd process, FRMA and FRCA were grounded for 2 hours in a Los Angeles abrasion machine, obtaining two fine aggregates with higher fines content, FRMA_M and FRCA_M respectively. Also in this process, the RMP₁₀₀ and RCP₁₀₀ powders (passing through the Mesh 100 sieve) and RMP₂₀₀ and RCP₂₀₀ (passing through Mesh 200) were obtained by sieving.

In the third process, the mixed and concrete powders that passed through Mesh 100 ($\phi < 0.15$ mm) were grounded for 0.5 hours in a PM 100 (Retsch) planetary ball mill, generating RMP_g and RCP_g powders. The comminution was carried out by a batch of 60 g in a 250 cm³ agate jar, with 50 agate spheres with an approximate diameter of 10 mm and a 500-rpm rotation. The rotation direction was automatically reversed after 15 minutes with a 1-minute break for cooling.

2.2. CHARACTERIZATION

The fine aggregates particle size distribution was verified by sieving (NBR 17054, ABNT, 2022) and for powders it was analyzed by laser (Cilas 1190) with a Reading range between 0.04 and 2500 μ m, in liquid environment (distilled water), no dispersant and with an obscuration index of approximately 25%, according to ISO 13320 [42]. To complement the analysis, it was determined the material content passing through Mesh 200. All in accordance with NBR 16973 [43]. The bulk density was determined in a gas pycnometer (He), model Ultrapyc 5000, from Anton Paar, with an operating pressure of 10 psi, as required by ASTM D5550-14 [44]. The specific surface was obtained by nitrogen adsorption (BET method) in Quantachrome equipment, model NOVA 3200. Before tests performing, samples were submitted to degassing (vacuum at 40°C per 16 hours) [45].

The semi-quantitative oxides chemical composition was determined by X-Ray Fluorescence (XRF) performed on a Rigaku model ZSX Primus IV spectrometer, equipped with a Rh tube, where an aliquot of approximately 10 grams each was separated by manual quartering and pulverized in a planetary ball mill (agate) for 10 minutes. After grinding, each sample was dried at 105 °C for 2 hours. For lost on fire determination (LOI) each sample was calcinated at 1000 °C for 5 horas in a Precisa prepASH 340 model 212 thermogravimetry analyzer.

The morphological characterization was performed by Scanning Electron Microscopy (SEM) in Zeiss equipment – EVO MA10, with image detection from secondary electrons. The particles (fine aggregates and powder) were lightly deposited in stubs with carbon tape, a quick blasting was performed to remove the particles not adhered to the tape and then subjected to gold/platinum deposition using Sputter Coater Quorum SC7620 (15mA for 1:30 minutes). The filament used was tungsten and the potential difference employed was 10kV.

2.3 WATER ABSORPTION (WA) DETERMINATION

To determine WA standard methods NBR 16916 [39] and thermogravimetry were used for fine aggregates and the Oliveira (2022) [46] and thermogravimetry for powders, according to Table 2.

Table 2- Methods for determining water absorption (WA)

Material	Method	Sample mass	Saturation	SSD*	Drying temperature
Fine Aggregates	Standard Method NBR 16916 (2021) [39]	500 g	24 hours	By cone collapse	(100°C ± 5) °C
Recycled Powder	Oliveira (2022) [46]	200 g	Sprinkling Water (~0.5 hours)	By cone collapse	(100°C ± 5) °C
Fine Aggregates and Recycled Powder	Thermogravimetry Analysis (TGA)	50 g	0.5 and 24 hours	Graphically	(40°C ± 5) °C

* Identification of the Saturated Surface Dry (SSD) condition

Tests were performed in a triplicate, adopting as the test result the arithmetic mean of the two closest results, which were subjected to analysis of variance – ANOVA to compare means and Turkey test with a probability level of 5% to verify significant difference (p -value < 0.05). The statistical analysis was performed in the SISVAR [47] software.

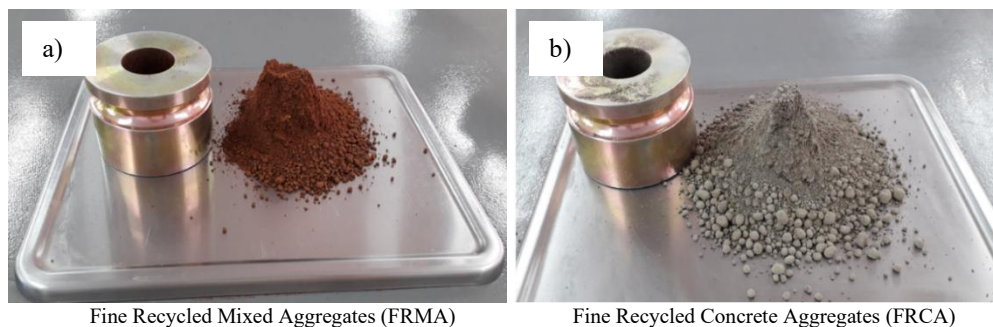
2.3.1 WA for fine recycled aggregates

The standard method of NBR 16916 [39] consisted of saturating the fine aggregate samples for 24 hours in atmospheric pressure (1 atm) with drying by air flow until Saturated Surface Dry (SSD) condition. The SSD condition is considered to have been reached when there is no water on the particles surface but the pores are completely filled [34] which in this test is identified at the moment when the partial cone collapse occurs (Figure 2), being strongly related to the observer's experience. The samples were weighed and dried at (100 ± 5) °C for 24 hours. WA was then calculated according to Equation 1.

$$WA = \frac{m_{SSD} - m_{dried,100^{\circ}C}}{m_{dried,100^{\circ}C}} \times 100 \quad \text{Equation 1}$$

Where: WA is the water absorption (%); m_{SSD} is the sample mass in Saturated Surface Dry condition (g) and $m_{dried,100^{\circ}C}$ is the mass in dried condition (100 ± 5) °C (g).

Figure 2- Saturated Surface Dry (SSD) condition – Standard Method – NBR 16916 (fines): a) Fine Recycled Mixed Aggregates (FRMA); b) Fine Recycled Concrete Aggregates (FRCA)

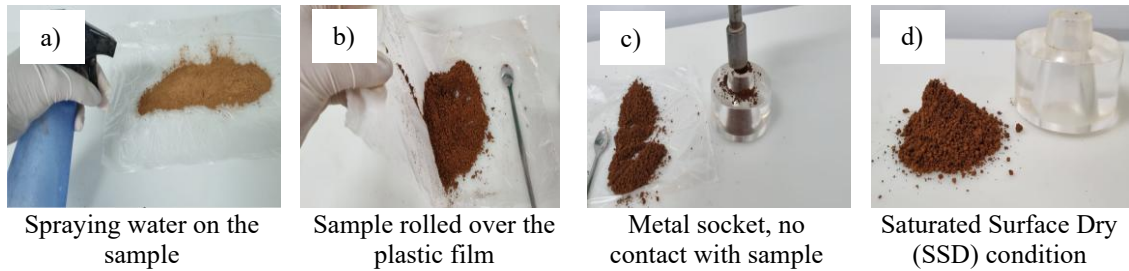


If total collapse occurs on the first attempt, the fine aggregate must have been dried beyond SSD condition, so a small amount of water (a few milliliters) must be added, mixed until the sample appears homogeneous and left in a closed container for 30 minutes, starting the drying and test process again.

2.3.2 WA for recycled powders

For powders WA determination, Oliveira (2022) [46] proposed an adaptation on the German method based on standard DIN 4226-10 [48]. The test consists of placing a 200 g dry sample on a plastic film and spraying water (Figure 3a) until the particles' surface is all slightly moistened, rotating the material to homogenize the moisture (Figure 3b). After 15 minutes, samples should be transferred to Kantro cone, lightly striking the mold 10 times with a metal socket, without contact with the sample, only to cause vibration and promote material adjustment inside the cone (Figure 3c). Then the mold is removed vertically, observing the cone's collapse shape. When partial collapse is verified, the SSD condition is observed (Figure 3d). In this case, material sample is weighed and subsequently placed in an oven (100 ± 5) °C for 24 hours, for WA determination according to Equation 1.

Figure 3- WA determination according to Oliveira (2022): a) spraying water on the sample; b) sample rolled over the plastic film; c) metal socket, no contact with sample; d) SSD condition



If the sample is dry, the procedure must be repeated until the cone shape partially collapses. Even with all due care, depending on the amount of water and combined with the plastic film movement, powders may clump together and form a paste, and, in this case, the sample must be discarded.

2.3.3 WA for CDW fines and powders

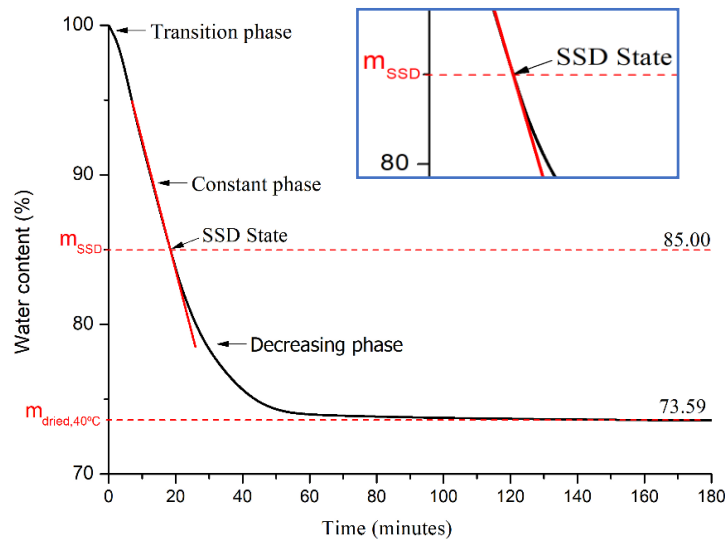
The thermogravimetric analysis (TGA) is based on the porous media theory, which recognizes that free water and absorbed water do not evaporate at the same rate [40,49–51]. In this study, the method was used to evaluate the water absorption of fine aggregates and CDW powders, using the same protocol.

Initially, 50 grams samples (fine or powders) were saturated for 24 hours in 100ml of deionized water at 1atm. Soon after, the materials were passed through a paper filter of 80g to remove excess water. Next, approximately 130 ± 10 mg of saturated sample was placed in an alumina crucible for testing in a thermoanalyzer (Percking Elmer – STA 8000). The test was conducted in a temperature of (40 ± 5) °C to avoid ettringite dehydration, overestimating WA value, as indicated in the literature [45,52,53], in a controlled atmosphere with a nitrogen flow of 20 ml/min for 180 minutes. Data were compiled using OriginPro 8.5 [54] software and plotted on a xy graph using a drying curve versus test time as Illustrated in Figure 4.

Figure 4 shows that there is initially a transition phase, which occurs until the experiment reaches a (40 ± 5) °C temperature (approximately 5 minutes), followed by a constant phase (surface water evaporation, between 5 and 20 minutes) and then a decreasing phase (absorbed water evaporation, between 20 and 55 minutes). From then on, experimente stabilizes (after 55 minutes) considering the moment when the sample reached the dry stage ($m_{dried,40^{\circ}C}$). The transition point between constant and decreasing phase, determined with the aid of a graphic line (in red) is considered SSD state. So, WA was calculated based on Equation 2 [55].

$$WA = \frac{m_{SSD} - m_{dried,40^{\circ}C}}{m_{dried,40^{\circ}C}} \times 100 \quad \text{Equation 2}$$

Where: WA is water absorption (%); m_{SSD} sample mass at saturated sample condition (SSD) (%) and $m_{dried,40^{\circ}C}$ is the dried sample at 40 ± 5 °C (%).

Figure 4- Example of drying curve versus time by thermogravimetry

To assess experimental reproducibility, the test was performed in triplicate, and three independent readers conducted the graphical interpretation. The results were compared to ensure consistency and to allow statistical analysis of variability among samples.

3 RESULTS AND DISCUSSION

3.1. PHYSICAL AND CHEMICAL PROPERTIES

3.1.1 Fine Aggregates

CDW fine aggregates physical characterization can be seen in Table 3. It can be seen that the FRMA sample has a fineness modulus similar to natural sand, which is generally between 2.20 and 3.5 [56]. The fine recycled mixed aggregates, due to the red ceramics presence, demonstrated higher surface area than the fine recycled concrete aggregates, since ceramic origin particles are more porous, less strengthened and a more lamellar shape [57].

Analyzing the grinding effect (2nd process), it is observed that FRMA_M and FRCAM, despite a finer particle size, presented a smaller surface area than FRMA and FRCA, respectively. This occurs due to the interaction between particles and Van der Waals adhesion forces becoming much stronger, resulting in agglomeration and on increase in particle size [58,59].

Table 3- Fine aggregates physical characteristics

Characteristics	Method	FRMA	FRMA _M	FRCA	FRCAM
Fineness modulus (FM)	NBR 17054 [41]	2.39	1.66	1.92	1.38
Density (kg/m ³)	ASTM D5550 [44]	2680	2670	2690	2740
Surface area- BET (m ² /g)	Scrivener et al. (2016) [45]	14.32	13.90	6.09	4.07
Passed through Mesh 200 (%)	NBR 16973 [43]	28.17	33.45	17.64	19.41

The specific masses are consistent with the values found in literature [60,61] and close to the value of quartz sand used in the study region (2590 kg/m³). Analyzing the amount of material passing through Mesh 200 sieve (%), all fine aggregates presented values higher than requirement prescribed by NBR 15116 [62] for application as inputs in Portland cement concrete, with and without structural function, prefabricated artifacts, mortars and similar.

X-ray fluorescence (XRF) analysis was carried out based on the quantification of major elements, expressed in the form of oxides. Major elements are defined as those with concentrations higher than 1.0 wt% of the analyzed material; minor elements have concentrations between 0.1% and 1.0%; and trace elements, with contents below 0.1%, are typically reported in parts per million (ppm). As shown

in Table 4, the main oxides identified were silicon dioxide (SiO_2), calcium oxide (CaO), and aluminum oxide (Al_2O_3), followed, in smaller amounts, by iron oxide (Fe_2O_3) and magnesium oxide (MgO). The sum of silica, alumina and iron oxide contents is associated with the silicates present in rocks, sands and ceramics. And the presence of CaO and MgO refers to the cement paste and rocks with limestone origin [63].

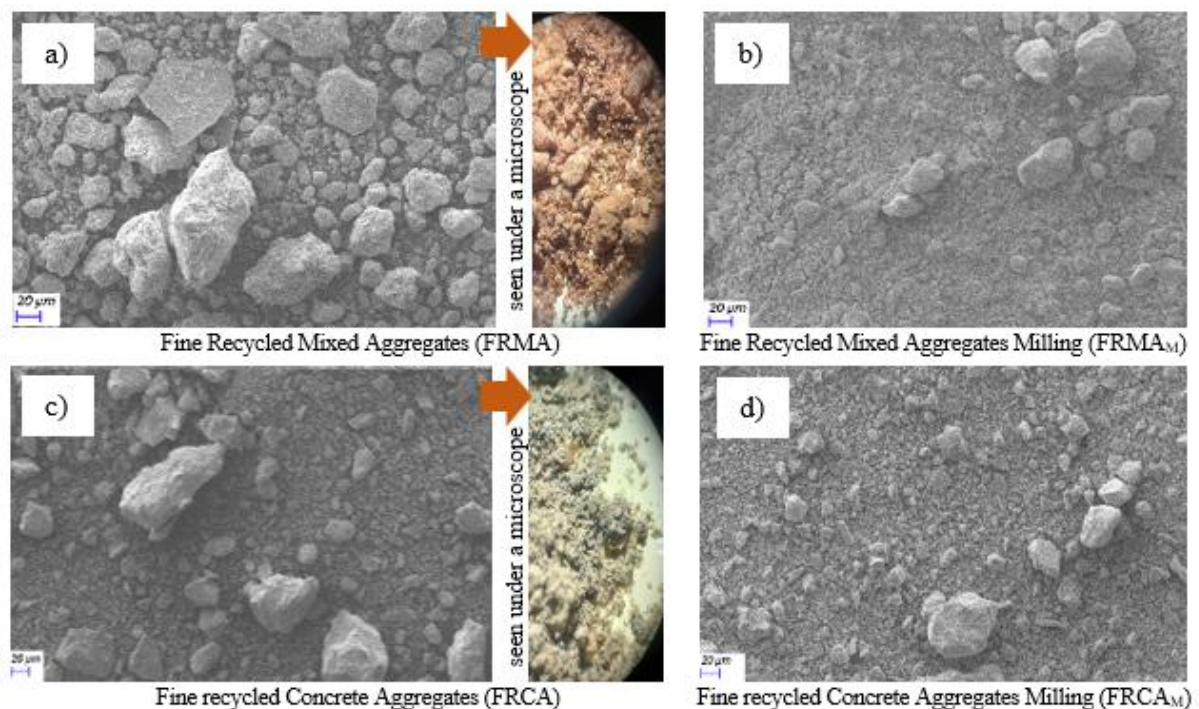
Table 4- Fine recycled aggregates chemical composition

Fine aggregates	Chemical composition (%)										
	SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	K_2O	SO_3	Na_2O	MnO	P_2O_5	LOI
FRMA	68.20	8.38	7.36	4.15	1.74	0.84	0.38	0.35	<LQ	<LQ	7.25
FRCA	53.02	15.91	8.25	8.43	3.18	1.95	0.29	1.49	0.14	0.27	5.10

LOI: Loss on Ignition; <LQ: below the quantifiable limit.

It is perceptible that FRCA presented greater amount of Fe_2O_3 than FRMA, which could be justified by the use of basaltic stone powder in concrete's composition [64]. Recycled sands morphological characteristics, obtained from scanning electron microscopy with $500\times$ magnification is presented in Figure 5. With an illustrative image obtained in a binocular stereomicroscope with WF10x eyepieces (Lumen), for FRMA and FRCA samples.

Figure 5- Morphology of fine recycled aggregates (magnification: $500\times$): a) Fine Recycled Mixed Aggregates (FRMA), b) Fine Recycled Mixed Aggregates Milling (FRMA_M), c) Fine recycled Concrete Aggregates (FRCA), d) Fine recycled Concrete Aggregates Milling (FRCA_M)



The mixed aggregates (Figures 5a and 5b) have particles with irregular shapes, apparently with high roughness and, due to ceramic materials presence, they contain sharp corners, irregular edges, cracks and notches that favor fine particles accumulation, which increases with improve in ceramic materials presence on the sample [65,66], findings made through both microscopes. For concrete materials (Figure 5c and 5d), it can be seen that particles presented a portion of roughness with adhered particles in the grains surface, corroborating the conclusions of Geng and Sun (2013) [67].

The variation in particle size and the amount of particles smaller than 0.15 mm can be considered the main difference between sands, for example, FRMA presents a continuous size distribution (Figure 5a),

while others have a discontinuous distribution with a with powder content (Figures 5b, 5c and 5d), corroborating with the physical characterization shown in Table 3.

3.1.2 Recycled Powder

The powders particle size distribution is demonstrated in the granulometric (Figure 6) and physical characteristics (Table 5). It can be seen (Figure 6) that the d50 diameter of RMP and RCP are larger than the other samples, indicating the efficiency of grinding and sieving (2nd and 3rd processes indicated on Figure 1) being: RMP>RCP>RCP₂₀₀>RMP₂₀₀>RCP_g>RMP_g, the latter, with a value like Ordinary Portland cement (OPC, 9.57 μ m).

Regarding density, the mixed recycled powders presented values between 2670 kg/m³ and 2690 kg/m³, while the concrete powders 2680 to 2704 kg/m³ values lower than the Portland cement particles that usually presented values in the order of 2900 to 3200 kg/m³. Although the particle size was refined with the third process (Table 5), it is observed that the Specific Surface Area - BET reduced, indicating particle agglomeration due to attractive Van der Waals forces [68]. It is noteworthy that mixed powders SSA-BET are higher than those of concrete origin powders, it can be justified by ceramic materials presence in mixed sample's composition.

Figure 6- Particle size distribution

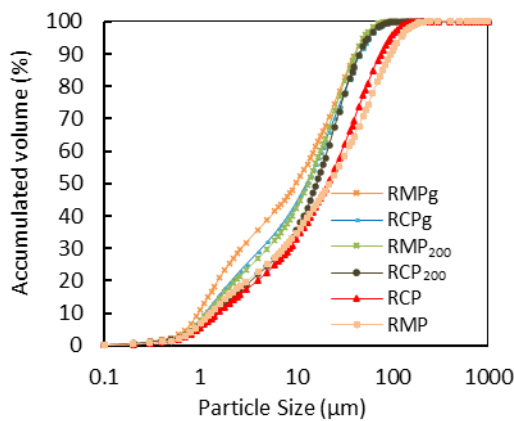


Table 5- Powders physical characteristics

Powders	d10 (μm)	d50 (μm)	d90 (μm)	Density (g/cm ³)	BET* (m ² /g)
RMP	1.308	22.071	96.347	2.67	23.80
RMP ₂₀₀	1.209	13.521	41.773	2.67	27.07
RMP _g	0.951	9.923	42.908	2.69	19.50
RCP	1.501	20.993	75.11	2.68	6.23
RCP ₂₀₀	1.435	16.189	44.842	2.68	6.53
RCP _g	1.122	12.624	46.622	2.74	5.08

* Specific surface area (SSA- BET)

Regarding density, the mixed recycled powders presented values between 2670 kg/m³ and 2690 kg/m³, while the concrete powders 2680 to 2704 kg/m³ values lower than the Portland cement particles that usually presented values in the order of 2900 to 3200 kg/m³. Although the particle size was refined with the third process (Table 5), it is observed that the Specific Surface Area - BET reduced, indicating particle agglomeration due to attractive Van der Waals forces [68]. It is noteworthy that mixed powders SSA-BET are higher than those of the concrete origin powders, it can be justified by ceramic materials presence in mixed sample's composition.

The powders microscopic morphologies determined by Scanning Electron Microscopy (SEM), are presented in Figure 7, while the Chemical composition on the oxides basis analyzed by X-ray fluorescence (XRF) is listed in Table 6. Comparing sources, it can be observed in Table 6 that RCP is rich in SiO₂ and CaO, and RMP₁₀₀ in SiO₂, CaO and Al₂O₃, similar results were found by other authors [69,70]. The greater amount of CaO, when compared to the fine aggregates (Table 4), is due to the greater cement paste or red ceramics content in these particle size distributions [71].

Figure 7- Morphology of recycled powders (magnification: 2.5 k \times): a) Recycled Mixed Powder sieved the mesh 100 (RMP), b) Recycled Mixed Powder sieved the mesh 200 (RMP₂₀₀), c) Recycled Mixed Powder – Grinding (RMP_g), d) Recycled Concrete Powder sieved the mesh 100 (RCP), e) Recycled Concrete Powder sieved the mesh 200 (RCP₂₀₀) f) Recycled Concrete Powder – Grinding (RCP_g)

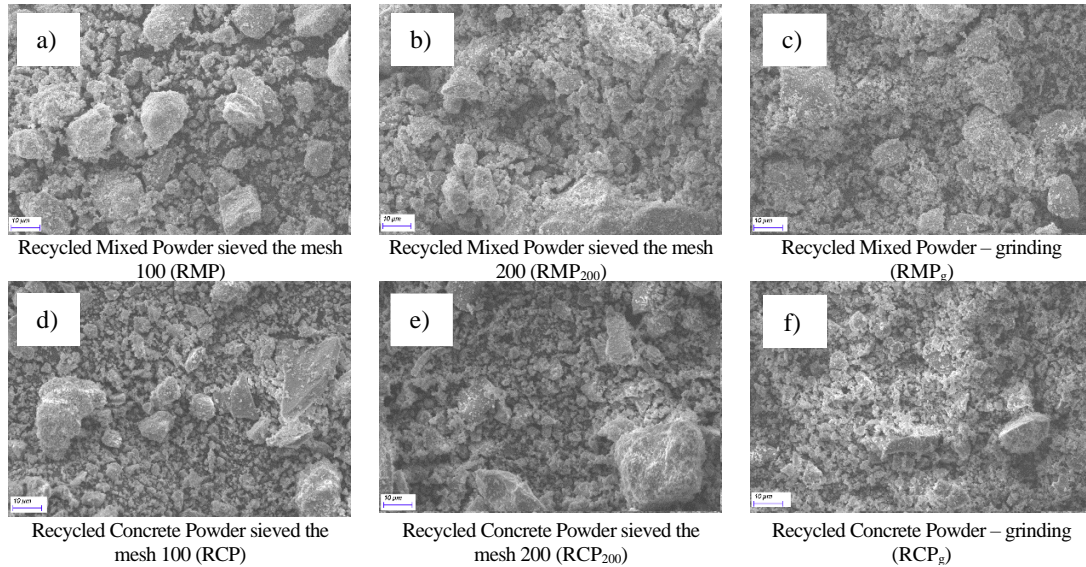


Table 6- Powders chemical composition

Powders	Chemical composition (%)										
	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	SO ₃	Na ₂ O	MnO	P ₂ O ₅	LOI
RMP₁₀₀ RMP_G	39.55	16.68	12.88	6.65	3.64	1.0	0.45	0.28	<LQ	0.11	17.08
RMP₂₀₀	33.48	17.42	12.97	9.18	3.95	0.52	0.80	0.27	<LQ	0.14	18.76
RCP₁₀₀ RCP_G	47.20	21.36	8.56	6.94	3.28	2.14	0.62	1.36	0.13	0.22	7.84
RCP₂₀₀	43.07	20.79	9.50	8.75	3.37	0.99	0.96	1.58	0.15	0.25	8.91

LOI: Loss on Ignition; <LQ: below the quantifiable limit.

3.2 WATER ABSORPTION

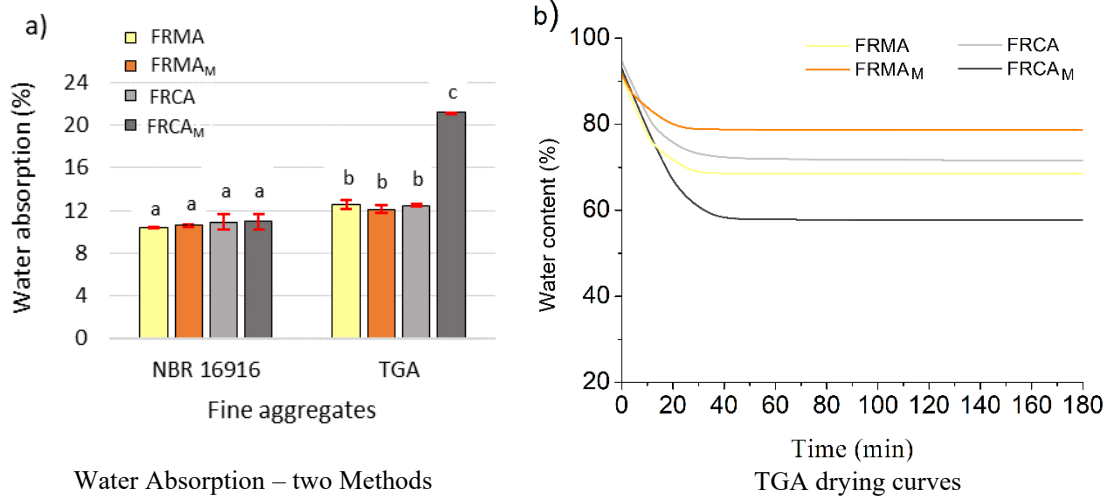
3.2.1 Fine aggregates

The average results of two water absorption determinations (closest values), with a 24-hours saturation, of recycled CDW fine by the Standard Method – NBR 16916 [39] and thermogravimetry analysis (TGA) can be visualized in Figure 8, as well as the drying curves as a function of time.

The water absorption of recycled sands ranged from 10 to 11%, corroborating results found in the fine recycled concrete aggregates literature (6 to 14%) [72] and ceramics (9 to 19%) [56]. Statistically, according to the Standard Method – NBR 16916 there was no significant difference between results (same letters), which indicates that the method did not capture the influence of particle size distribution, powder material content and waste source on water absorption, a finding also made by other authors [73,74].

During the Standard Method – NBR 16916 test, in addition to the time required to sample drying (about 3 hours), it was found that there was a loss of fine that dispersed during air current application, altering sample's characteristics. During SSD condition determination procedure, it is not possible to guarantee homogeneous particles drying, which can cause partial decomposition of hydrated cement phases due to the drying temperature (100 \pm 5) °C and increase samples' porosity. In addition, the heat applied during the test and the saturation time combination favored anhydrous grains hydration, forming clods, visually identified when SSD condition was determined (Figure 2b).

Figure 8- CDW recycled fine aggregates WA: a) Two methods; b) TGA drying curves



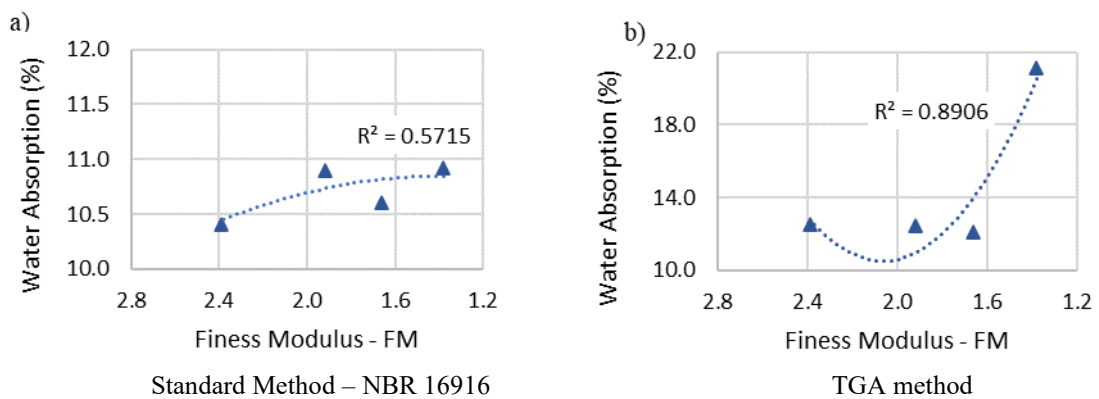
In Figure 8b, excluding the time related to the transition phase (approximately 5 minutes), the test lasted between 20 and 40 minutes. The results obtained by TGA indicated water absorption values higher than those obtained by Standard Method – NBR 16916, ranging from 12 to 22%, with FRCA_M being considered to have a statistically different water absorption than the others.

The finer particle size composition (fineness modulus = 1.38) the presence of greater amount of material < 0.075 mm (19.41%) and the residual anhydrous cement particles exposure during grinding (2nd process), combined with saturation time for 24 hours, favored the hydration reactions when in contact with water. Therefore, materials containing reactive powders should be tested with shorter saturation times.

The results obtained by TGA corroborate findings of Sosa *et al.* (2023) [37]. According to the authors, the main Variable that influenced recycled aggregate WA by the electrical conductivity method was the paste content, while the particle size and shape had no noticeable influence.

To complement the analysis, observe Figure 9. The TGA method presents a polynomial correlation between water absorption (WA) and fineness modulus (FM) ($R^2 = 0.8922$) compared to the NBR method ($R^2 = 0.5715$). This suggests that the TGA method is more reliable for evaluating WA for fine recycled aggregates that contain a significant amount of hardened paste (particles smaller than 0.15 mm).

Figure 9- Correlations between WA and FM: a) Standard method – NBR 16916; TGA method



In the Standard Method – NBR 16916 the SSD condition evaluation is left for the operator, while the TGA is done through graphic determination. In this way, the mean of three measurements is presented in Figure 10 just like the analysis of variance (ANOVA and the Tukey test).

The graphical analysis with the tangent line positioning is related to the reader’s experience in using thermogravimetry technique. Thus, according to ANOVA (Table 7), the operator has a statistical influence ($p < 0.05$) on the fine recycled aggregates’ water absorption by TGA method. Although the error associated with the operator can influence the test result, the TGA application is faster and eliminates the need for manual work by the operator to dry samples.

Figure 10- Water Absorption from Fine Aggregates by TGA for three Measurements: Readers R1, R2, R3

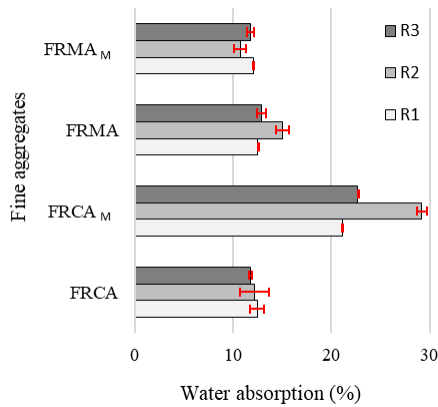


Table 7- WA ANOVA of three measurements

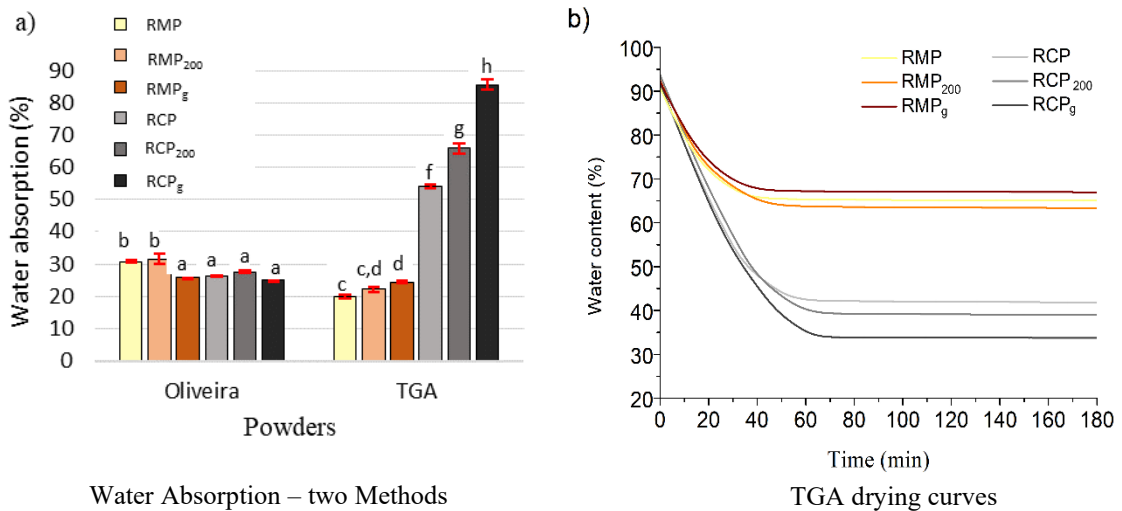
Variable	SS	DF	MS	Fc	p-value
Fine aggregate	327.340467	3	109.113489	1.00E+09	0.000
Readers	12.065217	2	6.032608	1.00E+09	0.000
Fine aggregate*Readers	29.508983	6	4.918164	1.00E+09	0.000
error	0	0	0.00E+00		

SS: sum of squares; DF:degrees of freedom; MS: mean square.
Number of observations: 12

3.2.2 Recycled Powder

The mean results of WA for recycled powders, applying Oliveira’s method (2022) [46] and TGA, with a 24-hour saturation time, can be visualized in Figure 11.

Figure 11- CDW Fine aggregates’ water absorption by: a) Two methods; b) TGA drying curves



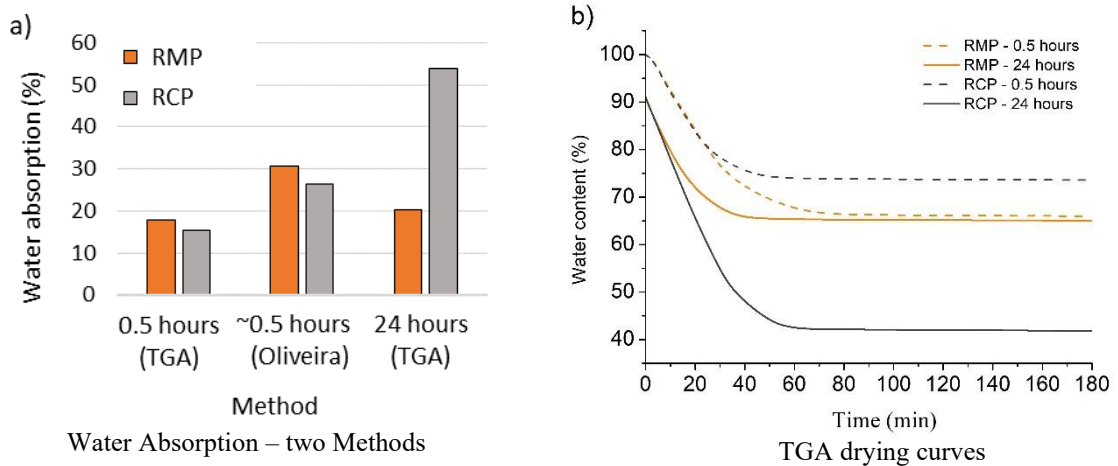
According to the method by Oliveira (2022) [46], mixed recycled powders presented values between 25 and 32% and for a concrete source between 25 and 28%, higher than those verified by Oliveira (2022) [46], with 14% for concrete powder ($d_{50} = 25.04 \mu\text{m}$) and 15% for the mixed type ($d_{50} = 35.52 \mu\text{m}$). These differences can be justified by the powders’ handling difficulty which, when in contact with sprayed water, tend to agglomerate due to interparticle attractions (Van Der Waals forces), specially in mixed powders.

In Figure 11a, for the mixed powders, there is a difference in WA depending on particle size, while the concrete powders were considered statistically equal (equal letters). In turn, using TGA method, it was

found that the mixed powders RMP_{100} , RMP_{200} and RMP_G , presented WA values lower than those found by Oliveira (2022), between 20% and 24%, with the RMP_{100} and RMP_G powders being considered statistically different, which indicated that, unlike fine aggregates, the powders particle size distribution influences this property (also confirmed by TGA).

Analyzing the drying curve (Figure 11b), it is observed that, for concrete powders the test lasted 50 to 80 minutes, longer than the mixed powders duration (30 to 50 minutes). Values between 54% and 86% suggest that, in addition to the WA, results reflect the total combined water, which is released between 35 and 450°C [75], and that the anhydrous particles exposure during milling, combined with saturation time, favored hydration. To confirm this hypothesis, the test was repeated with 30-minutes reduced saturation time and the results are compared in Figure 12.

Figure 12- WA depending on the method and saturation time of the powders: a) Two methods; b) TGA drying curves

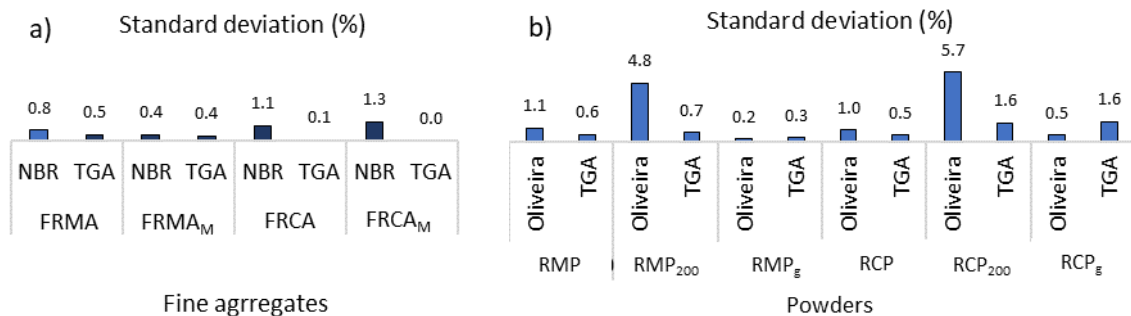


It can be observed (Figure 12) that, for 0.5-hours saturation by TGA, RMP_{100} presented a 18% absorption while RCP_{100} , 15%, following the same trend observed in the method by Oliveira (2022)[46]. Statistically, saturation time did not influence mixed powder water absorption (RMP_{100}).

In view of the above, in powders containing anhydrous cement particles, the saturation method and time influence the WA (Figures 12a e 12b). Thus, the TGA method can be used to determine water absorption in powders at saturation time of less than 24 hours. Typically, concrete production time is approximately 2 hours (mixing, Transportation and application) [76], in which case a reasonable saturation time to be considered could be between 0.5 and 2 hours, corroborating others authors recommendation [40].

Finally, for comparison purposes between methods, the WA standard deviations for fine recycled aggregates and powders were plotted in Figure 13, considering 3 repetitions of the same operator / reader. It should be noted that the results for TGA refer to those obtained by reader 1 (R1).

Figure 13- Standard deviation (%) of WA for 3 repetitions/measurements: a) Fine aggregates; b) Powders



Analyzing Figures 13a and 13b, it is observed that, except for powders obtained in the 3 ST process (RMP_G and RCP_G), the TGA method produced results with lower standard deviation than the Standard Method – NBR 16916 and Oliveira (2022), results that can contribute to the choice of most appropriate method for evaluating CDW fine aggregates and powders WA. For finer powders, it is recommended to test dispersant additives as a way to minimize standard deviation.

Further investigations are still needed to better understand the relationship between water absorption, particle size and water demand when recycled CDW fine aggregates and powders are used in cement mixtures.

4 CONCLUSIONS

Based on the results obtained, it can be concluded that:

- The determination of water absorption (WA) for fine recycled aggregates and powders revealed high variability among the evaluated methods, confirming that the measurement technique significantly influences the results.
- According to the Standard Method – NBR 16916, fine recycled aggregates exhibited WA values between 10 and 11%. In contrast, the thermogravimetric analysis (TGA) method yielded slightly higher values, ranging from 12 to 22%, depending on the material's characteristics.
- Using Oliveira's method (2022), recycled powders of mixed origin exhibited WA values between 25 and 32%, while those derived from recycled concrete ranged between 25 and 28%.
- With the TGA use, mixed recycled powders presented values between 20 and 24%. However, for the recycled concrete powders, saturation time influenced the property (anhydrous cements' hydration), and the use of shorter saturation times (0.5 hours, 2 hours) is recommended, depending on the application.
- Among the methods evaluated, TGA proved to be an efficient tool for assessing CDW fine recycled aggregates and powders WA, with less variability in results in general, as the SSD condition determination is performed graphically after testing, eliminating sample handling during measurement and reducing operator interference.
- The findings demonstrate that thermogravimetric analysis can serve as a promising thermoanalytical method for characterizing fine recycled materials. Its quantitative, reproducible, and operator-independent nature makes it a valuable approach for future standardization efforts and for enhancing the accuracy of mixture design and quality control in sustainable cementitious systems.

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Supplementary Information (SI)

This supplementary material presents an exploratory review of studies on water absorption methods for recycled CDW aggregates and powder.

Table S1- Studies in literature on water absorption of recycled CDW aggregates and powders

Authors	Method applicability			Study object	Main findings
	Coarse	Fine	Powders		
Mechling, Lecomte and Merriaux (2003) [49]			X	Mineral addition absorption measurement	i) The method (adapted oven with scale) is reliable; however, operational precautions are required, particularly concerning uniform distribution of granular mat on the bottom of the flat tray and the oven support horizontality. (ii) The method was used on sand and mineral additions to validate the suggested method.
Tam <i>et al.</i> (2008) [35]	X			Alternative method to determine density and water absorption in real time - Proposal	(i) The time required to fully saturate recycled aggregate depends on the mortar adhered to the particle. (ii) The WA measured in a pycnometer is higher in the first 5 hours (representing up to 80% of total WA).
Tegguer (2012) [33]	X			Hydrostatic weighting method proposal	(i) The water absorption coefficient of recycled aggregates after 24 h immersion produces approximately 60% and 70% of total water absorption obtained after 85 h and 110 h immersion for fractions 12,5–20 mm and 5–12,5 mm, respectively. (ii) The values obtained by NF EN 1097-6 method, with water elimination with a cloth, presented values for WA slightly higher than the hydrostatic weighing approach; The adsorbed water elimination by the cloth is less accurate for small aggregates with irregular shapes such as recycled aggregates.
Rodrigues, Evangelista and De Brito (2013) [32]		X		Alternative method to determine density and water absorption of recycled fines – Proposal	(i) The sodium hexametaphosphate solutions use in the WA test minimizes the cohesion between particles and helps to release trapped air. (ii) The WA given by hydrostatic balance is slightly lower than that of the proposed method (use of pycnometer and solution containing the dispersant).
Belin <i>et al.</i> (2014) [77]	X			Continuously assess WA of recycled aggregates based on hydrostatic measurement	(i) The 24-hour WA can be viewed as the sum of the residual cement paste and the initial natural aggregate capillary absorption. (ii) Recycled aggregates may show similar 24h absorption values but very different absorption kinetics. This kinetics appears to depend strongly on the porosity of bonded paste (original concrete).
Gentilini, Yazoghli-Marzouk and Delmotte (2015) [50]		X		WA determination of recycled fines containing particles <0,15 mm	(i) WA values Lack of reliability When determined in the presence of fines according to the cone test procedure. (ii) For recycled concrete fines containing particles < 0.15 mm, the use of evaporimetry indicated higher WA values than those determined in the absence of powders according to the cone test.
Rueda <i>et al.</i> (2015) [36]		X		Development of a fast and reliable method for testing recycled fines WA	(i) They established a correlation between WA after 10 minutes and 24 hours in water (91%); (ii) The TGA use to determine WA provides a quick (30 minutes) and simple test, without the need for an operator.
Bendimerad, Roziere and Loukili (2015) [78]	X			Investigates and improves existing	(i) The results of pycnometer method and hydrostatic weighing showed a very good correlation.

				procedures for determining WA	(ii) The WA between the immersion time and the first measurement is significant and should therefore be considered. (iii) The WA continued to increase between 24 hours and the end of tests, i.e. 7-10 days, but this increase is not significant for concrete mix design.
Damineli <i>et al.</i> (2016) [79]	X			Explore the combination of vacuum immersion saturation and microwave drying kinetics	The combination of vacuum saturation for 0.5 h and microwave oven drying kinetics for 2 hours is a fast method with similar results to ASTM C127, making it attractive for quality control operations.
Quattrone <i>et al.</i> (2016) [40]	X		-	Methods comparison	(i) The vacuum use during immersion is the best solution to reduce test time or increase the saturation level. (ii) Saturated Surface Dry (SSD) condition determination can be performed by means of centrifuge, air flow drum, laser scattering and microwave drying evaporimetry, without significant differences.
Yacoub, Djerbi and Fen-Chong (2018) [80]		X		Propose a new method to measure WA	The results showed that recycled fines cannot be fully saturated by full immersion or pre-saturation. They suggested vacuum saturation combined with an oven evaporation method, however this is unsuitable for construction sites.
Naël-Redolfi, Keita and Roussel (2018) [81]		X		Evaluation of drying kinetics of non-porous and porous particles, real and model, with various sizes and morphologies	(i) The water between particles evaporates at a constant drying rate until there is a drastic drop-in drying rate. (ii) Evaporative methods are not suitable for all porous particles but should work well for fine crushed sand and recycled sand.
Li <i>et al.</i> (2019) [38]		X		Propose a new method to determine fines WA based on pastes WA	(i) The WA for fines in the past was always lower than its WA at the same immersion time. (ii) The WA in the paste is related to its property and mortar mix proportions.
Théréne <i>et al.</i> (2020) [82]	X			Evaluate temperature impact during drying stage in WA	(i) A one-third increase in WA by the internal evaporation method was observed between drying at room temperature (30°C and low relative humidity) and drying at 105°C due to ettringite dehydration. (ii) The use of 105°C temperature leads to a systematic overestimation of water absorption.
Duan <i>et al.</i> (2022) [34]	X	X		Methods presented in the literature - Review	(i) RA absorption has four notable features, including higher capacity, higher standard deviations, and time and size dependence. (ii) the physical principles of evaporation, airflow drying, pycnometer, hydrostatic equilibrium and centrifugation are almost perfect. Although, infrared, conductivity and extrapolation are easy to operate, their physical principles need to be improved.
Sosa <i>et al.</i> (2023) [37]		X		WA determination of different particle size ranges in natural and recycled aggregates by electrical conductivity method	The results show that the paste content is the main variable influencing RA's WA, while the particles size and shape have no noticeable influence on measurements made by electrical conductivity method.

3.3 ARTIGO (ORIGEM)

O artigo 3 sob o título *Carbonatação acelerada de pós de resíduos da construção: uma estratégia de economia circular para mitigação de CO₂ em materiais cimentícios*, foi submetido a periódico da área. O objetivo do estudo foi investigar o potencial de fixação de CO₂ por diferentes pós de RCD para aplicação como material cimentício suplementar.

Foram avaliados pós, de origem mista e de concreto com três diferentes distribuições granulométricas, que foram submetidos à mineralização por carbonatação acelerada por 2, 8, 24, 48 e 168 horas a uma temperatura de 23 ± 2 °C, $60 \pm 10\%$ de umidade relativa e uma concentração de 15% de CO₂. As propriedades físico-químicas dos pós pré e pós mineralização foram avaliadas por meio de técnicas analíticas (TG/DTG, DRX, FTIR, MEV-EDS) e a fixação de CO₂ foi quantificada com base na análise termogravimétrica. Os pós carbonatados foram aplicados em argamassas considerando um nível de substituição de 25% de cimento Portland, que foram avaliados quanto às emissões de CO₂ e resistência à compressão aos 28 dias.

Foi verificado que as variáveis do processo de mineralização de CO₂ nos pós de RCD e a origem do pó são mais relevantes do que a distribuição granulométrica para a fixação de CO₂. Pós de concreto podem fixar entre 14,7 e 24,1 kg.CO₂/t, enquanto os pós do tipo misto, no máximo, 5,4 kg.CO₂/t. A aplicação de pós de concreto, quando mineralizados por 2 horas, com um nível de substituição de cimento de 25%, levou a uma redução de 24% na emissão de CO₂ por m³ de argamassa, quando comparado à aplicação de um cimento do tipo CP II F. A aplicação da carbonatação acelerada para a mineralização de pós de RCD, com vista ao uso como MCS, apresentou resultados promissores, destacando a reciclagem de resíduos, a redução das emissões associadas ao cimento Portland e uma alta relevância para a economia circular e o mercado de carbono.

Apresenta-se, a seguir, o artigo completo, enviado ao periódico indexado na base Scopus, passível de modificações conforme recomendação dos revisores para publicação.

ACCELERATED CARBONATION OF CONSTRUCTION WASTE POWDERS: A CIRCULAR ECONOMY STRATEGY FOR CO₂ MITIGATION IN CEMENTITIOUS MATERIALS

Highlights

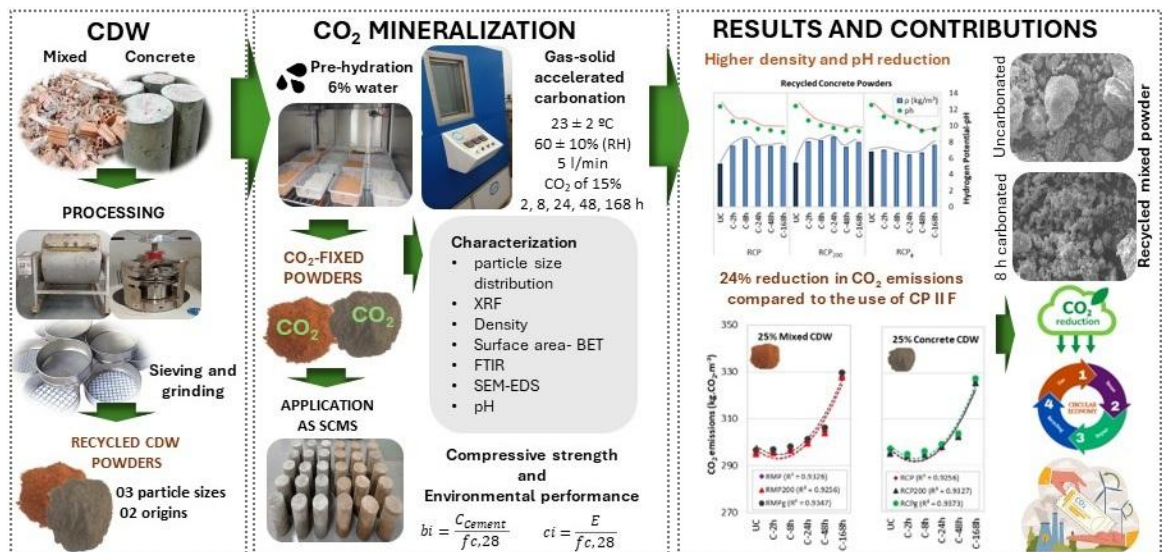
- Mixed and concrete powders of different particle sizes mineralized with CO₂ via gas-solid process.
- CO₂ mineralization in construction and demolition waste (CDW) powders induces physicochemical and morphological changes.
- Powder origin has greater influence on CO₂ mineralization than particle size.
- CO₂ fixation potential of recycled concrete powders ranges from 14.7 to 24.1 kg CO₂ per ton.
- Replacing 25% of cement with recycled concrete powders reduced CO₂ emissions by up to 24% per cubic meter of mortar compared to CP II F 32.

Abstract

This paper investigates the carbon dioxide (CO₂) fixation potential of different Construction and Demolition Waste Powders (CDWP) for application as Supplementary Cementitious Material (SCM). It was evaluated powders from a mixed and concrete source with three different particle size distributions that were submitted to mineralization by accelerated carbonation for 2, 8, 24, 48, and 168 hours at a 23 ± 2 °C temperature, 60 ± 10% relative humidity, and a 15% CO₂ concentration. The pre and post treatment powders physical-chemical properties were evaluated through analytical techniques (TGA, XRD, FTIR, SEM) the CO₂ fixation was quantified based on thermogravimetry analysis. The carbonated powders were applied in mortars considering a 25% Portland cement replacement level, which were evaluated for CO₂ emissions and compressive strength at 28 days. It was verified that the CO₂ fixation process and powder source are more relevant than particle size distribution. Concrete powders could capture between 14.7 and 24.1 kg.CO₂/t when mixed type captures, at maximum, 5.4 kg.CO₂/t. Concrete powders application, when mineralized for 2 horas, with a 25% cement replacement level, lead to a 24% reduction in CO₂ emission per m³ of mortar, when compared to the application of a CP II F cement type. The CO₂ application for CDWP mineralization for application as SCM showed promising results, highlighting waste recycling, reduction in Portland cement associated emissions and a high relevance for both circular economy and carbon market.

Keywords: Semi-dry carbonation; Thermogravimetric analysis (TGA); Supplementary Cementitious Material (SCM).

Graphical abstract



1 INTRODUCTION

The increase in global warming gases (GWG) emissions like carbon dioxide (CO₂), methane (CH₄) and nitrous oxide (N₂O), highlighted by the industrial Revolution, have been contributing to the global climate crisis, being the results more frequent and intense extreme climatic events, sea-level increasing and other maleficent effects on ecosystems and human health [1,2]. Worldwide, only in 2023 there was emitted 37.15 billion tons of carbon dioxide (CO₂), being the cement industry responsible for approximately 5 to 9% of the total emitted [3,4]. Portland cement is obtained thorough clinker grinding, which is produced based on limestone calcination on furnace at temperatures higher than 1400°C. On this production process 60 to 70% of CO₂ emissions occurs due to a the chemical reaction that converts calcium carbonate (CaCO₃) in calcium oxide (CaO) [5,6]. Since most of the CO₂ comes from raw materials decarbonization, inherent to the process, decreasing GWG emissions and, at the same time, satisfying the increased market need for cement, is a high complex challenge for companies, academia and engineers [7,8].

To face this situation, the global sectoral Roadmap drew seven strategies to reach carbon neutrality in the cement industry until 2050. Inside them, the Supplementary Cementitious Materials (SCM) use to decrease clinker application amount on cement production are highlighted, as Much as the Carbon Capture Utilization and Storage (CCUS) techniques implementation, with potential to reduce about 11 and 42% of CO₂ emissions, respectively [9]. SCM use can also be considered a well-done and satisfactory solution for other environmental issues, like energy saving and problems associated with waste management [10,11] and it is already applied in cement production with examples like fly ash, blast furnace slag and filler. The CCUS technologies, however, due to high costs and complexity [12], are solutions considered to be active for 2030 [13], depending on carbon market related law development in signatory countries for the Paris Agreement. In Brazil, law n° 15.042, that promotes the Brazilian System of Global Warming Gases Market (SBCE), was published in December 2024 [14], but it is still depending on complementary decrees regulation to an effective implementation.

Other sectorial environmental challenges are the Construction and Demolition Waste (CDW) recycling, management and valuation. It is estimated that, globally and yearly, about 10 billion tons of CDW are produced [15], a value that tends to increase due to urban expansion [16] and the building and structures end-of-life that are being demolished or destructed by conflicts and natural disasters [17]. This waste recycling have been studied since that last century, especially as course and fine aggregates replacement for concretes and mortars production, with an abundant bibliography on the subject [18–20]. On the latest decade, studies aiming to use powders as SCM directly obtained from recycled aggregate processing or specific sieving and grinding processes started to arrive [21,22]. These materials, which generally replace Portland cement at a 20% level, have been submitted to treatments (thermal, mechanical and others) and associated to dosage methods, leading to an amplified recycling rate [23–25] allowing up to 45% Cement replacement [26]. More recently, CO₂ use focusing on particles and carbon dioxide fixation improvement by mineral carbonation process have been highlighted as a promising approach for CDW appreciation, being align with circular economy principles [27,28].

Under these circumstances, CCUS Technologies like cementitious waste mineralization play a leading role in CO₂ emissions mitigation processes. As per the literature, CO₂ fixation for Recycled Concrete Aggregates (RCA, Ø > 4.8 mm) vary between 4.9 to 8.1 kg.CO₂/ton [29]. For Recycled Concrete Fines (RCF, 4.8 mm < Ø > 0.15 mm) between 8.6 and 16.3 kg.CO₂/ton [30,31] and 20 kg.CO₂/ton [32], being observed a growth tendency to CO₂ fixation as the particle size decreases [33,34]. In Recycled Concrete Powders (RCP, Ø < 0.15 mm) Kaliyavaradhan; Li; Ling [27] obtained a CO₂ fixation between 0.30 to 28.10 kg.CO₂/ton, inferior values when related to studies that use smaller powders from hydrated cement, which had a higher CaO content and were naturally exposed to CO₂ and which carbon fixation were reported as 115 to 270 kg.CO₂/ ton [35–37].

Although accelerated carbonation mineralization have been an object for several studies related to cementitious waste [38–41], most papers focused on hydrated cement paste powders [41,42]. Investigations are scarce when with powders obtained from inert materials due to fine and coarse aggregates presence [27,43,44]. Considering that, in Brazil, about 87% of the CDW received in recycling plants are the mixed type (mixed between cementitious, ceramic, soils, and others materials) [45], this study aimed to evaluate different CDW powders behavior when submitted to mineralization by Accelerated carbonation, considering its potential future application as supplementary cementitious materials in partial replacement to Portland cement. By addressing materials with a representative composition of what is actually received at CDW recycling plants, this research seeks to build technical knowledge applicable to fixed CO₂ quantification, expanding the potential understanding of these residues valorization and their role in emissions reduction with the construction sector, with a view to carbon credits.

2 MATERIALS AND METHODS

CDW powders were evaluated for CO₂ fixation potential and for application as a SCM by potential mortars production application. Initially, powders obtention processes are presented, followed by characterization for both recycled powders and Portland cement applied as reference material for mortars production. The powders were submitted to Accelerated carbonation mineralization and, again, characterized. In the end, mortars were molded to performance evaluation regarding compressive strength and environmental indexes analysis.

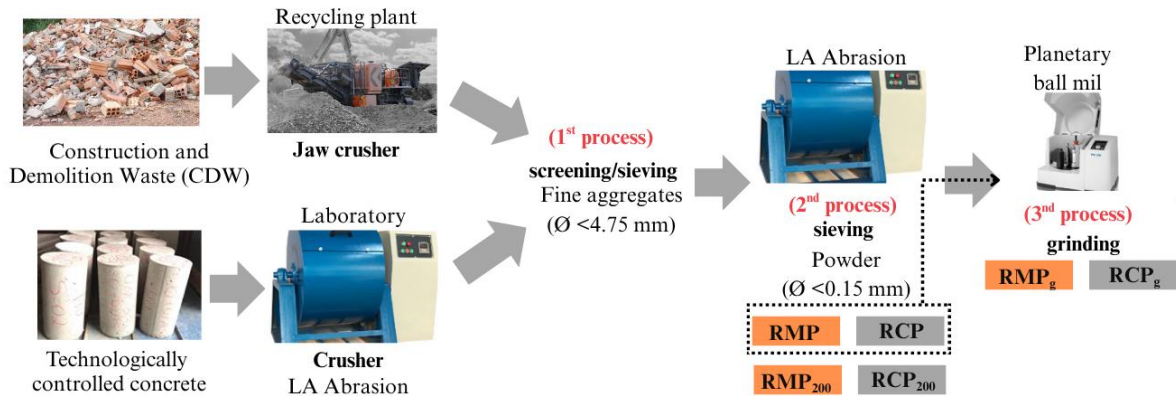
2.1 RECYCLED POWDERS

2.1.1 CDW Obtention and recycling process

The Construction and Demolition Waste (CDW) recycled powder were obtained through fine aggregates processing that belong to “Class A” according to resolution N° 307 [46], as presented in Figure 1 and Table 1.

The mixed recycled powder came from an industrial process for coarse and fine aggregates from CDW production (1st process), where were collected the fraction passing through Mesh 4 ($\varnothing < 4.75$ mm). According to gravimetric characterization [47] the mixed type waste is constitute by 50% hardened paste particles, 12% rocks, 30% red ceramics fragments with non-enameled surface, and 8% unwanted fragments of other materials. The recycled concrete powder was obtained by concrete specimens recycled after technological control tests, which processing occurred in a Los Angeles abrasion equipment, on laboratory environment (1st process), being reserved the fraction passing Mesh 4 ($\varnothing < 4.75$ mm) for following processing.

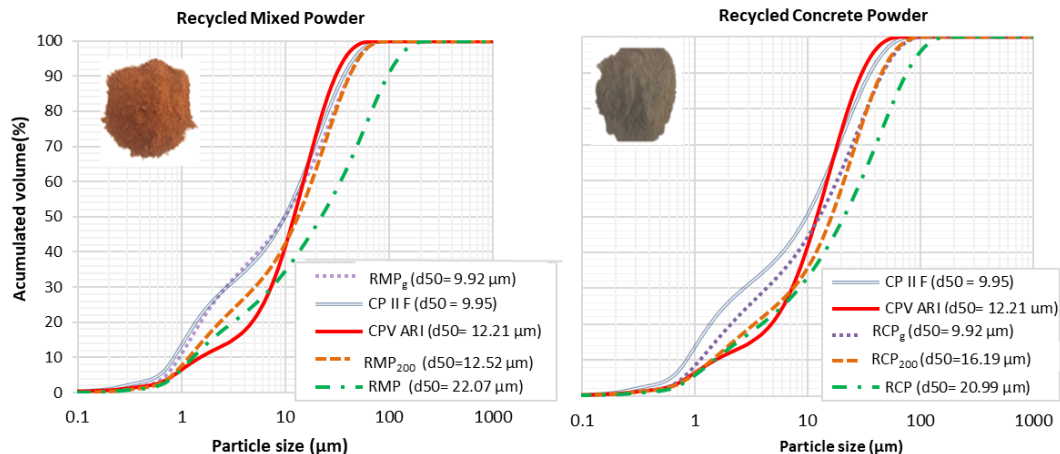
On the 2nd process, fine aggregates were processed for 2 hours on LA Abrasion equipment reaching, by sieving, the materials RMP and RCP (passing through Mesh 100) and RMP₂₀₀ and RCP₂₀₀ (passing through Mesh 200). Retained material was disposed. On the materials processing 3rd step, approximately 50% of mixed and concrete powders passed thorough Mesh 100 ($\varnothing < 0.15$ mm) were processed for 0.5 hours in planetary balls mill PM 100 (Retsch) leading to materials RMP_g and RCP_g. Comminution was conducted with 60g samples, in agata jar with 250 cm³, 50 agata spheres with an approximated diameter of 10 mm, 500 rpm rotation and e automatic flow inversion every 15 min with a 1-minute break for cooling.

Figure 1- Processes for recycled powder obtention**Table 1-** Powders identification

CDW origin	Designation	Description	Process*	
			Step	Equipment
Mixed	RMP	Recycled Mixed Powder (sieved - mesh 100)	2 nd	LA Abrasion/ Sieve
	RMP ₂₀₀	Recycled Mixed Powder (sieved - mesh 200)	2 nd	LA Abrasion/ Sieve
	RMP _g	Recycled Mixed Powder (grinding)	3 rd	Planetary ball mill
Concrete	RCP	Recycled Concrete Powder (sieved - mesh 100)	2 nd	LA Abrasion/ Sieve
	RCP ₂₀₀	Recycled Concrete Powder (sieved - mesh 200)	2 nd	LA Abrasion/ Sieve
	RCP _g	Recycled Concrete Powder (grinding)	3 rd	Planetary ball mill

* The processes are shown in Figure 1.

Recycled powder particle size distribution, just like Portland Cement type CP V ARI (High early strength Portland cement), equivalent to ASTM C 150/C150M Type III [48] and CP II F (Filler Portland Cement, FPC), equivalent to ASTM limestone cement (IL) [48] employed on mortar samples production in this study were determined by laser diffraction (Figure 2). The tests were conducted three times in equipment Cilas 1190 with reading range from 0.04 to 2500 μm , in liquid environment (distilled water) with no dispersant and obscuration index of approximately 25% [49].

Figure 2- Particle size distribution: a) Recycled Mixed Powder; b) Recycled Concrete Powder

It is noticed that the d50 diameter from RMP and RCP are larger than the others (RMP>RCP>RCP₂₀₀>RMP₂₀₀>RCP_g>RMP_g), indicating efficiency in 2nd and 3rd processes adopted to different particle sizes obtention (Figure 1). The powders and cements oxides composition were determined by X-Ray Fluorescence (XRF) in WDS Bruker S8 Tiger spectrometer, equipped with Rh tube. For larger elements samples, the calibration was made with a GeoQuant T® package from Bruker, composed of rocks and natural minerals patterns (Table 2).

Table 2- Powders and cement oxide composition (wt. %)

Materials	Chemical composition (%)										
	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	SO ₃	Na ₂ O	MnO	P ₂ O ₅	LOI
CP II F	19.20	54.05	4.50	2.61	5.25	0.61	2.45	0.15	<LQ	<LQ	10.54
CP V ARI	19.85	60.84	4.88	2.66	1.83	0.54	3.58	0.19	0.11	0.23	5.64
RMP ₁₀₀ e RMP _G	39.55	16.68	12.88	6.65	3.64	1.0	0.45	0.28	<LQ	0.11	17.08
RMP ₂₀₀	33.48	17.42	12.97	9.18	3.95	0.52	0.80	0.27	<LQ	0.14	18.76
RCP ₁₀₀ e RCP _G	47.20	21.36	8.56	6.94	3.28	2.14	0.62	1.36	0.13	0.22	7.84
RCP ₂₀₀	43.07	20.79	9.50	8.75	3.37	0.99	0.96	1.58	0.15	0.25	8.91

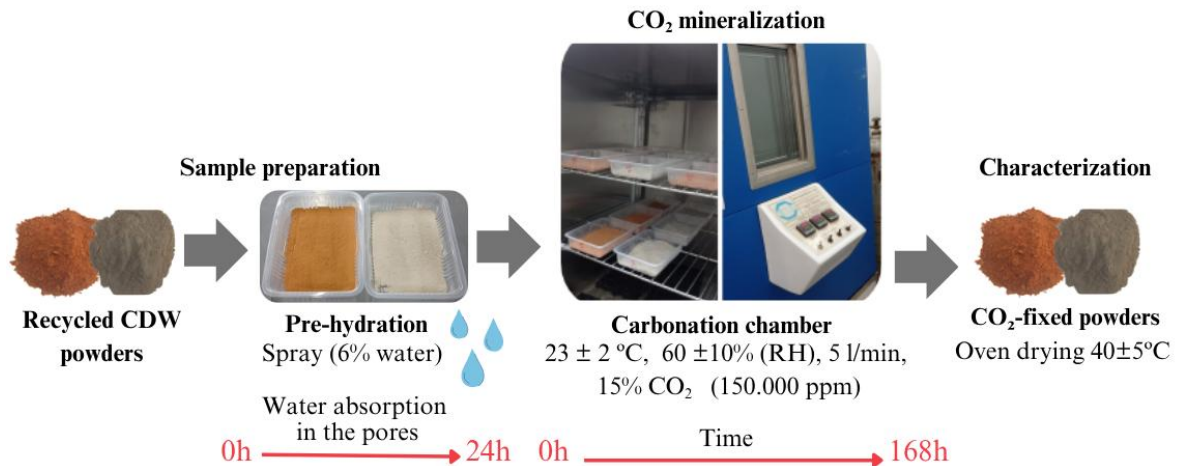
LOI: Loss on Ignition; <LQ: below the quantifiable limit.

It is noted (Table 2) that concrete powders had a greater silica (SiO₂) and calcium oxide amount (CaO) than mixed type, what can be justified by the stronger natural sand and hydrated cement compounds presence (concrete and mortars) [26,50,51]. The Loss on Ignition (LOI) is related, mainly, to the aggregates hardened cement paste presence (phases with CaO presence) and phyllosilicates from clay-minerals [50,52], being higher in the mixed type powder.

2.1.2 CO₂ mineralization via accelerated carbonation

The CDW powders CO₂ mineralization (by carbonation) was conducted in a static process, gas-solid, in accelerated carbonation chamber from BASS, model UUC-RH-STD-CO₂-1000/2016, according to the workflow presented in Figure 3.

Figure 3- Workflow for carbonated powders preparation



About 250g powder sample were placed in a recipient with a 1cm depth layer to facilitate CO₂ diffusion between particles. Considering that the CO₂ chemical reaction efficiency with hydration products from powders depends on materials humidity [53,54], it was necessary an water spray at 6% weight (pre-hydration) [55]. After water application, powders were moved for humidity uniformization and kept in a closed environment for 24 hours, made for particles humidity balance achievement. Passed this period, they were placed in the carbonation chamber at 23 ± 2 °C, $60 \pm 10\%$ relative humidity, 5 l/min CO₂ flow and CO₂ concentration at 15% (150.000 ppm), for 2, 8, 24, 48 and 168 hours. For each period of interest, after leaving the CO₂ chamber, powders were placed in a furnace at 40 ± 5 °C for 48 hours to avoid ettringite dehydration [56–58]. Finally, samples were kept in hermetic closed plastic recipients for posterior application and characterization.

2.1.3 Physico-chemical characterization

Every powder was characterized before and after accelerated carbonation mineralization. Density test was performed in a helium gas equipment (He), model Ultrapyc 5000, from Anton Paar, with operational pressure of 10 psi [59]. The Specific Surface Area (SSA) by the Brunauer-Emmett-Teller (BET) method was determined by nitrogen adsorption in a Micromeritics TriStar II Plus Analyzer Version 3.0, with degas at 160 °C for 8 hours in vacuum. The pore volume was obtained through isothermal adsorption $P/P_0 = 0.95$, assuming complete pore saturation [25,60].

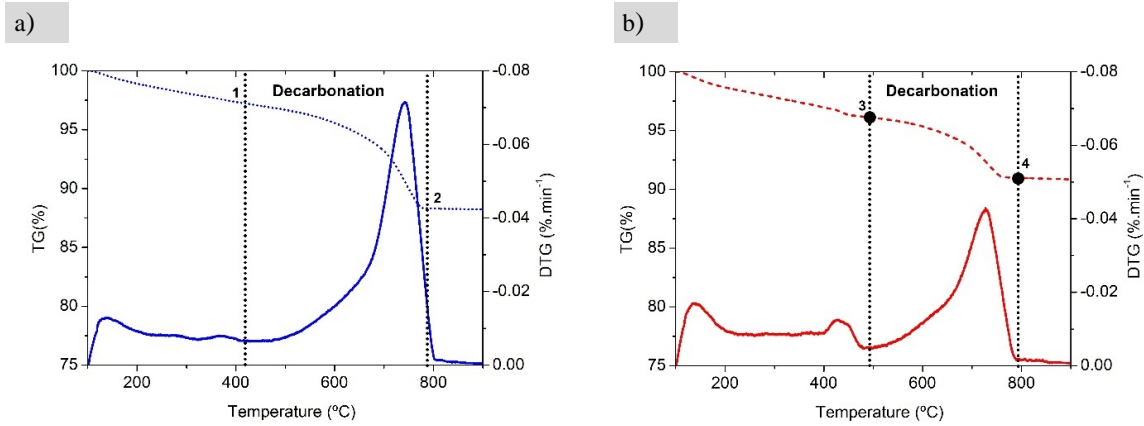
The mineralogical composition analysis was investigated based on X-ray diffraction (XRD) method by pore method, in diffractometer (Panalytical) with θ - ρ Bragg-Brentano configuration, Cu K α radiation and $\lambda = 1,5418$ Å, operating at 20 mA, 40 kV. The peaks were analyzed in an angular range from 5° to 100° in a 26-minute period. Diffractogram interpretation was made in a X'Pert High Score Plus software V4.8 from PANalytical [61] comparing results to COD (Crystallography Open Database).

The spectra from Fourier Transform Infrared Spectroscopy (FTIR) on infrared region were registered using a PerkinElmer spectrometer model Frontier, on mode Attenuated Total Reflectance (ATR), by the pellets KBr model. For each sample, 1 mg powder was mixed with a 100 mg KBr and pressed to pastille preparation. The scanning transmission was made with a 4 cm⁻¹ resolution in the region between 4000 cm⁻¹ to 600 cm⁻¹.

Morphological characterization was made by Scanning Electron Microscopy (SEM) in a Zeiss – EVO MA10 equipment, with imagens detection through secondary electrons and Energy Dispersive X-Ray Spectroscopy (EDS). Powders were deposited in carbon tape with posterior gold metallization with a Sputter Coater Quorum SC7620 (15mA for 1:30 minutes). A tungsten filament was used. The potential difference was 10kV. The samples hydrogen ionic potential (pH) was determined using a digital pH meter. To do this, 5g of each sample was diluted in 50ml of deionized water, and the reading was taken after stabilized equipment (approximately after 5 minutes).

2.1.4 Estimation of CO₂ fixation by TGA/DTG

The powders estimative for CO₂ fixation was calculated based on the results obtained from thermogravimetry analysis (TGA) of carbonated (C) and uncarbonated samples (UC). About 10 mg were placed in an alumina crucible on a PerkinElmer analyzer, model STA 6000, with a nitrogen flow of 20 mL.min⁻¹, heating range from 100 °C to 900 °C and heating rate of 20 °C.min⁻¹. With the program OriginPro 8.5 [62] TGA and derivative thermogravimetry (DTG) curves were plotted in percentual weight (Figure 4).

Figure 4- TG/DTG profile examples with data extraction points: a) carbonated (C); b) uncarbonated (UC)

By the thermogravimetry profiles it is possible to quantify a few hydration products by its temperature range: plaster, ettringite and hydrated calcium silicate dehydration (100 °C), calcium hydroxide decomposition ($\text{Ca}(\text{OH})_2$) (380 °C to 520 °C) and calcium carbonate decarbonation (CaCO_3) (520 °C to 800 °C) [63,64]. The mass loss exact limits due to decarbonation were determined using the tangential method [57]. Each sample were analyzed separately, not assuming the same range for all samples since thermal decomposition temperature changes depending on CaCO_3 crystallinity degree [65,66]. Like this, with the decarbonation mass losses quantified, CO_2 fixation were estimated with the Eq. 1 and 2 [65,67–69]. The degree of carbonation (%) was calculated using Eq. 3 [65,67].

$$\text{CO}_{2,\text{fixation}}(\%) = \left((TG_C^1 - TG_C^2) - (TG_{UC}^3 - TG_{UC}^4) \right) \times \frac{MM_{\text{CO}_2}}{MM_{\text{CaCO}_3}} \quad \text{Eq. 1}$$

Where: $TG_C^1 - TG_C^2$: Carbonated sample mass loss by TG (%); $TG_{UC}^3 - TG_{UC}^4$: Uncarbonated sample mass loss by TG (%); MM_{CO_2} : CO_2 molar mass (~44.01 g/mol), MM_{CaCO_3} : CaCO_3 molar mass (~100.09 g/mol).

$$\text{CO}_{2,\text{fixation}}\left(\frac{\text{kg} \cdot \text{CO}_2}{\text{t}}\right) = \frac{\text{CO}_{2,\text{fixation}}(\%)}{100} \times 1000 \quad \text{Eq. 2}$$

$$\text{Carbonation degree}(\%) = \frac{\text{CO}_{2,\text{fixation}}}{\text{Max.capacity of CO}_2 \text{ fixation}} \times 100 \quad \text{Eq. 3}$$

Each materials *Max.capacity of CO_2 fixation* depends on CDW chemical composition and source, primarily the CaO reactivity potential. On this way, with calculated $\text{CO}_{2,\text{fixation}}$ values from TGA, the CaO obtained by XRF, it is possible to calculate the *Max.capacity of CO_2 fixation* with Eq. 4, 5, and 6.

$$\text{Max.capacity of CO}_2 \text{ fixation}(\%) = \text{CaO}_{\text{free}} * \left(\frac{MM_{\text{CO}_2}}{MM_{\text{CaO}}}\right) \quad \text{Eq. 4}$$

$$\text{CaO}_{\text{free}}(\%) = \text{CaO}_{\text{XRF}} - \text{CaO}_{\text{eq.CaCO}_3} \quad \text{Eq. 5}$$

$$\text{CaO}_{\text{eq.CaCO}_3}(\%) = \text{CO}_{2,\text{fixation}} * \left(\frac{MM_{\text{CaO}}}{MM_{\text{CO}_2}}\right) \quad \text{Eq. 6}$$

Where: CaO_{XRF} is the proportional rate of CaO presented in the powders by x-ray fluorescence (Table 1).

2.2 APPLICATION AS SCMS

The potential application as SCM evaluation was conducted for carbonated (C) and uncarbonated (UC) samples by compressive strength. For this, mortars were produced according to the Brazilian standard NBR 7215 [70], equivalent to ASTM C1437-20 [71], with a 25% Cement Portland mass replacement, cement which had a 10% filler composition (CPV ARI) by recycled powders, with a sum of 15 different mixtures. As reference material, a mortar was produced with Filler Portland Cement (CP II F), since this type could have until 25% filler [72]. Mortars were produced with the proportion of 1:3:0.48 (cement: normal Brazilian sand: water/cement ratio), with a mechanic mixer. The normal sand had density of 2.65 kg/dm³, and particle size distribution defined by standard, being employed proportion of 25% for each fraction 1.18 mm, 0.60 mm, 0.30 mm, and 0.150 mm. Compressive strength was determined at 28 days, in hydraulic press (model I-3025-B), with strength rate of 0.15 ± 0.05 MPa/s, adopting the mean value between 4 cylindrical specimens (50 mm × 100 mm) cured in limestone saturated water (3g/l). Results were analyzed with Sisvar Software [73] and submitted to Scott-Knott test.

2.3 ENVIRONMENTAL PERFORMANCE

Recycled powders environmental performance before and after mineral carbonation was evaluated through the indicators: binder intensity (*BI*) and CO₂ intensity (*CI*) [23,74], according to Eq. 7 and 8, respectively.

$$BI \left(\frac{kg}{m^3} / MPa \right) = \frac{C_{Cement}}{f_{c_{28}}} \quad \text{Eq. 7}$$

Where: C_{Cement} : Cement consumption (clinker, calcium sulfate and filler, kg/m³); $f_{c_{28}}$: Compressive Strength at 28 days (MPa).

$$CI \left(\frac{kg.CO_2}{m^3} / MPa \right) = \frac{E}{f_{c_{28}}} \quad \text{Eq. 8}$$

Where: E : Total CO₂ emissions associated to materials consumption (cement and powders) for a 1 m³ de mortar production (kg.CO₂/m³);

Cement and uncarbonated powder emissions were calculated according to equations 9 and 10. For carbonated powders (Equation 11), besides all emissions associated with processes the materials were submitted (Table 1), emissions associated with electric energy use for the carbonation chamber were added and the recorded fixed CO₂ was subtracted (Table 3).

$$E_{cement} \left(\frac{kg.CO_2}{m^3} \right) = C_{Cement} * E_{Cement} \quad \text{Eq. 9}$$

$$E_{powder,NC} = (C_{powder,NC} * E_{processing}) \quad \text{Eq. 10}$$

$$E_{powder,C} = (C_{powder,C} * E_{processing}) + (C_{powder,C} * E_{Carbonation\ chamber}) - (CO_{2,fixation}) \quad \text{Eq.11}$$

Where: C: Materials consumption (cement and powder) for a 1 m³ mortar (kg/m³); E: CO₂ emissions for cement and powder production (kg.CO₂/t); $E_{processing}$: CO₂ emissions for sieving and grinding processes in powder production (kg.CO₂/t); $E_{Carbonation\ chamber}$: CO₂ emissions for electric energy production for the carbonation chamber operation (kg.CO₂/t); $CO_{2,fixation}$: Fixed CO₂ on powders mineralization (kg.CO₂/t), obtained by Eq.2.

On the CO₂ calculus related to the carbonation chamber energy consumption, it was considered a mean annual emissions factor in 2024 ~46kg.CO₂/MWh (0.046 kg.CO₂/kWh) informed by the ministry of science, technology and innovation [75]. It was admitted a 5 kW [76] power capacity and a production effectiveness of 150 kg of powder for exposure time.

Table 3-Values for carbon emissions

Data	Value	Reference
$E_{CP\ II\ F}$	762.7 kg.CO ₂ /t (medium value)	SIDAC [77]
$E_{CP\ V\ ARI}$	776.4 kg.CO ₂ /t (10% filler)	SIDAC [77]
$E_{processing\ (2^{nd})}$	6.36 kg.CO ₂ /t	Paz <i>et al.</i> [78]
$E_{processing\ (3^{rd})}$	24.5 kg.CO ₂ /t *grinding period	Oliveira <i>et al.</i> [23]
$E_{carbonation\ chamber}$	1.53 kg.CO ₂ /t * exposure time	Authors (2025)

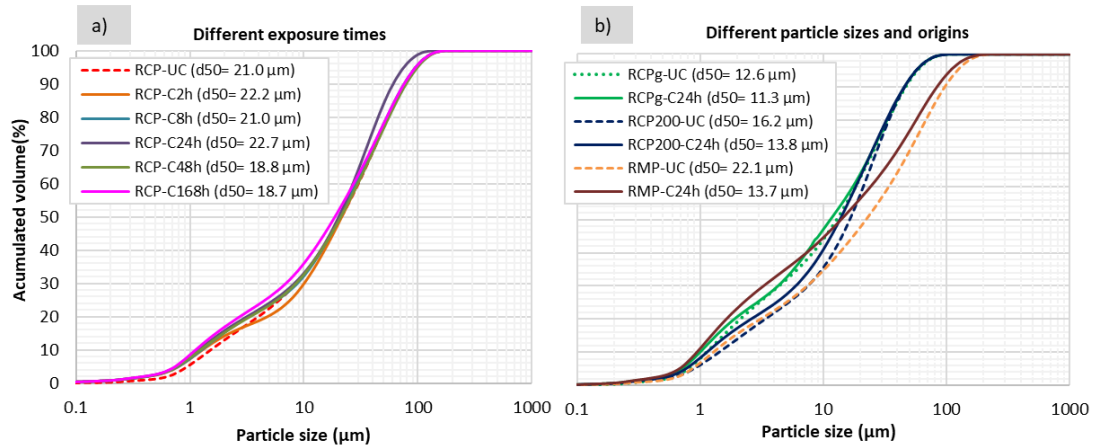
In this study, gas and powders transportation emissions were not considered, nor were the emissions associated with CO₂ in metal cylinders, since these parameters depend on the technological route adopted.

3 RESULTS AND DISCUSSION

3.1 POWDERS PHYSICO-CHEMICAL CHARACTERISTICS

The mineralization effect on powders particle size distribution is shown in Figure 5. It is noted that, for RCP (Figure 5a), the d50 increased with a 24-hour mineralization from 21 µm to 22.72 µm. After this period, there was a reduction, reaching 18.7 µm with a 168-hour process (7 days). With an exception for RCP, all d50 values for 24 hours (5b) had a reduction. For example, for RCP_g it decreased from 12.6 µm to 11.3 µm. For mixed origin powder (RMP), the CO₂ effect seems to be more significant, with a reduction from 22.1 µm to 13.7 µm. An increase in d50 after mineralization is frequently explained in the literature by the following effects: (I) particles agglomeration; (II) CaCO₃ deposition on particles surface; (III) particles linked by silica gel and/or (IV) additional phase hydration on uncarbonated powders [79,80]. Zhao, S. *et al.* [55], observed a d50 increase from 18.65 µm to 33.30 µm in recycled hydrated pastes powder after the mineralization process. Mao *et al.* [81] identified that an increased under dry mineralization process can be attributed to water lack, resulting higher particles' agglomeration. Besides that, the higher the temperature, CO₂ rate and exposure time, the greater will be this effect under particle size distribution [81].

Figure 5 - Powders particle size distribution after mineralization with a) Different exposure times; b) Different particle sizes and origins



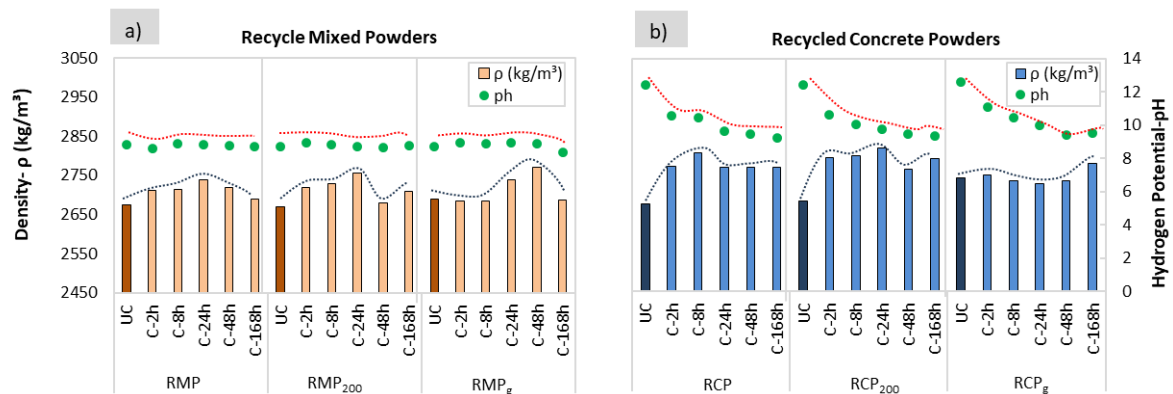
On the other hand, some studies report a d50 decrease due to CaCO₃ crystal formation in a smaller size. According to Mehdizadeh *et al.* [82] and Kaliyavaradhan; Li; Ling [27] the powders structure increase in CaCO₃ content can decrease particles size ranges, improving particle size distribution. Similar results were observed by Lu *et al.* [83], who showed a d50 decrease from 27µm to 24µm after mineralization. The C-S-H decalcification and polymerization can contribute to volumetric particle decrease during prolonged

carbonation. This happens due to chemically linked water consumption during C-S-H mineralization [84,85].

For mixed type powders (RMP) the d50 reduction can be associated with cementitious phases pre-hydration during sample preparation, promoted by water sprinkling. This process could highlight $\text{Ca}(\text{OH})_2$ formation in a greater amount which structure, when developed in an unorganized and superficial form, tends to form fragile aggregates. These agglomerates are easily unmade during drying or dispersion for particle size analysis, resulting in smaller particles and, consequently, a smaller d50 [86].

In Figure 6 density and pH values are shown for mineralized powders. It is observed that for all studied particle sizes, for both mixed and concrete powders, there was a density increase after mineralization. This increase could be related to two main factors: (1) CaCO_3 formation and a polymorph type generated, mainly the calcite in this study condition, that was a larger molar volume, approximately 11% greater than calcium hydroxide (33.2 cm^3) [87] promoting microstructure densification; and (2) particle hydration processes triggered by samples humidification [88,89].

Figure 6- Powders density and pH after mineralization: (a) mixed CDW; (b) concrete CDW



For concrete powders, a more elevated mean increase was verified on density: 3.81% for RCP and 4.18% for RCP₂₀₀, indicating that the particle size reduction positively influenced this property. For RCP_g the increase was only 1.35%. These results could be related to a higher difficulty in CO_2 diffusion on smaller particles due to agglomeration, a common phenomenon on this kind of material [90,91]. Besides that, larger particle size could have favored a faster CO_2 fixation, promoting particles superficial densification, limiting mineralization advance through particles' center [42,92,93]. According to Jiang *et al.* [94], smaller particle sized showed, after mineralization, a larger CaCO_3 content when compared to larger particles. This tendency was verified on mixed source powder, with a mean increment of 1.49, 1.81, and $0.90 \text{ kg}/\text{m}^3$ for RMP, RMP₂₀₀ and RMP_g.

The mineralization period influenced changes in density, however, a linear relation between CO_2 exposure time and this property increment was not observed. The results suggested that variables like pore water and consequently CaCO_3 formation can differently on each powder type, individually affecting its physico-chemical characteristics. As reported by Zajac *et al.* [95], water content has a dominant impact on mineralization extension kinetics. Other factors to improve carbonation reaction kinetic, which are controlled by CO_2 diffusion, are an increase in CO_2 pressure or concentration and an increase in the process' temperature ($20\text{-}60 \text{ }^\circ\text{C}$) [96].

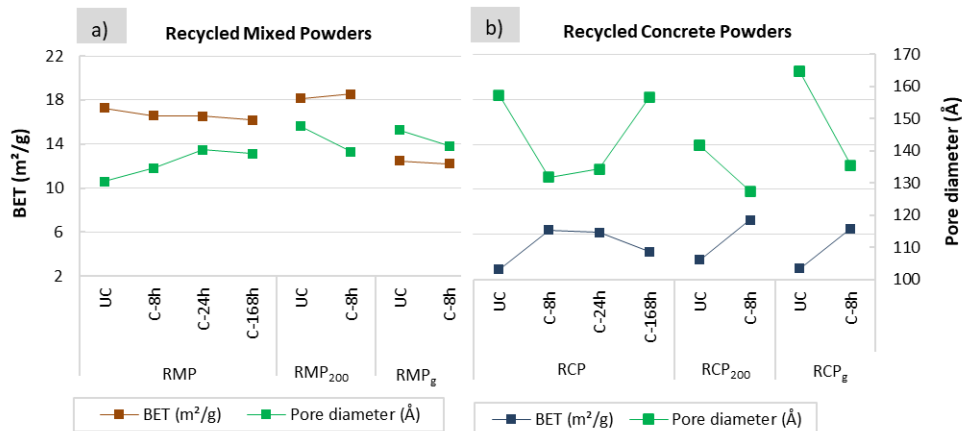
Analyzing pH alterations, it is observed that with mineralization a pH reduction occurred in concrete powders. For the mixed type, values remained between 8.5 and 9, while for concrete ones the results were

12.5 and 9.9, considering different exposure periods and particle size distributions. This reduction could be associated to hydration phases dissolution and simultaneous CaCO_3 precipitation [97–100]. Shen *et al.* [92], in a gas-liquid process, verified calcite crystals growth in paste powders before 20 minutes, while amorphous alumina gels and silica seemed to precipitate mainly after 25 and 30 minutes, respectively, while the pH decreased from 12.67 to 6.90. pH values lower than 8 indicated that the cement hydrates are completely carbonated, which suggests that there is a remaining CO_2 fixation potential on evaluated powders [101].

As verified in density, pH reduction on recycled powders also did not present a linear behavior, independent from particle size, showing a stabilization tendency between 24 and 48 hours. This pH decrease must not be considered a limitation for mineralized powders application on steel concrete structures, especially when the minerals formed contribute to materials desired characteristics, consequently improving its efficiency as a supplementary cementitious material [102].

The superficial surface area (SSA-BET) increased with concrete powder mineralization, resulting in a mean pore diameter reduction. In contrast, mixed source powders represent the opposite result, in exception to RMP_{200} , that also registered an increase, possibly due to a greater CaO available amount when compared to other particle sizes (Table 2). As presented in Figure 7, the RCP, for example, went from $2.91 \text{ m}^2/\text{g}$ to $6.50 \text{ m}^2/\text{g}$ after an 8-hour mineralization process, with correspondent decrease in mean pore diameter, tendency also observed in the other particle size distributions. This behavior is attributed to calcite crystals formation during the carbonation process [27]. The calcium hydroxide carbonation leads to a decrease in mean pore diameter, since calcium carbonate volume is larger than calcium hydroxide's [103]. By the time when the exposure time was extended to 168 hours, an increase in pore diameter and a decrease in SSA-BET was observed, the latest reached $3.69 \text{ m}^2/\text{g}$. This behavior is associated with portlandite carbonation and consequently C-S-H decalcification [92,104,105]. RPM ($17.29 \text{ m}^2/\text{g}$), which has more irregular and more porous particles due to CDW source [24,26], presented specific surface areas, posterior to the mineralization, of 16.55 and $16.17 \text{ m}^2/\text{g}$ after 8 and 168 hours. A huge values range can be found in the literature, for recycled concrete (0.6 a $12.8 \text{ m}^2/\text{g}$) and ceramic waste powders (1.1 e $17.0 \text{ m}^2/\text{g}$). These variations are associated with mechanical processing applied, CDW composition and source, tests protocols and others [21,22,24].

Figure 7- Specific Surface Area (BET) and pore diameter: a) mixed CDW; b) concrete CDW



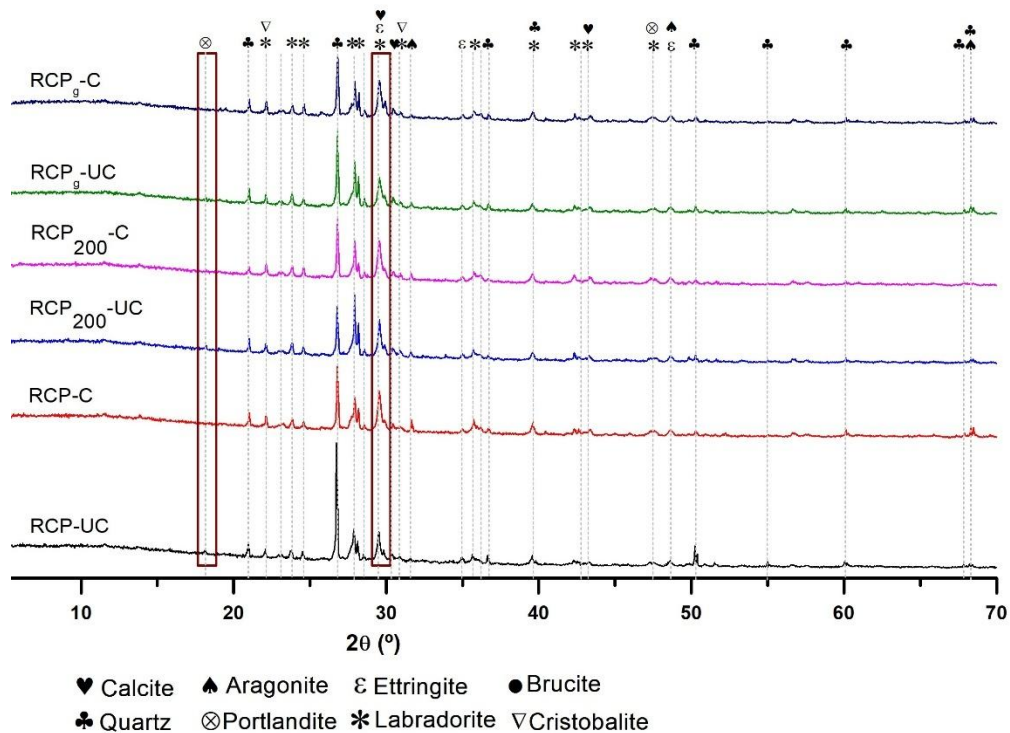
Generally, powders physico-chemical properties alterations, like particles diameter (d_{50}), density, pH, specific surface area (SSA-BET) and pore distribution, indicated that the mineralization effects are more pronounced in materials made from concrete CDW. It was observed that particles densification and increase on SSA occurs until a given mineralization period which suggests that variables like water content, temperature and CO_2 pressure need to be optimized to highlight process efficiency. pH reduction

(alkalinity), for values above 8, indicate that there is CO₂ potential fixation on these particles. For powders obtained in the 3rd process (RMP_g e RCP_g), particles agglomeration seems to have made it difficult for CO₂ diffusion, resulting in a lower densification when compared to RMP e RCP.

3.2 MINERALOGICAL, MORPHOLOGICAL AND FTIR ANALYSIS

XRD data are shown in Figure 8 for uncarbonated (UC) and 168-hour carbonated (C) recycled concrete powders. The main crystalline phases verified are quartz (SiO₂), portlandite (Ca(OH)₂), labradorite (feldspar), cristobalite (a silica polymorph), calcite, aragonite (both calcium carbonate polymorphs) and ettringite. Feldspar came from a basaltic rock commonly used in the region that is also applied as an artificial fine aggregate from concrete production. All phases are consistent with the patterns found on literature [21,24,26]. Calcite and aragonite, CaCO₃ polymorphs, are a gas-liquid mineralization evidence [104]. It is observed that with a decrease in particle size (processes indicated on Table 1), the quartz-related diffraction peak intensity decreases. Quartz has a high elastic modulus and it is present in larger fractions [106], this way, the restriction in particle size distribution on RCP₂₀₀ and the grinding in high-efficiency mill on RCP_g justify these results. It is observed that powders mineralization leads to a portlandite peak absence and a smooth increase in calcite related peak, for all particle sizes, confirming powders chemical interaction with CO₂, supporting data found on the literature [83,106].

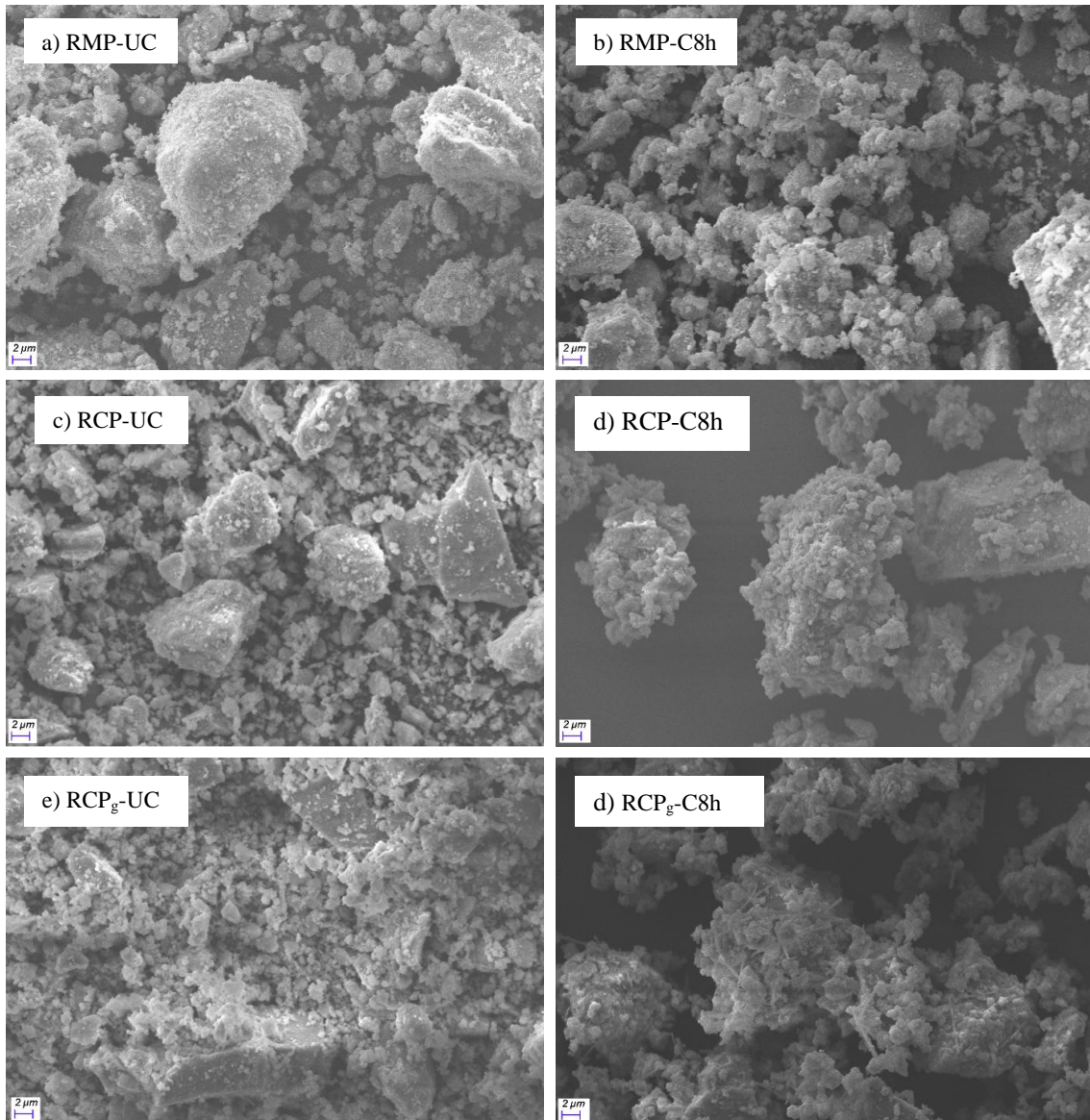
Figure 8- Recycled concrete powders XRD diffractogram after a 168h mineralization



Morphology before and after mineralization is shown in Figure 9. RMP is composed of particles with edges and corners, and of fine particles adhered to the surface [24,25,107], justifying the higher SSA-BET, while RCP presented an irregular and angular format [27,108]. Analyzing carbonated powders during an 8-hour mineralization (C-8h), agglomerated CaCO₃ crystals are observed being formed on the particles surface [92,109]. For RCP_g surface perpendicular crystal formations were visualized, characteristics to aragonite [110]. Aragonite and vaterite formation could be related to highly densified hydrated calcium silicate presence due to a high CO₂ concentration (higher than 10%) [111–113]. This suggests that the particle size

reduction, with higher SSA could have benefited C-S-H carbonation. In the face of the mixed powder (RMP), denser agglomerates are seen in concrete powders (RCP e RCP_g), indicating that the chemical composition indeed influences CO₂ fixation.

Figure 9- Scanning electron microscopy (SEM) of CDW powders (Mag.= 5.00 K X): a) RMP-UC; b) RMP-C8h; c) RCP-UC; d) RCP-C8h; e) RCP_g-UC; c) RCP_g-C8h

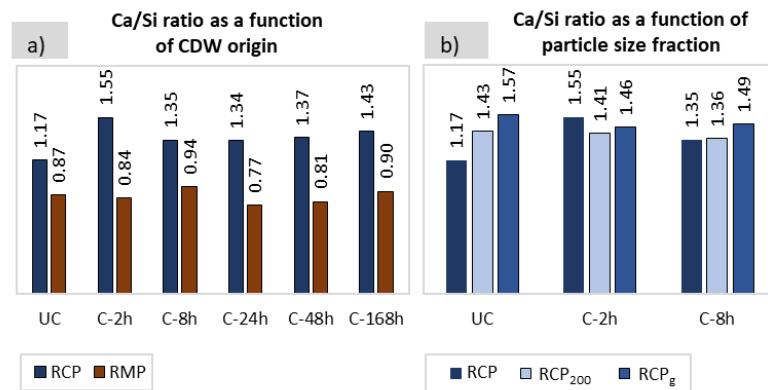


To analyze amorphous silica gel formation, one of the final mineralization products [41,114,115], it was accounted for the calcium-silicate ratio (Ca/Si) obtained by SEM-EDS (Fig 10). It is verified that, for concrete powders (Figure 10a), mineralization increased Ca/Si ratio, varying between 1.24 and 1.55, with the highest data recorded after a 2-hour exposure (C-2h). However, for mixed powders (RMP), values were between 0.77 and 0.94, occurring a rate reduction after 2 hours.

By analyzing different concrete powder particle size distribution (Figure 10b), it is noted that grinding and sieving processes resulted in a higher Ca/Si, with higher values for smaller particles (RCP_g). After

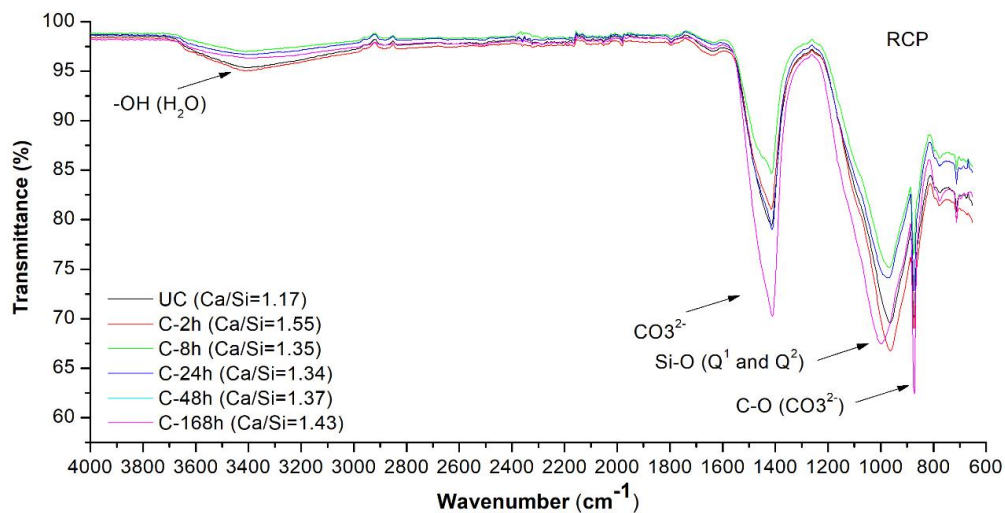
mineralization, RCP₂₀₀ (d₅₀ = 16.19 μm) and RCP_g (d₅₀ = 12.62 μm) powders presented a decreased in Ca/Si ratio on the first 2 hours, comparing it to uncarbonated samples, which is a evidence of C-S-H decalcification promoted by CO₂ [104,116]. On the RCP (d₅₀ = 20.99 μm) case, a drop in Ca/Si occurred only after 8 hours, which indicates that smaller particles are more susceptible to decalcification in shorter periods. Ca/Si ratios near 0.67 are associated with C-S-H decalcification and with silica gel conversion [101,104] (amorphous silica [117,118]), so, results indicated that there was no C-S-H polymerization and, for this reason, mineralized powders did not presented pozzolanic characteristics when applied as SCM [95,119]. These gels formation depends on as much the initial composition as the mineralization process conditions [120]. For example, materials with lower Ca/Si ratio tends to generate higher amounts of silica gel [121].

Figure 10- Recycled concrete powders atomic ratio by SEM-EDS after CO₂ exposure: a) Ca/Si ratio as a function of CDW origin; b) Ca/Si ratio as a function of particle size fraction



FTIR analysis results were used to detect possible structural alteration on C-S-H gel after mineralization (Figure 11). The RCP FTIR spectrums indicated a band between 3600 cm⁻¹ and 3300 cm⁻¹ that could be related to stretching vibration of O-H and N-H bonds, free or associated, leading to an overlapping possibility that difficult band attribution [122,123].

Figure 11- Mineralized RCP powders FTIR spectra at different times



The carbonated (CO_3^{2-}), associated to the calcite [124] reached peaks at 1600 cm^{-1} and 1200 cm^{-1} , with a vibration at 1416 cm^{-1} for uncarbonated samples (UC) and 1412 cm^{-1} for a 168 hours mineralized sample (C-168h). Previous studies showed that bands centered in 2914 cm^{-1} , 1400 cm^{-1} , and 870 cm^{-1} were related to stretching O–C–O and carbonates (CO_3^{2-}) bonds in cementitious materials [125–127]. The Si-O (ν_3) bond vibration peak of Q1 and Q2 C-S-H species that were at 964 cm^{-1} changed to higher waves after mineralization, between 968 and 1000 cm^{-1} . These alterations indicated Q1 and Q2 C-S-H species consumption by the carbonation process [128,129]. The absence of a Si-O (Q3 and Q4) related peak, around 1.070 to 1.080 cm^{-1} , indicated that amorph silica gel was not formed [130]. Q3 Species (decalcified C-S-H) are characterized by lower Ca/Si C-S-H (ideal ratio = 0.67) and latticed silicate tetrahedrons, while the Q4 species (polymerized silica) are defined like amorph silica with a silicate tetrahedrons highly condensed net [112,130,131].

Mineralization promoted significantly morphological changes on powders composition. On materials that came from concrete CDW it was verified a denser CaCO_3 agglomeration surrounding particles, in comparison to mixed source materials. Aragonite similar crystals presence on RCPg, associated with a Ca/Si decrease, indicated a particle size shortening that favored carbon fixation and C-S-H decalcification. Although the literature points silica gel pozzolanic activity to be one of the main factors that turn mineralized powders by accelerated carbonation fit to application as supplementary cementitious material [94,132], the FTIR spectrums did not indicated this gel formation. This way, by being applied as a partial cement replacement, these powders benefits are mainly physical, like a particle size distribution improvement and the nucleation sites decrease for hydration products [10].

3.3 RECYCLED POWDERS CO_2 FIXATION

Powders TG and DTG curves before and after accelerated carbonation mineralization are shown in Figure 12. For mixed powders, it is observed an initial mass loss between 100°C and 200°C (peak 1), indicating ettringite gel and C-S-H dehydration [23,27], followed by a mass loss associated to water released present on montmorillonite (peak 2) [133]. Between 300°C and 550°C (peaks 3 and 5), alkalis mass loss occurred, like magnesium and calcium hydroxides. The $\text{Mg}(\text{OH})_2$ came from mortars waste produced in Brazil with dolomitic limestone [24,26]. On concrete powders, the fourth peak ($400\text{--}480^\circ\text{C}$), related to $\text{Ca}(\text{OH})_2$ decomposition [66,134], was not observed after mineralization. This indicates that, in 2 hours, all portlandite was consumed. In comparison to uncarbonated samples, a highlighted mass loss is observed on DTG curves for carbonated samples in a temperature range from 500 to 800°C (peak 6), attributed to CaCO_3 decarbonation [135,136], which indicates powders CO_2 fixation. This mass loss is related to poorly crystalized polymorphs, the calcite, indicating that the thermodynamic factor was predominant and no other kinetic factors that favored aragonite and vaterite precipitation [112].

Figure 12- Mixed powders TG and DTG curves of uncarbonated (UC) and carbonated (C) powders after different mineralization periods: a) recycled mixed powders; b) recycled concrete powders

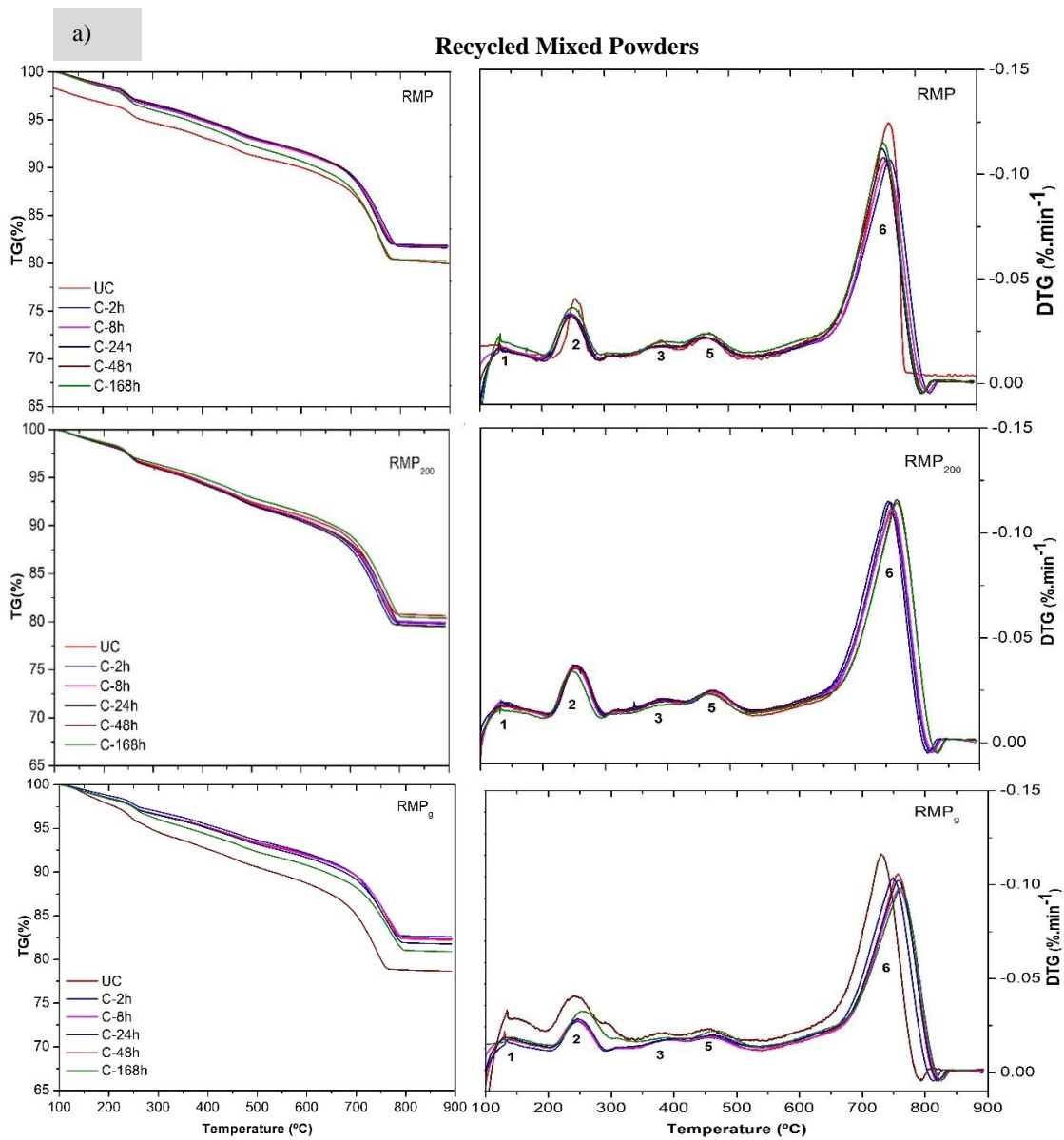
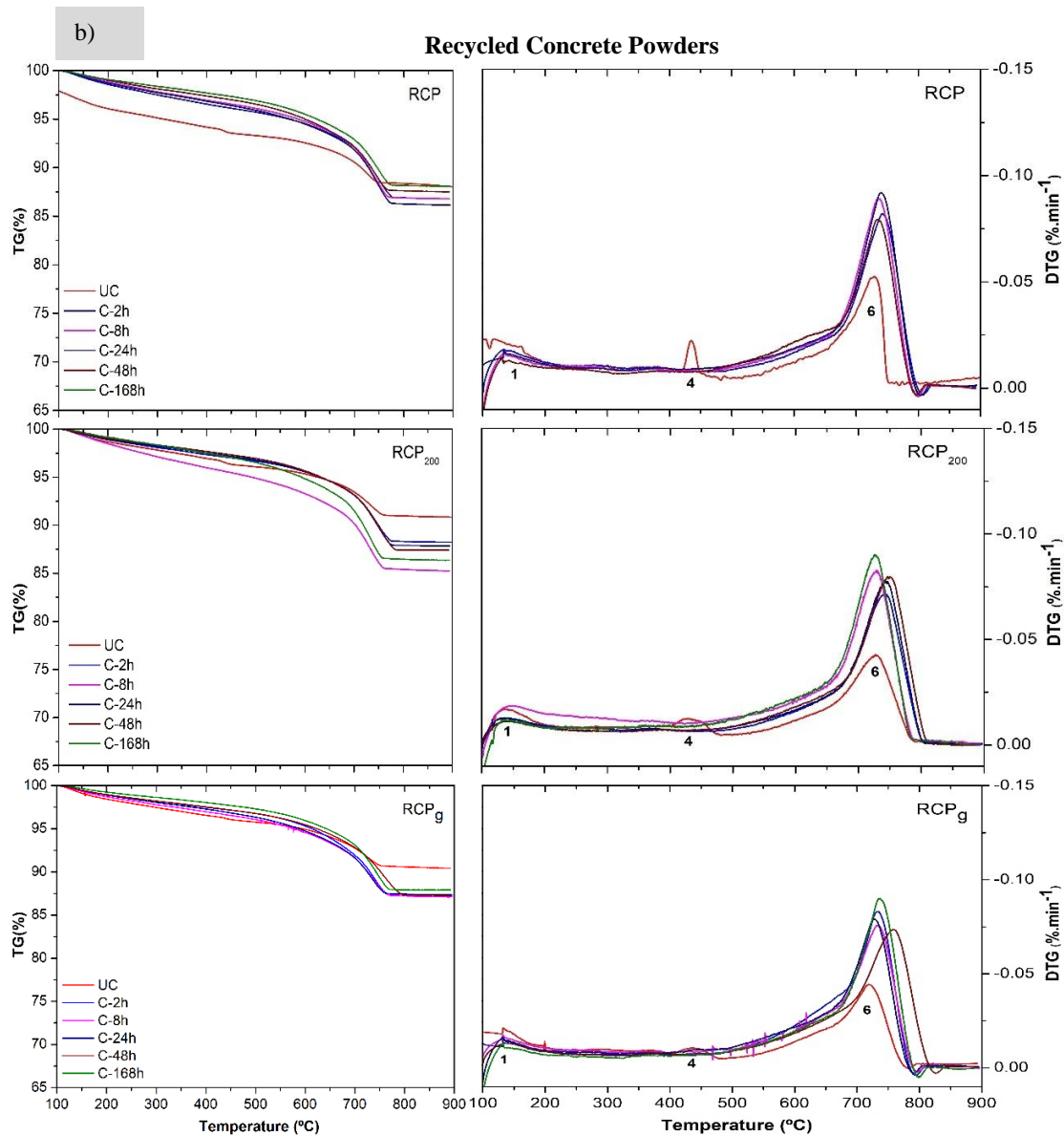
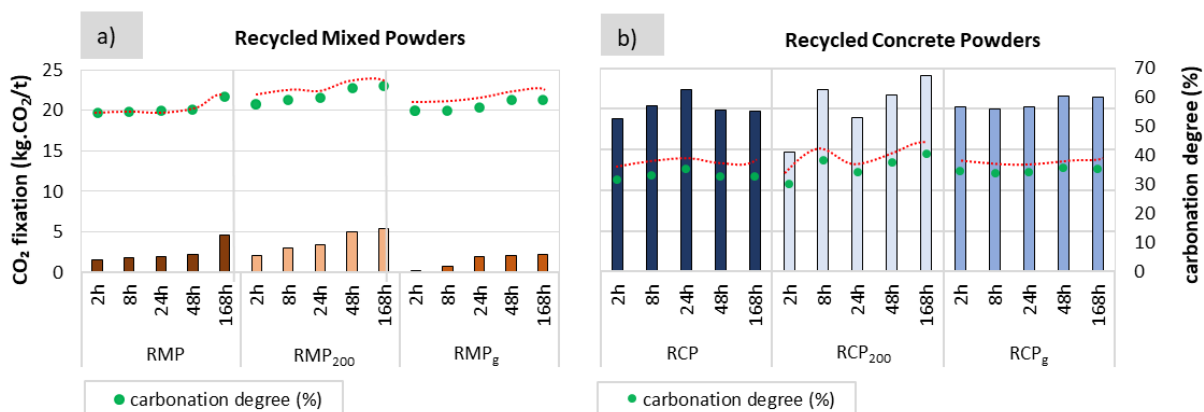


Figure 12- TG and DTG curves of uncarbonated (UC) and carbonated (C) powders after different mineralization periods: a) mixed CDW; b) concrete CDW



The CO₂ fixation and the carbonation degree on concrete and mixed CDW powders were quantified like presented in Figure 13. It is observed that there is a more elevated CO₂ fixation on concrete powders than on the mixed. For the mixed source (Figure 13a), it was verified values between 0.20 and 5.4 kg.CO₂/t, with a growth that accompanies the mineralization period. The concrete powders, in another way, presented superior data, between 14.7 and 24.1 kg.CO₂/t. This difference can be attributed to smaller available calcium amount for mixed powder, making it more difficult to carbonates formation [101], as verified on TG/DTG curves (Figure 12b).

Figure 13- Powders CO₂ fixation and Carbonation Degree for a) mixed CDW; b) concrete CDW



On literature, different carbon dioxide fixation potentials are reported to cementitious industries subproducts, the cement kiln dust (CKD, CaO > 45%) and cement bypass dust (CBD, CaO > 65%) presented data between 25 kg to 80 kg de CO₂/t [137,138]. For concrete slurry waste (CSW, Ø < 0.15 mm and CaO > 40%) the fixation capacity reaches 110 kg.CO₂/t of dry CSW [139]. The cementitious pastes recycled powders submitted to mechanic-chemical processing reached 221 kg.CO₂/t (CaO = 44.35%) [139]. Jiang *et al.* [94], in a gas-liquid process, obtained a 29.8% less absorption in the smaller fraction (0.6 mm) obtained from concrete milling (CaO = 27.8%) when compared to the obtained from hydrated cement paste fraction (CaO = 66.9%), reinforcing direct chemical composition influence on CO₂ fixation capacity. It is observed that different operational conditions were adopted on mineralization studies, including different raw materials, particle size distributions and calculation methods. These factors have difficult direct comparison between results, but, even so, are evidence of cementitious waste CO₂ fixation potential.

Under this study similar conditions (gas-solid, 70 ± 3% relative humidity, 20 ± 2 °C temperature and 20 ± 3% CO₂ concentration) Ouyang *et al.* [85], by evaluating recycled hydrated pastes powders with a 48.28% CaO content, verified that Ca(OH)₂ mainly reacted to form CaCO₃ on the first 6 hours. After 1 day, C-S-H gel started to gradually be transformed into silica gel, especially after a 7-day mineralization (168h), highlighting chemical composition and time role on mineralization efficiency. The authors reported a 48.3% CO₂ fixation after 7 days, which corresponded to a 483 kg.CO₂/t. This value is one of the more elevated from literature and very distinct from the ones found in studies with concrete powder. On this case, values are similar to the observed in fine recycled concrete aggregates (FRCA, Ø < 5 mm and CaO = 17.90%), with a potential around 20 kg.CO₂/t [32]. This limited performance could be attributed to raw materials composition, that had elevated SiO₂ content (>33%) and lower available CaO content (< 22%) to carbonates formation, results that reinforced challenges related to materials selection and/or processing still to be overcome CCUS technologies solidification [140].

By analyzing particle size distribution, powders obtained in the second process (RMP₂₀₀ and RCP₂₀₀), with the d₅₀ 12.52 µm and 16.19 µm presented, in comparison to the others, the higher CO₂ fixation all through the mineralization period. These results, indicate that particle size reduction is favorable for CO₂ fixation, however, fines agglomeration in finer powders like RMP_g (d₅₀ = 9.92 µm) and RCP_g (d₅₀ = 12.62 µm) could have limited the CO₂ diffusion and, consequently, reduced carbon fixation potential. Jiang *et al.* [94], observed that smaller fractions from fine aggregates and powders (2.36 mm, 0.60 mm, 0.30 mm, and 0.15 mm) fixed more CO₂ when compared to larger particles, corroborating this study findings.

Mixed powders carbonation degrees were superior to 50% while for concrete powders the data were between 30 and 40%, which points to remaining carbon fixation potential. On literature, many authors report significantly lower carbonation degrees, between 40 and 60%, for gas-solid mineralization while the

aqueous carbonation almost always reached full carbonation [120]. The insufficient free water amount difficult dissolution and reprecipitation in calcium phases, besides that, CaCO_3 can block the connected pores, gradually blocking CO_2 diffusion [40].

In synthesis, results show that concrete powder (with higher CaO content) presents higher CO_2 fixation compared to the mixed source, being the particle size and the mineralization period two important and influential variables. Considering powder agglomeration when the material is obtained in high efficiency mill (RMP_g e RCP_g), some others strategies can be applied like: aqueous carbonation, more elevated temperatures ($> 20^\circ\text{C}$) [66], water-solids ratio decrease (w/s) [54], and mechanic-chemical carbonation application [141]. All these alternatives can amplify CO_2 fixation in evaluated materials, including the smaller fraction, also reducing mineralization time.

3.4 POWDERS SCM PERFORMANCE

Mortars compressive strength results at 28 days (f_{c28}) with a 25% substitution content from cement to different particle sizes powders, before and after mineralization with different CO_2 exposure periods, are presented in Figure 14. Letters are applied (a, b, c, d, e) indicates different statistical significance between mean, according to the determined in the ANOVA followed by Scott-Knott test ($p < 0.05$). Statistical analysis can be verified on the supplementary material.

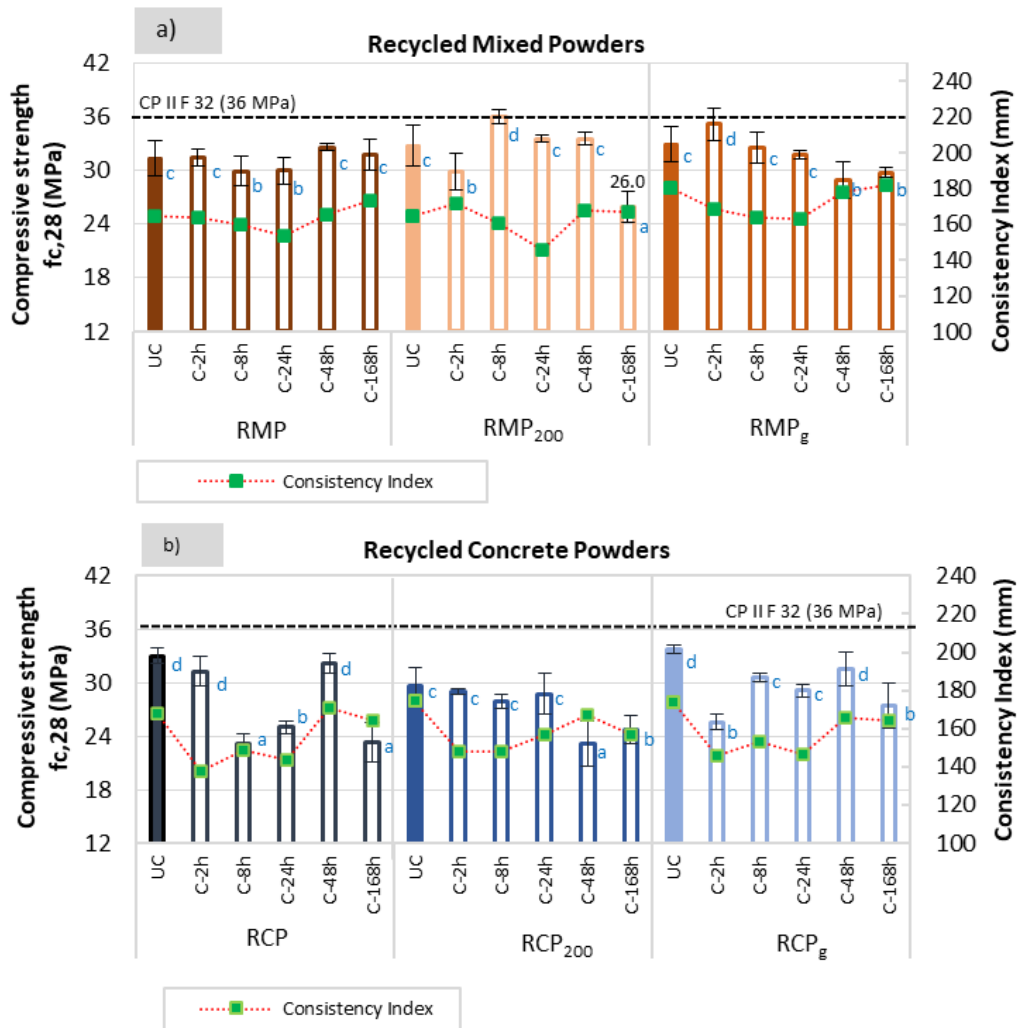
For all mixed powders (14a) the variable “particle size” did not significantly influenced mortars compressive strength (p -value < 0.05). Being the mortars with 25% RMP, RMP_{200} e RMP_g , uncarbonated (UC), statistically equals (31.43, 32.9, and 32.9 MPa) with all values near strength-class C32 and inferior to the presented by CP II F (36 MPa). In another side, for carbonated powders, the mineralization period did influence f_{c28} , decreasing compressive strength to 26 MPa in the case of RMP_{200} (C-168h).

For concrete powders (14b), the “particle size” and “mineralization period” variables statistically influenced mortars compressive strength, being considered equals the powders RCP-UC ($d_{50} = 16.19 \mu\text{m}$) and RCP_g -UC ($d_{50} = 12.62 \mu\text{m}$). For all particle sizes a decreasing tendency was verified for f_{c28} with 168 hour mineralized powders application. Even with a higher CO_2 fixation, the materials substitution indeed compromised powders performance due to the possible C-S-H decalcification [142]. Besides that, prolonged mineralization processes are not considered neither economic or efficient strategies [143].

With a 2-hour mineralization, it was verified that RCP and RCP_{200} powders presented compressive strength statistically equal to non-mineralized powders, even with the incorporated CO_2 . On the other hand, RCP_g (with a smaller particle size and higher carbon fixed) negatively affected f_{c28} , reducing it from 33.73 MPa to 25.59 MPa, which represents a 24% drop.

On literature, it is observed compressive strength improvements only when there is a higher CaO content with silica gel formation. Kaliyavaradhan, Li and Ling [27] reported an 8% increment in mortars f_c by replacing by 20% the cement by recycled concrete powders (CaO = 32.49%). More expressive results were reported by Lu *et al.* [83], with a 35% increase on pastes strength with 50% carbonated powder (CaO = 64.5%) replacement level after silica gel formation and full carbonation. In a similar way, Wu *et al.* [143] observed a mean gain of 12.6% with 30% carbonated paste powder use, attributed to $\text{Ca}(\text{OH})_2$ transformation in stable CaCO_3 and silica gel. It can be highlighted that studies conducted with powders from hydrated paste processing tend to present more satisfying mechanical and CO_2 fixation performance, since hydrated pastes do not have inert aggregates in its composition. Depending on concrete strength class and processing type, concrete powders could reach elevated inert materials content.

Figure 14- Mortars with 25% replacement compressive strength and consistency index for: a) mixed CDW; b) concrete CDW



Regarding fresh state behavior, a reduction is observed on mortars consistency index, effect that can be attributed to pore size reduction by superficial area increase, leading to a higher water demand [144,145]. This impact is even higher when there is the silica gel presence due to its elevated superficial area and water absorption capacity that intensifies consistency loss [146].

So, by analyzing powders performance, it is observed that recycled concrete powders (RCP e RCP₂₀₀), with 2-hour mineralization, even by presenting incorporated CO₂ in its structure, resulting in compressive strength statistically equal to uncarbonated samples, still had a consistency index decrease. Considering observed changes in particle size, density and particles morphology, water optimization or plasticizers use (considering a standard consistency index) could highlight powders use with fixed CO₂. So, prolonged mineralization periods did not demonstrate additional Properties gain, reinforcing reduced periods feasibility.

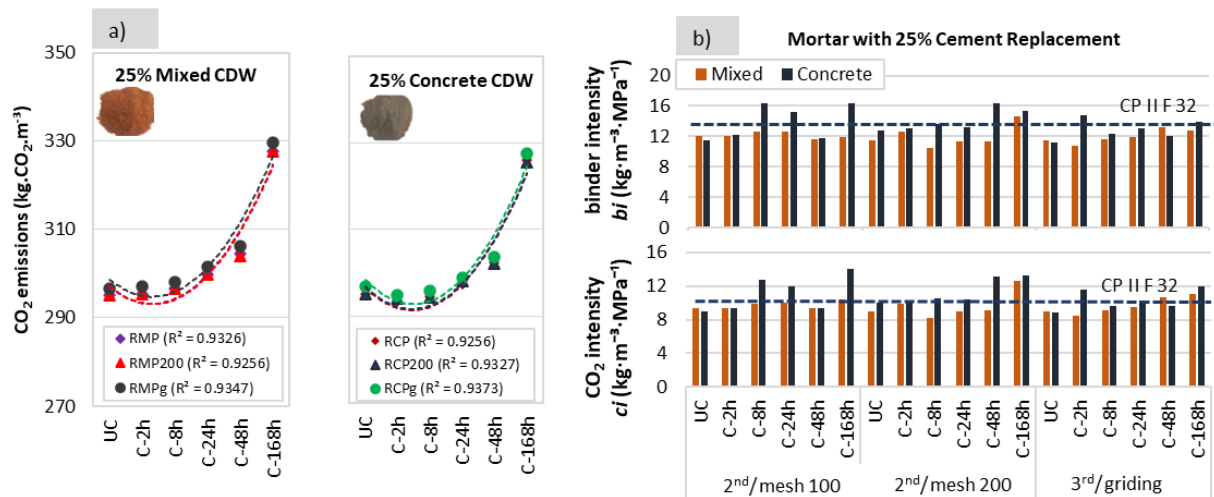
3.5 ENVIRONMENTAL PERFORMANCE

Considering cement and powders consumption for 1m^3 mortar production, total CO_2 emissions with 25% replacement level were significantly reduced, according to the presented-on Figure 15. Values for the mixed source material varied between 290 and 330 $\text{kg.CO}_2/\text{m}^3$, while concrete powder had between 294 and 328 $\text{kg.CO}_2/\text{m}^3$, both inferior to CP II F cement environmental impact that was 388 $\text{kg.CO}_2/\text{m}^3$. These results highlighted mineralized powder real potential use to emission mitigation on civil construction cement production.

By analyzing the mineralization effect, a reduction even higher was observed in 2-hour mineralized concrete powders (C-2h). For mixed one this decrease was kept almost constant throughout the evaluation period, indicating that mineralization is especially efficient on concrete-source CDW powders. Other processes like calcination could improve the mixed-source powders performance [24,25]. Besides that, a second order polynomial correlation was identified between the variables ‘mineralization period’ and ‘ CO_2 emissions’, that increases with the time passing. This occurs due to high impact from process electric energy consumption.

On European Union Emissions Trading System (EU ETS), a carbon credit in April 2024 had a mean prize of US\$ 61/t. $\text{CO}_2.\text{eq}$ [147]. Considering the lowest CO_2 emission registered (294 $\text{kg.CO}_2/\text{m}^3$) in comparison to CP II F, mineralization process resulted in an economy of about 24% CO_2 emissions, which presents in carbon credits a US\$ 5.70 economy per m^3 .

Figure 15- Powders environmental performance: (a) CO_2 emissions/ m^3 ; (b) binder intensity (BI) and CO_2 intensity (CI) indicators.



By analyzing the Binder intensity (BI) and CO_2 intensity (CI) indicator, presented in Figure 15b, it is observed that BI values are between 10.5 and 14.6 $\text{kg}/\text{m}^3.\text{MPa}$ for mixed-source and between 11.2 and 16.4 $\text{kg}/\text{m}^3.\text{MPa}$ for concrete-source powders. For the CI, values ranged between 9.0 to 11.2 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$ on concrete materials. With an exception for RCP_g , obtained from the third grinding process with a 2 hours mineralization, that resulted in mortars with both CI and BI inferior to the observed in reference Portland cement (CP II F 32 - 14.0 $\text{kg}/\text{m}^3.\text{MPa}$ and 11.2 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$, respectively).

Even with a significant particle size decrease promoted by grinding on RCP_g , morphological alterations due to mineralization summed with grinding related emissions compromised this material environmental efficiency. On this context, dispersant additives use could be an alternative to improve this waste efficiency

[90]. Similar results were obtained by Oliveira *et al.* [23], who investigated grinding time influence (0.5 h, 2 h and 6 h) on materials environmental performance when applied in a 25% cement replacement level. The authors indicated values of 13.77, 14.07, and 15.77 kg/m³.MPa for BI and 10.01, 10.39, and 12.10 kg.CO₂/m³.MPa for CI. These data confirm that an increase in processing time is directly related to higher energy consumption and, consequently, a CO₂ emissions increase that compromises powders environmental performance.

Considering the emissions avoided by a 25% replacement, the environmental benefits observed were significant. This strategy not only contributed to natural resources conservation and associated emissions reduction, but also demonstrated recycled powders CO₂ fixation potential, which turns them into eligible materials for regulatory markets like carbon credits. With an exception for RCP_g, the 2-hour mineralization showed promising efficiency for indexes reduction like BI and CI. However, to make this technique industrially feasible, a shorter period is required and must be evaluated.

4 CONCLUSIONS

Based on developed experimental planning to evaluate carbon fixation as much as compressive strength and CO₂ emissions in a Portland cement mortar with a 25% replacement level for Construction and Demolition Waste (CDW) powders from a mixed-source (RMP) and from a concrete-source (RCP), with different particle sized, a few conclusions can be drawn:

- Powders CO₂ mineralization promoted alterations on particles physical-mechanical properties, changing mean diameter, reducing pH (>8) and promoting an increase in density and surface area due to calcium carbonate formation.
- Mixed powders carbon fixation was 0.2 and 5.4 kg.CO₂/t with a carbonation degree superior to 50%, while concrete powders were 14.7 and 24.1 kg.CO₂/t, with carbonation degree varying between 30% and 40%, indicating residual carbonation potential.
- The water amount applied for pre-hydration, combined with the CaO content (<50%), did not favor C-S-H polymerization and silica gel formation, so mineralization powders act by filler effect when applied as supplementary cementitious material.
- RCP milling in planetary mill favored CO₂ fixation (values between 20 and 21.5 kg.CO₂/t), but particles agglomeration limited gas diffusion and the higher energetic consumption negatively impacted this process environmental performance.
- RCP and RCP₂₀₀ powders, obtained from the second process and mineralized for 2 hours, presented compressive strength at 28 days statistically equivalent to uncarbonated powders (UC), with values of 31.28 MPa and 28.95 MPa respectively. These powders use as a 25% replacement promoted a 24% CO₂ emissions reduction per m³, when compared to the reference Portland cement (CP II F- 32).

For practical mineralized powder application (when carbonated by gas-solid) period inferior to 2 hours must be studied just like the temperature and water-solids ratio influence. Dynamic reactors application or mechano-chemical carbonation could eliminate fine powder agglomeration problem, improving CO₂ fixation capacity and amplifying feasibility for application in carbon market regulatory systems. This could also contribute to civil construction circular economy principles. Different technologies roads must be considered based on life cycle assessment to identify more sustainable and efficient strategies for large-scale adoption.

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SUPPLEMENTARY MATERIAL

The parameters of the Scott-Knotts test are shown in Table S1, where p-values less than 0.05 ($p > 0.05$) mean that there is no significant difference between the means observed.

Table S1- Parameters of the Scott-Knotts for mortars compressive strength

Origin	Variable	SS	DF	MS	F	p-value
mixed powders	Particle size (RMP, RMP ₂₀₀ , RMP _g)	6.568226	2	3.284113	1.425	0.2539
	Duration of mineralization (0h, 2h, 8h, 24h, 48h and 168h)	76.341281	5	15.268256	6.623	0.0002
	Particle size x Duration of mineralization	206.426285	10	20.642629	8.954	0.0000
	error	82.992400	36	2.305344		
Origin	Variable	SS	DF	MS	F	p-value
concrete powders	Particle size (RCP, RCP ₂₀₀ , RCP _g)	58.231559	2	29.115780	12.166	0.0001
	Duration of mineralization (0h, 2h, 8h, 24h, 48h and 168h)	240.565481	5	48.107096	20.101	0.0000
	Particle size x Duration of mineralization	314.720641	10	31.472064	13.150	0.0000
	error	86.15840	36	2.393289		

SS: Sum of Squares; DF: Degrees of Freedom; MS: Mean Square.

3.4 ARTIGO 4 (OTIMIZAÇÃO)

A partir dos resultados obtidos nos artigos 1 a 3, e considerando que os resíduos de concreto já foram previamente expostos ao ambiente durante sua vida útil, este estudo buscou ajustar os principais parâmetros em câmara de carbonatação acelerada para otimizar as condições de fixação de CO₂ em pós reciclados de concreto (RCP). Assim, no artigo 4, intitulado *Otimização da mineralização de CO₂ por carbonatação acelerada de pós de concreto reciclado para a produção de cimento com baixa emissão* avaliou-se o efeito da umidade da amostra (0%, 6%, 12%, 18% e 24%) e da temperatura de ensaio (20, 40 e 60 °C) no processo de mineralização de CO₂ em RCP, em um sistema estático gás-sólido (15% de CO₂, umidade relativa de 60 ± 10%), ao longo de diferentes tempos de exposição.

Além dos ensaios de caracterização como densidade, pH, área superficial (BET) e morfologia (MEV), realizou-se análises de DRX, FTIR e TG/DTG, antes e após o processo de mineralização de CO₂. O uso dos pós como MCS foi testado em argamassas com substituição de 25% do cimento, por meio da avaliação da resistência à compressão aos 7, 28 e 56 dias.

Verificou-se que existe uma faixa ótima de umidade da amostra, entre 6% e 18%, que favorece a fixação de CO₂, e que o aumento da temperatura de tratamento para 60 °C contribui significativamente para o incremento da fixação de CO₂ no mesmo tempo de ensaio (30 minutos), alcançando valores entre 10.15 e 10.95 kg CO₂/t.

Aos 28 dias, utilizando RCP com 12% de umidade, expostos ao CO₂ por 0,5 h à 40 °C e 60° C, foram obtidas argamassas com resistência à compressão compatível com a classe C32 (32 MPa) e fixação de 7.24 kg.CO₂/t e 10.95 kg.CO₂/t de RCP respectivamente. Observou-se que a quantidade de CO₂ fixada no RCP não é diretamente proporcional ao ganho de resistência nas argamassas, indicando que a otimização do processo deve considerar a finalidade do uso do RCP: maximizar a reatividade ou a fixação de CO₂.

Por fim, considerando que utilizando o protocolo com 0,5 h de exposição ao CO₂ em câmara de carbonatação acelerada, temperatura de 60 °C e umidade do RCP de 12%, obteve-se uma fixação de CO₂ de 10,95 kg CO₂/t de RCP. E com a substituição de 25% da massa de cimento CPI por RCP carbonatado, as resistências à compressão das argamassas, determinadas conforme a NBR 7215 (ABNT, 2019), foram de 32,05 MPa e 38,88 MPa aos 28 e 56 dias, respectivamente, no Artigo 5, esse protocolo foi padronizado, e o estudo foi então ampliado para incluir RCPs de diferentes origens e outros teores de substituição.

Os resultados desse artigo evidenciam que a associação entre tecnologias de CCUS e o uso dos RCP como MCS pode viabilizar a produção de cimentos com menor pegada de carbono, contribuindo para a descarbonização da indústria da construção. O artigo, submetido ao periódico indexado na base Scopus, é apresentado a seguir em sua íntegra, podendo apresentar modificações em função das revisões editoriais.

OPTIMIZING CO₂ MINERALIZATION BY ACCELERATED CARBONATION OF RECYCLED CONCRETE POWDERS FOR LOW-EMISSION CEMENT PRODUCTION

Highlights

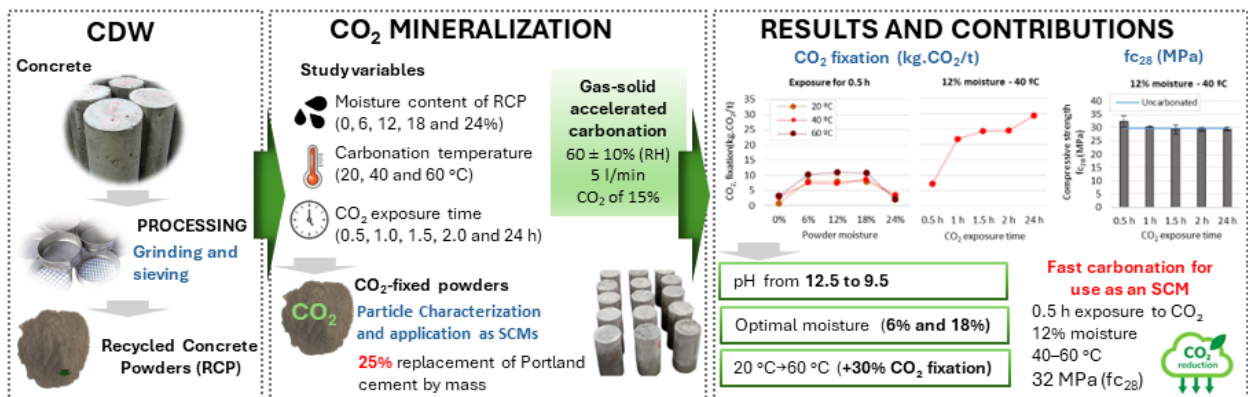
- Recycled Concrete Powder (RCP) with 5 humidity levels were submitted to CO₂ action (15%) with controlled temperatures at 20, 40 and 60 °C.
- The optimized CO₂ fixation happened between 6% and 18% sample humidity.
- A temperature addition from 20 °C to 60 °C improved in 30% CO₂ fixation.
- An increased CO₂ exposure period from 0.5 h to 24 h, increased CO₂ fixation from 7.24 to 29.47 kg.CO₂/t.
- CO₂ mineralization process increased in 3% powders bulk density, reduced the pH from 12.5 to 9 e and changed the Ca/Si ratio.
- The RCP reactivity is strongly conditioned to materials physico-chemical characteristics and mineralization conditions applied.

Abstract

The carbon dioxide (CO₂) fixation uses on concrete aggregates and recycled powders, using technologies of carbon capture, use and storage (CCUS), represents a promising alternative for emissions reduction and a hard-decarbonized sector like the cement industry. This study evaluates sample humidity (0%, 6%, 12%, 18% e 24%), test temperature (20, 40 e 60 °C) and exposure period effect (0.5, 1, 1.5, 2 e 24 h) on CO₂ mineralization applied in recycled concrete powders (RCP). For this, a static gas-solid system was applied with 15% CO₂ and relative humidity between 60 ± 10%. Several properties were evaluated like density, pH, surface area (BET), morphology, and DRX, FTIR and TG/DTG analysis before and after CO₂ mineralization on RCP. The application of RCP as supplementary cementitious material (SCM) after mineralization were tested in mortars with a 25% mass Portland cement replacement by a compressive strength evaluation at 7, 28 and 56 days. It was verified that a exists a great humidity for the carbonated samples between 6 and 18% that favors CO₂ fixation. For the same exposure period to CO₂ (30 minutes), the treatment increased temperature from 20 to 60 °C significantly contributed to an increment on carbon fixation, reaching values between 10.15 e 10.95 kg.CO₂/t. The powders application as SCM (RCP with 12% humidity, exposed to CO₂ for 30 minutes at 40 °C), obtained a compressive strength like the reference material. It was verified that the increased CO₂ capture did not directly promote a higher mechanical strength, highlighting the materials characteristics can be optimized independently since, in these conditions, the silica gel formation did not happen through the calcium silicate hydrate polymerization (C-S-H). The results evidenced that the association between CCUS technologies and the use of RCP as SCM can promote a lower carbon footprint cement production, contributing to construction industry decarbonization.

Keywords: Fast carbonation; Carbon utilization; CO₂ emissions; Circular economy; CO₂ uptake.

Graphical abstract



1 INTRODUCTION

Climate change represents one of the greatest challenges of our time, imposing global coordinated mobilization to mitigate its impacts. The answers to the 2015 Paris Agreement [1] and the established directives of the conference of the parts (COP), signed by 195 countries including the European Union, are compromised to search for strategies to limit global warming to 1.5°C until 2030 [2]. However, the global carbon dioxide (CO₂) emissions reached 37.41 billion gigatons (GtCO₂) in 2024 [3] which indicates that the already implemented measures are not enough to contain the increase on global temperature [4].

A key sector to achieve these goals is civil construction, responsible for approximately 37% of CO₂ emissions [5]. Within this sector, the cement industry are highlighted as the main source of emissions, contributing to a significant amount of the global total, with values in 2023 of approximately 2.4 billion tons of equivalent CO₂ (Gt.CO₂.eq) [6]. Currently, the biggest challenge is decreasing emissions while maintaining cement production that are produced due to the increasing demand for infrastructure, promoted by increased population [7–9]. This cement demand are estimated to reach 3 billion tons per year by 2050 [10].

To face this issue, a roadmap were elaborated with strategies to guarantee the cement and concrete supply chain decarbonization until 2050 [11,12], and within these strategies is the application of supplementary cementitious materials (SCM) to reduce clinker content on cement and the implementation of carbon capture, utilization, and storage technologies (CCUS) production plants, with a CO₂ decrease potential between 9 and 36% respectively [13].

The CO₂ mineralization by accelerated carbonation or mineral carbonation (MC) is a CCUS technology pointed by the literature as promising to CO₂ emissions removal [14–16], which imitates the natural process of rock weathering where the CO₂ reacts with calcium or magnesium-composed minerals to become permanently stored as stable carbonates (CaCO₃/MgCO₃). This way, the storage is considered safe and there is almost null necessity of monitoring [17,18].

Several waste materials have been investigated as possible candidates for carbonation material, between them, the recycled aggregates from construction and demolition waste (CDW), particularly concrete source CDW [19–21]. This solution, besides definitive fixing CO₂, improves particles characteristics by the increased density and lower water absorption promoted by CaCO₃ formation [22,23], enhancing concrete recycled aggregates use and favoring circular economy [24,25]. The recycled materials mineralization process could fix between 4.9 to 20 kg.CO₂/ton [26–29], being considered an asset on carbon market.

Over the last five years, the search for strategies to improve recycled powder utilization are growing, specially recycled concrete powder (RCP) as SCM on Portland cement [30–32]. The RCP, with particles smaller than 0.15mm, is generated during the CDW recycling process [33] and presents in its composition hydration products like calcium silicate hydrate (C-S-H), Ca(OH)₂, AFm (ettringite) and sand [30,34,35] which results in low reactivity [36,37]. Promising results found in the literature indicated that, with CO₂ use in mineral carbonation on powders obtained from hydrated cement pastes, promoted CO₂ fixation between 115 to 270 kg.CO₂/ ton and improved material performance when used as SCM, resulting in CaCO₃ and silica gel formation [38–40].

The mineral carbonation can be conducted by different procedures like gas-solid, liquid-solid, mechanochemical and others [41,42], which makes difficult a direct comparison between results obtained on literature. Several operational parameters like temperature, reaction time, CO₂ concentration, liquid/solid ratio, solution pH and rotation energy [14,19,43] influence fixation efficiency and also affect its performance when applied as SCM. The non-optimization of all these parameters leads to inefficiency on particles application as SCM. So, it is essential to search for a balance which will maximize the particles' performance, improve carbon fixation and promote a less emissive cement production. With this, it is

possible to capture carbon by the mineralization process and apply the treated waste as a product to promote circular economy and decarbonization.

Due to inert particles presence, inherent to concrete production, the CaO content on RCP is generally lower than 40% [33,44]. By being heterogeneous [33,45] and being previously carbonated on natural environment occurred during the life cycle, the CO₂ mineralization process is challenging due to the amount of variables involved. Facing this scenario, and considering that the RCP use favors complete concrete recycling, aligned with circular economy principles [43,46], this papers investigated different humidity and temperature combinations on accelerated RCP carbonation, aiming to optimize CO₂ mineralization and evaluate this material potential to produce less emissive cements.

2 MATERIALS AND METHODS

2.1 MATERIALS

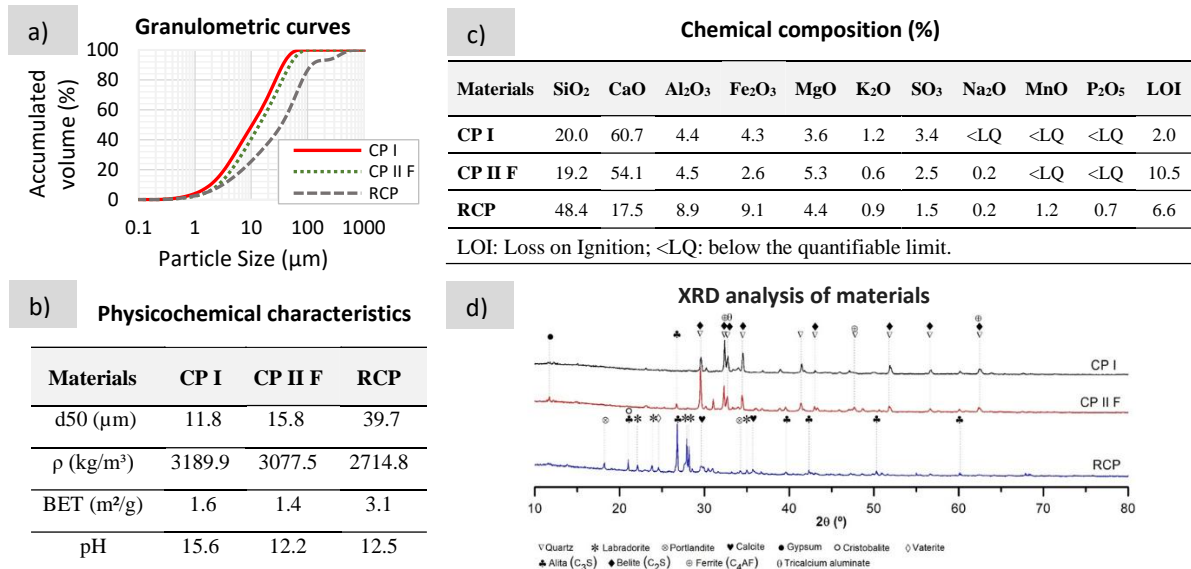
On this study were applied: RCP (Recycled Concrete Powder), Cement type CPI (Common Brazilian Portland Cement), containing 3.5% calcium sulfate (CaSO₄) [47], like ASTM Type I or cement CEM I [48], the CP II F (Filler Portland Cement, with up to 25% carbonate material [47]) equivalent to ASTM limestone cement (IL) [49] river sourced sand. The sand had density of 2.65 kg/dm³, and particle size distribution defined by standard, being employed proportion of 25% for each fraction 1.18 mm, 0.60 mm, 0.30 mm, and 0.150 mm. The RCP, with particle size passing Mesh 100 (Ø <0.15 mm), were obtained by concrete old specimens processing with grinding and sieving.

Raw materials characterization is presented on Figure 1. The particle size and mean diameter (d₅₀) [50] were made in a laser granulometer type Mastersizer 3000 (Malven Instruments) equipped with Aero S for dry powder dispersion. The instrument analyzes particles between 0.01 µm and 3500 µm, operating the dispersion by 2 bar air pressure at a 35% rate, each analysis was made in triplicate and with absorption index of 0.1. The density (ρ) was analyzed in an helium (He) equipment, model Ultrapyc 5000, from Anton Paar, with 10 psi operational pressure [51]. The surface area - BET was determined by nitrogen adsorption on a Micromeritics TriStar II Plus analyzer Version 3.0, using vacuum to degas at 160°C for 8 horas and nitrogen [52]. The hydrogen potential (pH) was determined using a digital pHmeter. For this, 5g samples were diluted in 50 ml of deionize water, which reading were made after equipment stabilization (approximately after 5 minutes) [53].

The semi quantitative Chemical composition was determined by X-Ray Fluorescence (XRF) in a spectrometer WDS Bruker S8 Tiger equipped with Rh tube. The mineralogical composition was determined by X-ray diffraction (XRD) on power mode, in diffractometer (Panalytical) with configuration θ-γ Bragg-Brentano, radiation Cu Kα and λ = 1,5418 Å, operating on 20 mA, 40 kV. The peaks were analyzed on a 5° to 100° angular range and in 26 minutes' time. The diffractogram analysis was made of software X'Pert High Score Plus V4.8 from PANalytical [54] and the data were interpreted with data pattern by Crystallography Open Database.

Considering the processing use, it is observed that RCP was a d₅₀ superior to CPI and CP II F cements (Figures 1a and 1b). The CaO content on RCP (Figure 1c) is inferior to the powders obtained from pastes and used in similar researches (>60%) and superior for SiO₂ (<20%) [38,41,55]. Kaliyavaradhan, Li and Ling [31], Feng *et al.* [56] and Qian *et al.* [57] evaluated concrete pavement powder, recycling plants concrete and mortars powders respectively, registering a mean value for CaO content of 30.35% and 53.22% for SiO₂. Agreeing with these results, by XRD (Figure 1d) it was observed that RCP is mainly composed of quartz (SiO₂) from the natural aggregates, labradorite from basaltic rocks available on the region and also used as artificial sand, polymorphs calcite and vaterite from carbonated and portlandites (Ca(OH)₂), coming from residual non-hydrated cements from the source concrete CDW [58–60].

Figure 1-Materials Characterization by: a) Granulometric curves; Physicochemical characteristics; c) Chemical composition; d) XRD analysis



2.2 POWDERS MINERALIZATION

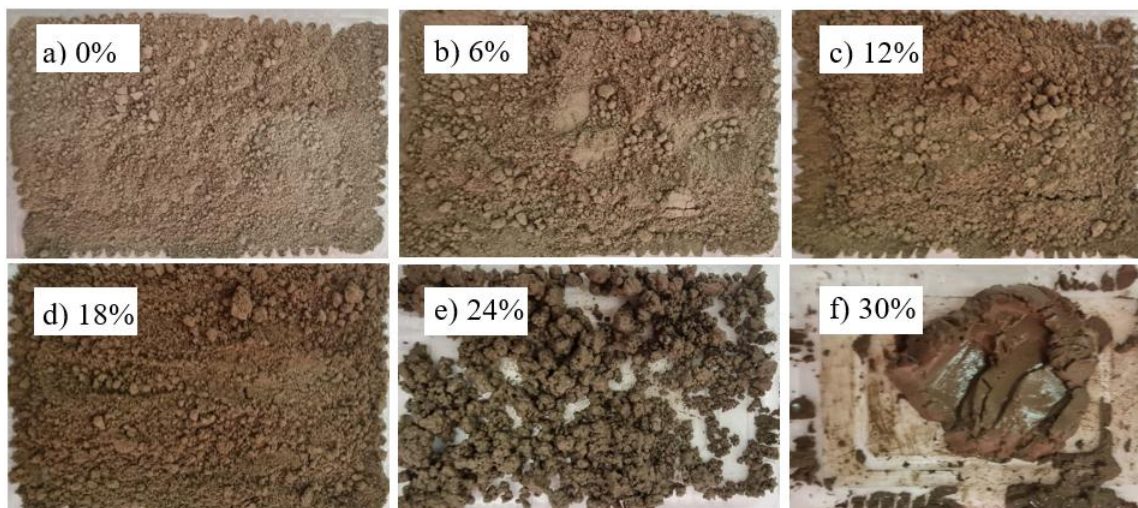
The CO₂ mineralization on recycled concrete powders was conducted by a gas-solid static process, in an Accelerated carbonation chamber BASS, mode, UUC-RH-STD-CO₂-1000/2016, considering fixed relative humidity at 60 ±10%, and CO₂ flow of 5 l/min, at 15% with exposure period of 30 minutes. In accordance with experimental design (Table 1), the testing temperature were controlled at 20, 40 and 60°C with material humidity at 0, 6, 12, 18, and 24%. For central point the CO₂ exposure time was also varied in 0.5, 1, 1.5, 2, and 24 h, totalizing 19 carbonation-mineralized powder (C), which were compared to uncarbonated RCP (UC).

Table 1-Experiment design for CO₂ mineralization in RPC

Moisture content (%)	Temperature and carbonation time in CO ₂ Chamber (in hours)		
	20 °C	40 °C	60 °C
0	(0.5)	(0.50)	(0.5)
6	(0.5)	(0.50)	(0.5)
12	(0.5)	(0.5, 1, 1.5, 2, 24)	(0.5)
18	(0.5)	(0.5)	(0.5)
24	(0.5)	(0.5)	(0.5)

To reach the humidities of 0%, 6%, 12%, 18% and 24% by sample mass, initially 250 grams of RCP previously dried at 35 °C, was put in a tray, being manually humidified by water aspersion and moved for homogenization. The mixing and aspersion process was standardized in 20 and 30 minutos. It is observed that the physical aspect of powders humidified with 24% humidity (Figura 2) there is the formation of hydrated agglomerates (aspect of farofa) and, with 30%, a paste formation which leads to the conclusion that saturation occurred.

Figure 2-Appearance of powders depending on the amount of water used in sample preparation:
a) 0%; b) 6%, c) 12%, d) 18%, e) 24% and f) 30%



After water aspersion for the conditions presented on table 1, the powders were put individually on carbonation chamber (to avoid humidity changes). After the mineralization process, powders were put in a furnace at 40 ± 5 °C temperature to avoid ettringite dehydration [61–63] until balance humidity condition achievement. Posteriorly the samples were stored to further characterization and use as SCM (by cement replacement).

2.2.1 Powders characterization

Besides the characterization by density, pH and surface area – BET performed according the described on item 2.1, the pore volume was obtained through isothermal adsorption $P/P_0 = 0.95$, assuming complete pore saturation [52,64].

The morphological characterization was made by Scanning Electron Microscopy (SEM), applying Zeiss – EVO MA10 equipment, with image detection by secondary electrons and X-Ray Energy Dispersion Spectroscopy (EDS). The powders were deposited in carbon tape, with posterior gold metallization using Sputter Coater Quorum SC7620 (15 mA for 1 minute and 30 seconds). A tungsten filament was used with potential difference of 10 kV [58].

For silicon-related phases identification, were registered spectrums by infrared spectroscopy with Fourier transform (FTIR), in rage from 4000 cm^{-1} to 600 cm^{-1} , in a PerkinElmer equipment model Frontier, in attenuated total reflected mode (ATR), by pellets method with KBr. The scanning was made with spectral resolution of 4 cm^{-1} .

2.2.2 Thermogravimetric analysis (TGA)

For CO_2 powders fixation estimative (Table 1), Thermogravimetry Analysis (TGA) were applied, based on the comparison between peaks corresponding to calcium carbonate (CaCO_3) decarbonation, that occurs in the temperature between 550 and 800 °C (on calcite form), from carbonated and non-carbonated samples (Equation 1 and 2) [65,66]. For this, after powders accelerated carbonation mineralization, the samples were put in isopropyl alcohol (isopropanol) to guarantee hydration interruption [62,67]. After 7 days, the excess of alcohol was removed, and powders were kept in vacuum to avoid natural carbonation by atmospheric air contact. The TGA was performed in a simultaneous thermal analyzer (STA) Perkin Elmer STA 6000. Samples were kept at 35°C for 1 hour and, than, a nitrogen flow was applied in a rate of $30 \text{ ml} \cdot \text{min}^{-1}$, with

a heating range between 30°C and 1000 °C and heating rate of 10°C.min⁻¹, according to the literature [24,58,68].

$$CO_2(\%) = \Delta m_{(550-800\text{ }^\circ\text{C})} \times \frac{MM_{CO_2}}{MM_{CaCO_3}} \quad \text{Equation 1}$$

$$CO_{2,fixation}(\%) = CO_2(\text{carbonated}) - CO_2(\text{uncarbonated}) \quad \text{Equation 2}$$

Where: MM_{CO_2} = CO₂ molar mass (~44.01 g/mol), MM_{CaCO_3} = CaCO₃ molar mass (~100.09 g/mol). The thermal decomposition beginning can change according to the CaCO₃ cristallinity [69,70], so the data was individually extracted from the DTG/TG curves. The carbonation degree (%) was calculated according to Equation 3 [70,71].

$$\text{Carbonation degree } (\%) = \frac{CO_{2,fixation}}{\text{Max.capacity of } CO_{2,fixation}} \times 100 \quad \text{Equation 3}$$

Each material *Max. capacity of CO_{2,fixation}* depends on the original CDW Chemical composition, mainly due to CaO differences. This way, with the $CO_{2,fixation}$ values obtained by DTG/TG and the CaO obtained by XRF, the *Max. capacity of CO_{2,fixation}* was calculated according to Equations 4, 5 and 6.

$$\text{Max. capacity of } CO_{2,fixation} (\%) = CaO_{\text{free}} * \left(\frac{MM_{CO_2}}{MM_{CaO}} \right) \quad \text{Equation 4}$$

$$CaO_{\text{free}}(\%) = CaO_{\text{XRF}} - CaO_{\text{eq.CaCO}_3} (\%) \quad \text{Equation 5}$$

$$CaO_{\text{eq.CaCO}_3} (\%) = CO_{2,captured} (\%) * \left(\frac{MM_{CaO}}{MM_{CO_2}} \right) \quad \text{Equation 6}$$

Where: CaO_{XRF} = proportional presence of CaO obtained by FRX.

2.3 Mineralized powders use

The mineralized and raw powders were used in mortars production, prepared according to NBR 7215 [72], similar to ASTM C1437-20 [73]. For a proportion, in mass, of 1: 3: 0.48 (cement: quartz sand: water/cement), 25% of cement Portland (CP I) mass was replaced by recycled concrete powders. The sand presented density of 2.65 kg/dm³ and particle size distribution defined by standard, being applied 25% for each fraction: 1.18 mm, 0.60 mm, 0.30 mm, and 0.150 mm. Compressive strength was determined at 7, 28, and 56 days in hydraulic press (model I-3025-B), with loading rate of 0.25 ± 0.05 MPa/s, applying the mean value between 4 cylindrical specimens (50 mm × 100 mm) cured in humid chamber (temperature at 23 ± 2 °C and relative humidity higher than 50 %). The results were analyzed in Sisvar Software [74] and submitted to Analysis of Variance (ANOVA) with means comparison (Tukey test) and significance level of 95% (p ≤ 0.05).

To evaluate the environmental performance, the Carbon Index (CI) was applied, in kg.CO₂.m³/MPa, that indicates how much carbon dioxide (in kg) were emitted to produce 1m³ at 1 MPa, according to Equation 7. Carbon emissions were calculated based on Portland cement consumption and the RCP necessary to produce 1 m³ of mortar, expressed in kg.CO₂/m³, as demonstrated on Equation 8. For powder-associated emissions, Equation 9 was used.

$$CI \left(kg \cdot \frac{CO_2}{m^3} / MPa \right) = \frac{CO_2 \text{ emissions } \left(\frac{kg \cdot CO_2}{m^3} \right)}{\text{Compressive strenght } (MPa)} \quad \text{Equation 7}$$

$$CO_2 \text{ emissions} = (C_{CP I} * E_{CP I}) + (C_{RCP} * E_{RCP,processing}) \quad \text{Equation 8}$$

$$E_{RCP,processing} = E_{RCP,obtaining} + E_{carbonation \text{ chamber}} - CO_{2,fixation} \quad \text{Equation 9}$$

Where: $C_{CP I}$ = CP I consumption for 1 m³ mortar production (kg/m³); $E_{CP I}$ = Emissions associated to CP I production (kg.CO₂/t); C_{RCP} = RCP consumption for a 1 m³ mortar production (kg/m³); $E_{RCP,processing}$ = Emissions associated to RCP processing (kg.CO₂/t); $E_{obtaining}$ = Emissions associated to RCP obtention (kg.CO₂/t); $E_{carbonation \text{ chamber}}$ = Emissions associated to electric energy consumption by the carbonation chamber and $CO_{2,fixation}$ = Amount of CO₂ fixed during mineralization, that was already calculated by Equation 2 (kg.CO₂/t).

For the CO₂ emissions calculus for carbonation chamber consumption, an emissions mean factor was applied ~46 kg.CO₂/MWh (0.046 kg.CO₂/ kWh) informed by the Brazilian Ministry of Science, Technology and Innovation [75]. A equipment potential was considered at 5 kW [76] and a production capacity of 150 kg of treated powder per exposure time. The CO₂ emissions data are indicated on Table 2.

Table 2-Values for carbon emissions

Data	Carbon emissions	Reference
$E_{CP I}$	890.29 kg.CO ₂ /t (Mean value)	[77]
$E_{CP II F}$	762.70 kg.CO ₂ /t (Mean value)	
$E_{RCP,obtaining}$	6.36 kg.CO ₂ /t	[78]
$E_{carbonation \text{ chamber}}$	1.53 kg.CO ₂ /t * duration of mineralization	[79]

3 RESULTS AND DISCUSSION

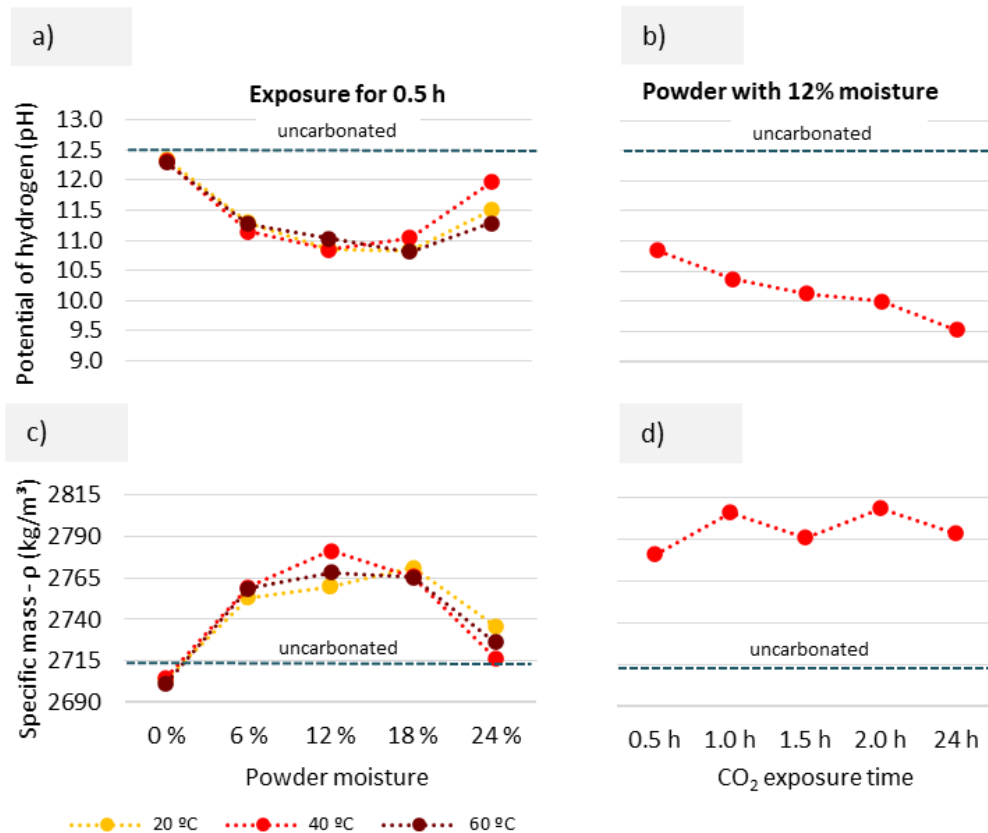
3.1 MINERALIZED POWDER CHARACTERISTICS

3.1.1 pH and Specific mass

The RCP physico-chemical Properties were altered after CO₂ mineralization, noting the content and humidity influence (Figure 3). The RCP pH for uncarbonated (12.5) decreased significantly in function of the humidity rate for all temperatures (Figure 3a) and in function of the time (Figure 3b). For the dried samples, 0% humidity, there was practically no pH alteration, which can be justified by the lower water rate on pores, not allowing the gas dissolution and posterior CO₂ particles fixation [80].

For 12 and 18% humidity, there was a pH reduction for values closer to 11, indicating CaCO₃ formation [19,31,81], result that is in accordance with literature data, that affirm that water content is a key parameter to promote CO₂ mineralization on powders, leading to CO₂ fixation [82,83]. For 24% rate the amount of water applied on RCP could have resulted on pore saturation [84,85], harming diffusion and CO₂ mineralization. This effect is evidenced by a more elevated pH compared to results obtained in other materials. All powders presented pH superior to 9, that indicates that not all hydroxides were fully carbonated (pH<8) [86].

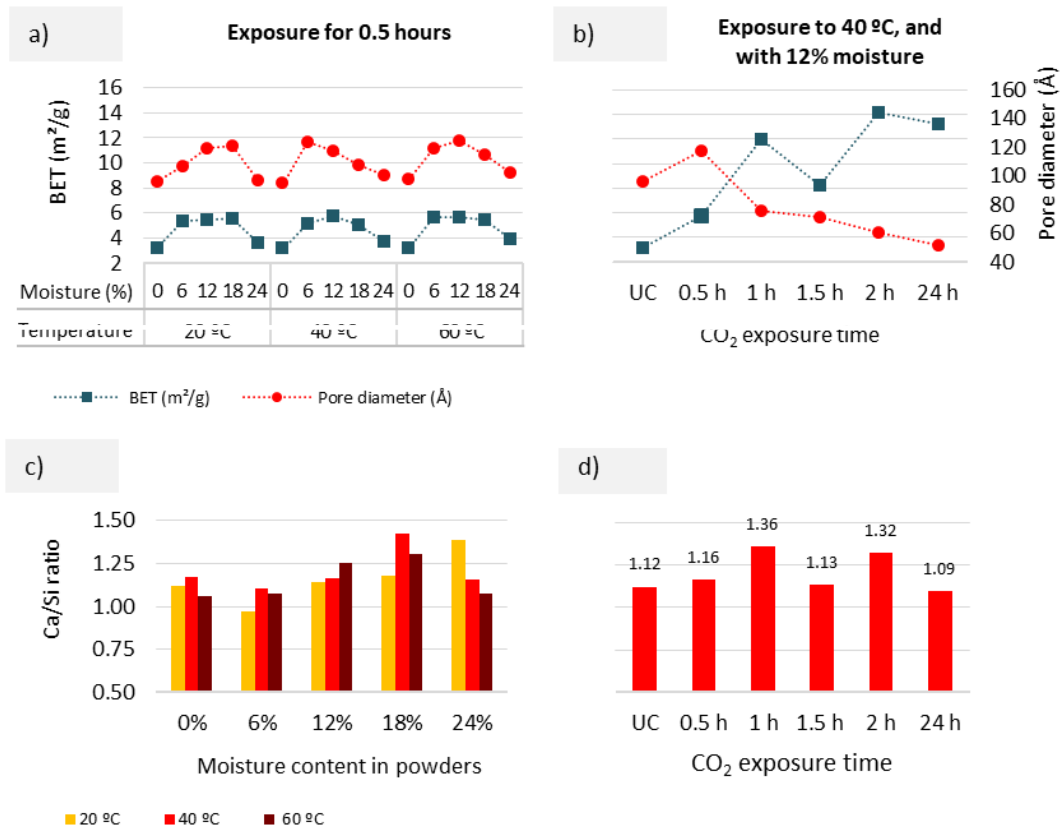
Figure 3-Mineralized RCP physicochemical characteristics: a) pH after a 0.5 h exposure; b) RCP pH with 12% moisture content; c) Density after a 0.5 h exposure; d) RCP density with 12% moisture content



An increase on specific mass was observed in powders submitted to CO₂ (Figure 3c), for all temperatures and humidities evaluated (in about 2%). The difference between the hydrates and solid volume after mineralization can justify this property alteration [87]. For the exposure time (Figure 3d) it was obtained values between 2781.0 and 2808.6 kg/m³, an increase of about 3%.

It is noted that the mineralized RCP surface area – BET is independent from test conditions and varied between 3.1 and 6m²/g (Figure 4a), reaching values close to 14 m²/g with a 24-hours CO₂ exposure (Figure 4b). A wide range of values was verified in the literature, between 5 and 40 m²/g depending on the mineralization process and raw material source (concrete or cement paste) [69,88,89]. When comparing the uncarbonated RCP (3.3 m²/g), there was an increase in surface area with mineralization, begin more highlighted in a 12% humidity condition at 40°C (5.7 m²/g) and 60 °C (5.6 m²/g). This surface area increase could be related to the formation of smaller CaCO₃ particles when submitted to higher temperatures [69,90] and to the decrease on capillary pores diameter due to carbonate hydration products conversion [91], finding also reported on this research (Figure 4a and 4b). According to Xiao *et al.* [92], the temperature arises as a critical factor between the mineralization conditions, improving the vaterite formation from 30 ~ 40 °C and aragonite above 60°C, exerting different effects when applied to cementitious materials [93,94]. It was reported that the silica gel precipitation induced by aqueous carbonation originated an increase on surface area from 95 to 132 m²/g [95], while the gas-solid process there is a reduction in this property with the increase on exposure time [96].

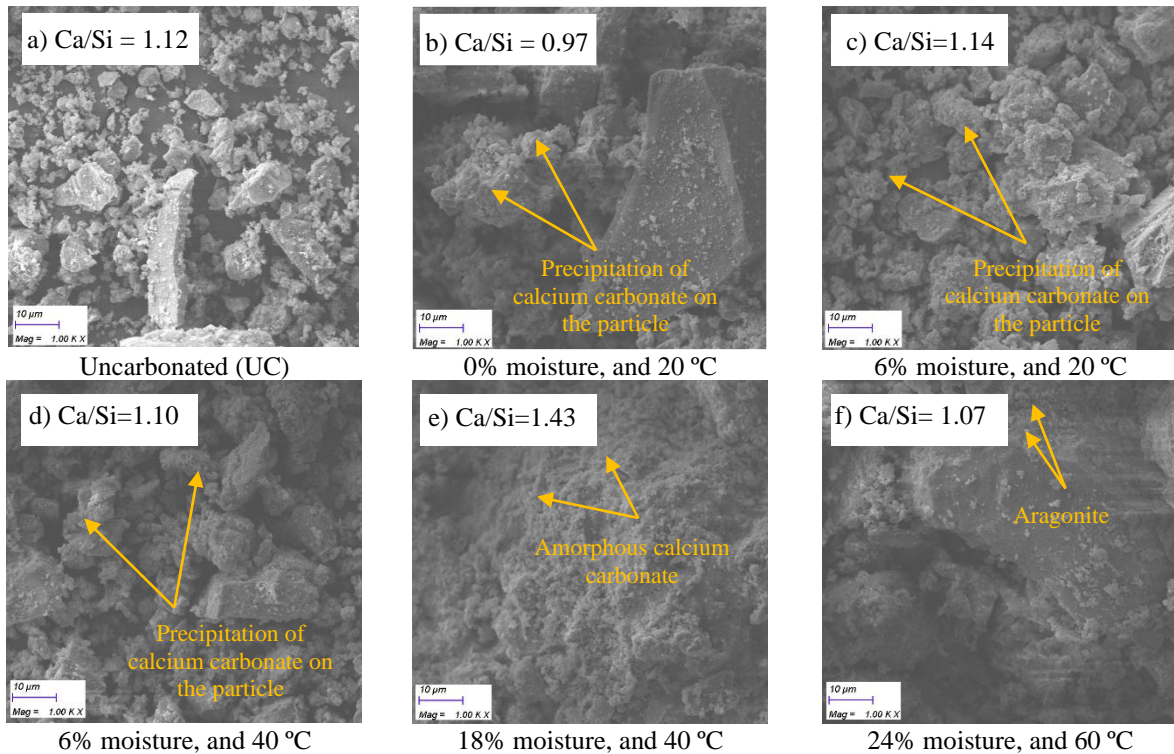
Figure 4—Relationship between BET Surface Area, Pore Diameter, and Ca/Si Ratio of Mineralized RCP: a) BET Surface Area after a 0.5 h exposure; b) BET Surface Area of RCP with 12% moisture content; c) Ca/Si Ratio after a 0.5 h exposure; d) Ca/Si Ratio of RCP with 12% moisture content



With RCP mineralization there was a variation on Ca/Si ratio from 0.97 to 1.31, for the combination between humidity and processing temperature (Figure 4c), while for the exposure time the values were presented between 1.09 and 1.36 (Figure 4d). According to Sevelsted *et al.* [97] during the mineralization, initially there is the gradual C-S-H decalcification [98] until the Ca/Si ratio reach 0.67 and, in sequency, occurs the amorph silica formation. Based on this, the results suggest that the carbonation products changed RCP microstructure, forming a CaCO₃ ‘shield’ on particles surface, fact that could be preventing the total C-S-H decalcification and polymerization [20,99,100].

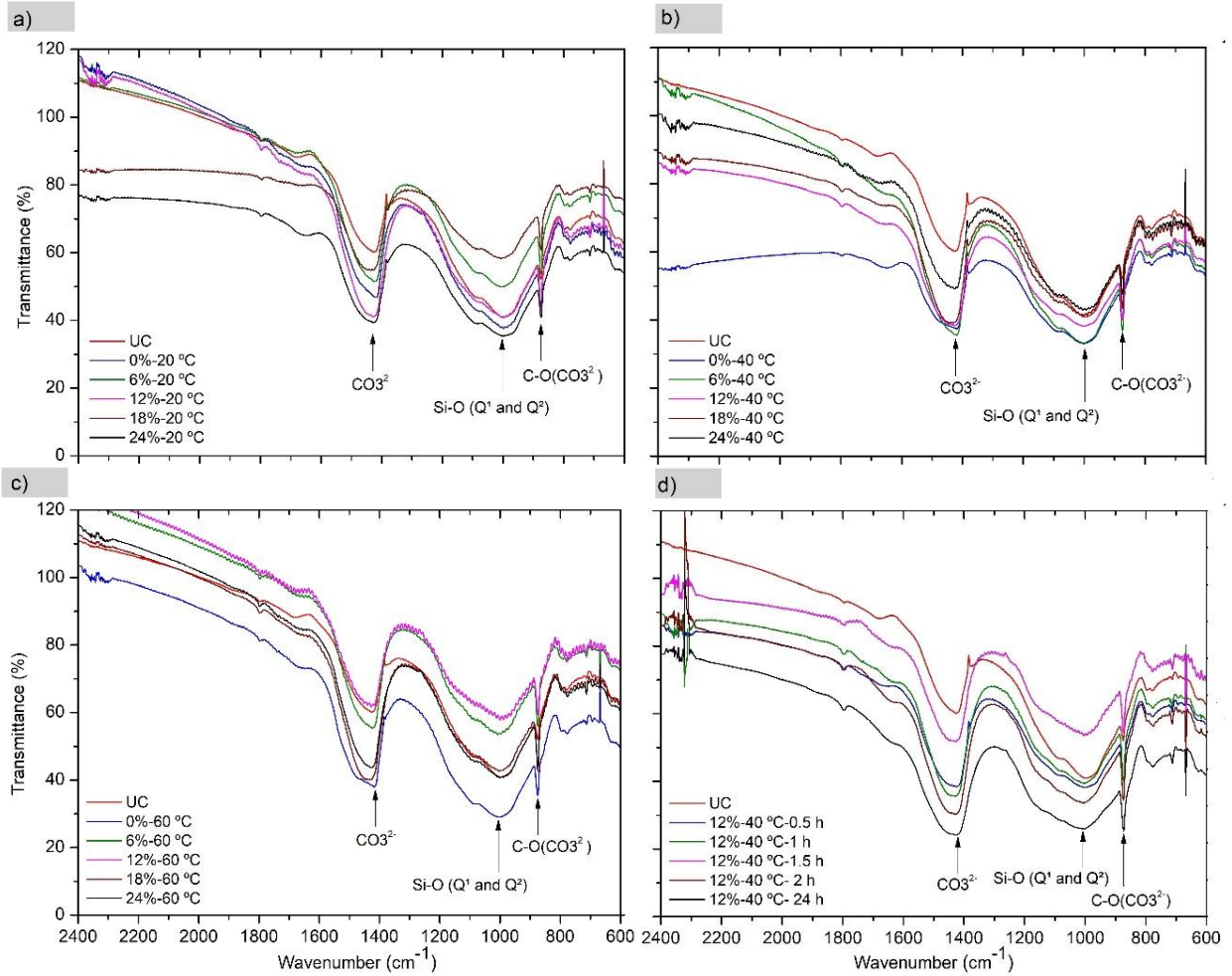
According to morphological alterations observed on RCP (Figure 5), this barrier formation by CaCO₃ precipitation was intensified by the water presence on humidified samples and the mineralization temperature. A small quantity of aragonite crystals in needle shape was detected in RCP mineralizaed with 24% humidity at 60°C (Figure 5f). It is noted the water presence and temperature influence on mineralization efficiency converging to the observations reported by Lu *et al.* [94] and Ding, Li; Chang [101] that stated that the aragonite formation is favored in temperatures higher than 60°C. The calcite and vaterite precipitate on lower temperatures, however, the vaterite is easily turned into calcite or aragonite [90,92,102].

Figure 5-Scanning Electron Microscopy (SEM) image of mineralized RCP under the specified conditions, at 1.00 KX magnification: a) uncarbonated; b) 0% moisture, and 20 °C; c) 6% moisture, and 20 °C; d) 6% moisture, and 40 °C; e) 18% moisture, and 40 °C; f) 24% moisture, and 60 °C



On FTIR Analysis, sudden vibrations were observed around 875 cm^{-1} , 1410 cm^{-1} and in the region $940\text{--}1200\text{ cm}^{-1}$ (Figure 6). The band located around 1410 cm^{-1} is related to asymmetric stretching (ν_3) on the C-O bond present on the CaCO_3 and the band located around 875 cm^{-1} corresponds to the out of plan flexion vibration (ν_2) from the same bond C-O [103,104]. The region between 800 to 1200 cm^{-1} indicates Si-O bond stretching [99], being the exact position dependent on Ca/Si ratio [105]. The stronger silicates band, present at 990 cm^{-1} , which exist on the naturally carbonated powder, changes on mineralized samples, which can be attributed to C-S-H decalcification-polymerization (Q^1 e Q^2) during carbonation [106,107], being 998 cm^{-1} and 994 cm^{-1} respectively for RCP mineralized with 0 and 12% at 20°C , 1001 cm^{-1} for 12%- 40°C , 1001 cm^{-1} for 18% - 60°C and 1006 cm^{-1} for 12%- 40°C and a 24-hours exposure. This change for a higher wavenumber occurs while the carbonation progresses [34,99]. These results confirm water amount, temperature and exposure time influence on recycled concrete powders CO_2 fixation. The decalcified C-S-H production (Q^3) is characterized by C-S-H with lower Ca/Si ratio and lattice silicate tetrahedron, and the polymerized silica (Q^4), defined by an amorph silica with a highly condensed silica tetrahedron network [108,109] that were not identified on spectrums, are in agreement with Ca/Si ratios superior to 0.67 and pH values higher than 9 verified on this study.

Figure 6-Mineralized FTIR spectrum: a) 20 °C; b) 40 °C; c) 60 °C; d) Different CO₂ exposure times

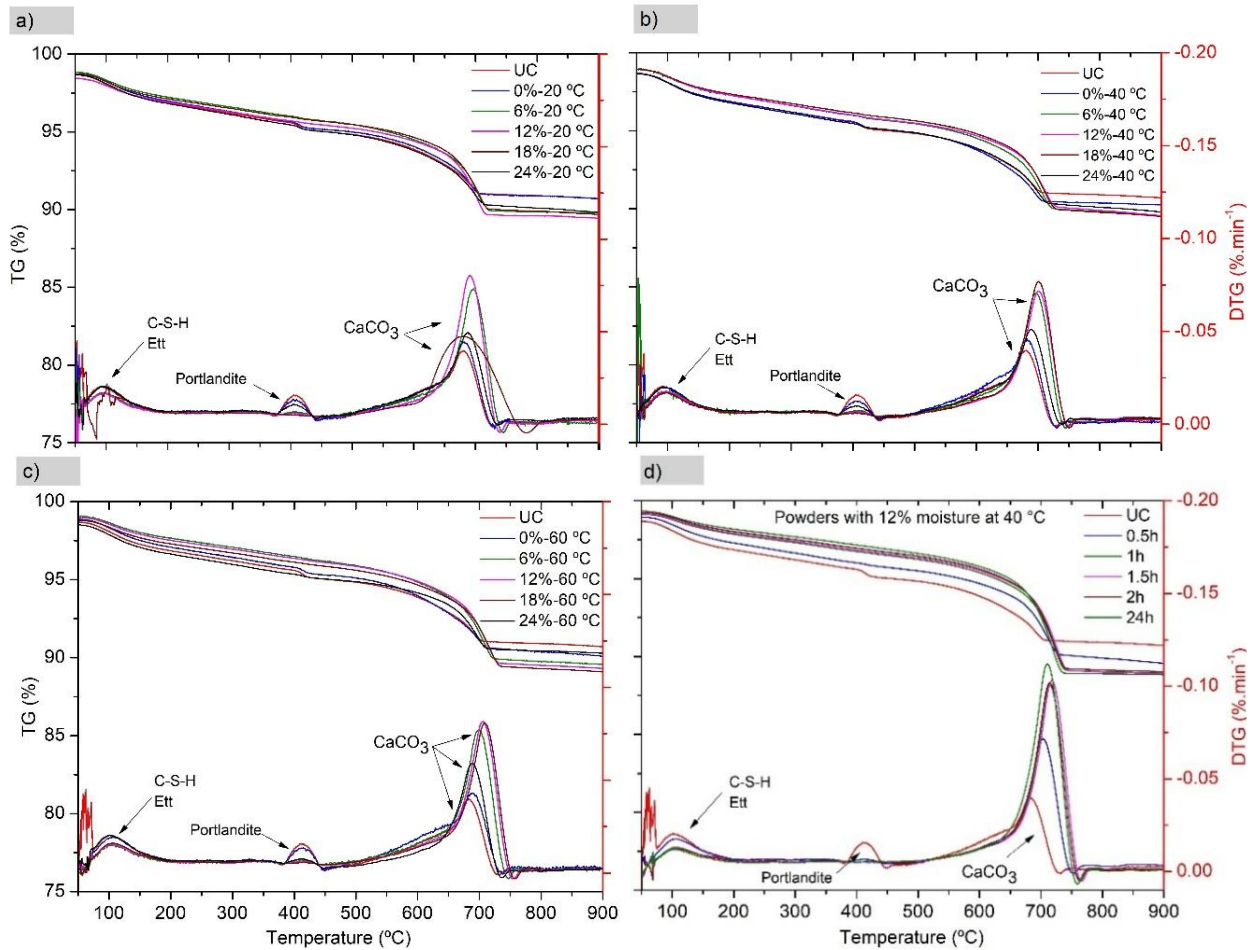


3.2 CO₂ FIXATION

By the thermogravimetry analysis mass losses are observed in temperature ranges of 50–200 °C and 380–450 °C, that corresponds to C-S-H hydration products dehydration (C-S-H and ettringite – Ett) and portlandite (CH) [66,69]. For RCP with humidity between 6 and 18%, after mineralization, the CH peaks intensity decreases accompanied by a higher mass loss after 550 °C, associated with CaCO₃ decarbonation (Figure 7).

Considering the same humidity, the higher the carbonation temperature the more significant is the peak related to CaCO₃ decomposition and wider if the temperature range with mass loss (Figures 7a, 7b and 7c). This indicates that the carbonation degree was higher and, consequently, there is a higher amount of amorph CaCO₃ formed [98,110,111] and more CO₂ are permanently fixed. With a prolonged carbonation period (Figure 7d), a higher CO₂ fixation are verified, according to the calcium carbonate decomposition peaks. This behavior is corroborated by pH results, that presented reduction from 12.5 to 9.53 after CO₂ exposure for 24 hours, indicating the mineralization process progression.

Figure 7- TG/DTG curves for mineralized powders at: a) 20 °C; b) 40 °C; c) 60 °C; d) Powders with 12% moisture at 40 °C under different CO₂ exposure times



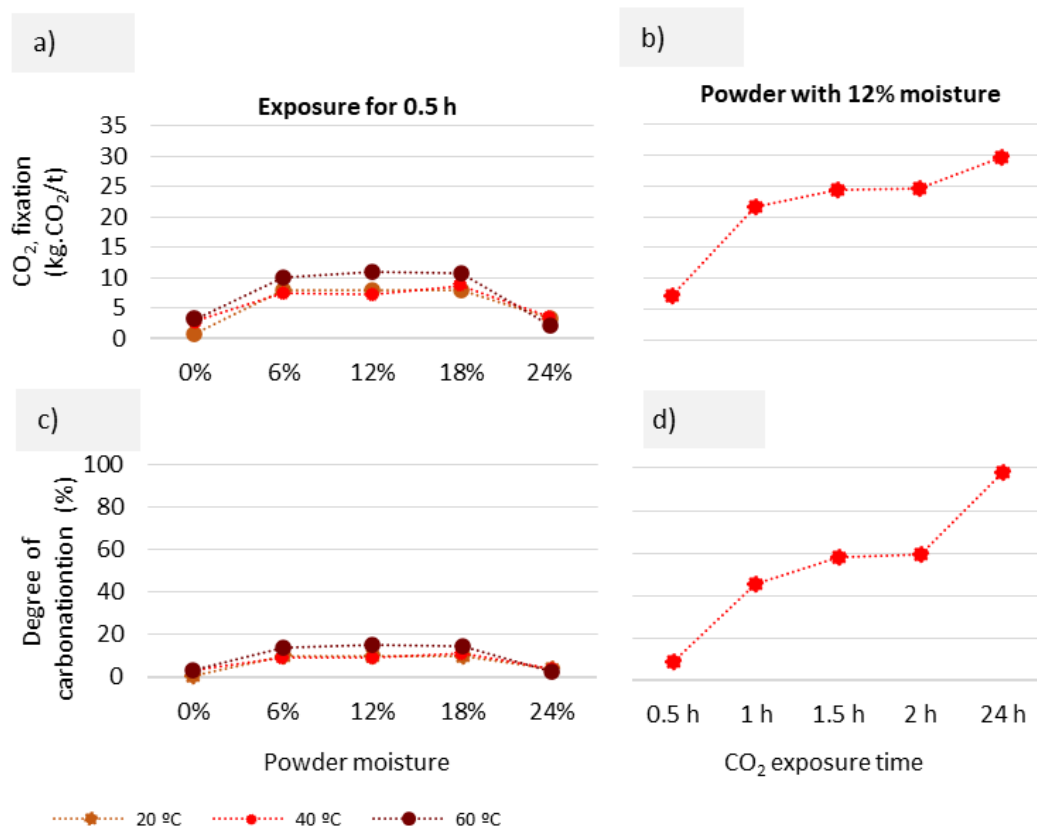
Through the thermogravimetry Analysis, RCP carbon fixation values were quantified and are shown on Figure 8. The humidities between 6 and 18% favored carbon fixation, being obtained – for this humidity range – values between 7.90 to 8.01 kg.CO₂/t for 20 °C, 7.24 to 8.66 kg.CO₂/t for 40 °C and 10.15 to 10.95 kg.CO₂/t for 60 °C. With 24-hours exposure 29.47 kg.CO₂/t was fixed. It is noted that, from 1.5h to 24h, the carbon fixation was less pronounced, behavior that could be related to carbonates formation on particles surface that reduced porosity and CO₂ diffusion [112–114]. On Kaliyavaradhan, Li and Ling [31] research, for mineralization conditions of 10 to 70% humidity and exposure periods between 1 and 168 hours, 0.3 to 29.4 kg.CO₂/t were fixed for recycled concrete powders. Values found are inferior to the obtained when employed powders from hydrated cement pastes or powder from cement furnace (CKD) which residues are composed basically of cement (with CaO rates superior to 60%). With this, the carbon fixation obtained was in the order of 115 to 270 kg.CO₂/t [38–40] for cement pastes and 270 kg.CO₂/t [115] for CKD.

The humidity showed a similar effect on carbonation performance for RCP in all temperatures, with carbonation degrees between 0.5 and approximately 15%, but being superior to 60°C temperatures. The rise on temperature accelerated CO₂ diffusion on RCP pores [114] elevating the carbonation degree. By adopting temperatures lower than 100°C, water evaporation was avoided, what could reduce the reaction necessary humidity length of stay, not compromising RCP mineralization efficiency [14,110]. The RCP with 0%

humidity fixed almost no carbon, proving that water presence is a necessary way to achieve the carbonation reaction due to carbonic acid formation [110,116].

On the other side, the water excess (24%) harmed mineralization efficiency on the gas-solid process, since the CO_2 diffusion coefficient on water is much lower than in air [117,118], so for 30 minutes exposure to CO_2 (fast carbonation) the great humidity range for carbon fixation on RCP is between 6 as 18%, being higher the fixation obtained at 60°C. With the exposure time increased to 24 hours, a 98% carbonation degree could be achieved, noted the CaCO_3 deposition around the particles seems to be limiting carbon fixation for the timeframe between 2 and 24 hours. The mechanochemical carbonation can be considered an alternative to improve mineralization efficiency, noticing the surface area exposure in function of the time [119,120].

Figure 8- CO_2 Fixation and Carbonation Degree for: a) CO_2 fixation as a function of temperature and moisture combination; b) CO_2 fixation as a function of CO_2 exposure time; c) Degree of carbonation as a function of temperature and moisture combination; d) Degree of carbonation as a function of CO_2 exposure time.



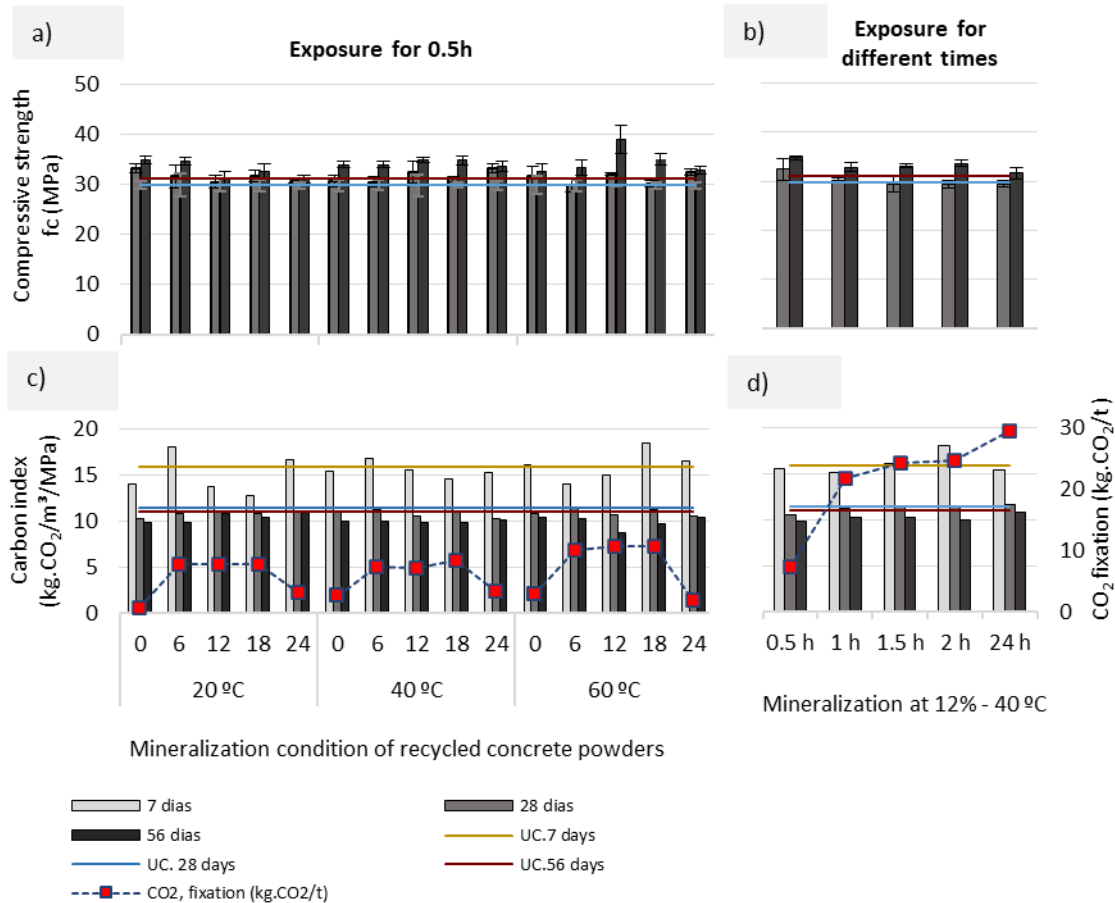
It is observed that humidity, temperature and CO_2 exposure time are key variables for the process performance, directly affecting the CO_2 fixation capacity, the particles morphology, the specific mass and the RCP surface area.

3.3 MORTARS' COMPRESSIVE STRENGTH

It is observed that, on 28 and 56 days, all mortars with 25% mineralized powder presented compressive strength superior to mortars with uncarbonated powders (UC) (Figure 9a). The compressive strength results are promising, considering that the replacement level of 25% are superior to the indicated and recommended by the literature, that recommend lower or equal to 20% [31,34].

CaCO_3 formation on particles surface acts as nucleation point, accelerating the cement hydration, which leads to an increase on compressive strength [34,66]. The RCP with 12% humidity, mineralized at 40°C and 60°C for 0.5 hours (Figure 9a), resulted on mortars with strength class C32, according to the Brazilian standard [72]. Simultaneously, the combination between partial cement replacement and particles CO_2 fixation promoted improvements on mortars environmental performance. This is reflected on the Carbon Index (CI), that were 8.7% (10.49 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$) and 7.9% (10.59 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$) lower, respectively, when compared to the values obtained on RCP uncarbonated (11.43 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$). Values are also inferior to the CI from cement CPI at 28 (10.81 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$) and 56 days (10.23 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$).

Figure 9–Compressive strength and Carbon Index considering the 25% replacement of cement mass



Considering the 56 days age (Figure 9a and 9c), the mineralized RCP with 12% humidity at 60°C presented a compressive strength of 38.88 MPa, reflecting in a CI 25% inferior (8.73 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$) to the one from uncarbonated powders (UC). On advanced ages, an improvement tendency is observed on both indicators (higher compressive strength and lower CI), which suggests a slower hydration on early stages, behavior widely reported on literature about recycled concrete powders [58,121].

The CO_2 exposure period influences the CI (Figure 9b and 9d). More elevated periods allow higher carbon fixation; however, it reduces the particles reactive potential for employment as SCM. Also, it elevates the energy demand for particles treatment (mineralization) and the carbon dioxide consumption. RCP mineralization when with 12% humidity at 40°C per 24 hours resulted in a CI of 11.58 $\text{kg.CO}_2/\text{m}^3.\text{MPa}$, a value 1.3% superior to the one obtained from the non-carbonated RCP. The Analysis of Variance (Table 3)

showed that the interactions between humidity and temperature, CO₂ exposure period and age directly interfere on mortars compressive strength.

Table 3-ANOVA analysis parameters for the mortar's compressive strength

Variables	Sum of squares	df	Mean Square	F-value	p-value	Remarks
Temperature (20, 40 and 60 °C)	260.07	2	130.04	1.0E+0.0009	0.0000	significant
Moisture (0, 6, 12, 18 and 24 %)	704.02	4	176.01	1.0E+0.0009	0.0000	significant
Time (0.5, 1, 1.5, 2 and 24 hours)	1097.95	4	274.49	1.0E+0.0009	0.0000	significant
Temperature*Moisture	-7.23E+0001	8	-9.04E+00	1.0E+0.0009	0.0000	significant
Error	-7.32E+0001	0	0.00E+00			

df: degrees of freedom

So, focusing on the CO₂ fixation, the strength class C32 and the carbon indexes, powders mineralized for only 30 minutes with 12% humidity in temperatures between 40 and 60°C presented a promising way for less emissive cement production, for CCUS technology applications and circular economy promotion, being the application a 25% cement replacement for recycled concrete powders.

4 CONCLUSIONS

On this study, the sample humidity (0 to 24%), temperature (20, 40 and 60°C), and CO₂ exposure period (0.5, 1, 1.5, 2, and 24 hours) influence were evaluated for recycled concrete powders (RCP) submitted to mineralization by carbonation on a gas-solid process by applying different techniques and characterization methods. With these results, a few conclusions can be drawn:

- It was concluded that the mineralization process control conditions, especially sample humidity, treatment atmosphere temperature and CO₂ exposure time, affects powders' carbonation rate and mechanical performance when applied as supplementary cementitious material;
- The CO₂ Mineralization reduced powders pH from 12.5 to values above 9, with increment on specific mass and surface area and reduction on pore volume, indicating microstructural alterations;
- With the CO₂ mineralization on RCP, a variation on the Ca/Si ratio was verified with values between 0.97 and 1.31, with some even lower than the presented on the reference material (1.21). An increased Ca/Si ratio on RCP and the mineralization conditions did not favor silica gel formation, according to the data from FTIR analysis;
- The great sample humidity for recycled concrete powder CO₂ mineralization is between 12 and 18%, for a 60°C temperature;
- The temperature is a critical factor on powders mineralization with CO₂, favoring the formation of vaterite 30 ~ 40 °C between and aragonite at temperatures higher than 60°C;
- Recycled concrete powders mineralization in periods superior to 0.5h promoted higher CO₂ fixation, reaching 29.47 kg.CO₂/t, however did not promoted improvement on mortars compressive strength when the material were applied as supplementary cementitious materials as a 25% cement mass replacement;
- Considering the compressive strength at 28 days (>32 MPa) and the carbon index indicator (CI), RCP mineralization with 12% humidity for 30 minutes at 40°C was the one with more adequate condition for employment as SCM. In more advanced ages, like 56 days, in is recommended mineralization at 60°C;
- Silica gel formation absence, resulting from the calcium silicate hydrate (C-S-H), highlights that the RCP reactivity is strongly conditioned to physico-chemical characteristics and mineralization conditions applied.

RCP carbon fixation represents a promising strategy to construction waste valorization, promoting circular economy, that could be considered in carbon credits negotiations. Future research can explore in humidity grinding method application as well as mechanochemical carbonation. It can also introduce additives during mineralization to improve carbon fixation. Besides that, durability tests for longer periods and resistance to aggressive agents should be evaluated for future RCP mineralized application on real scale.

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3.5 ARTIGO 5 (COMP. FÍSICO-QUÍMICA)

O artigo 5: *Mineralização de CO₂ em pós reciclados de concreto de múltiplas fontes para uso como material cimentício suplementar em cimento Portland*, apresenta resultados parciais, uma vez que algumas análises dependem da disponibilidade de equipamentos laboratoriais conduzidos em parceria com outras instituições. O método aplicado e os resultados obtidos até o momento oferecem contribuições relevantes para os objetivos propostos e serão aprofundados em trabalhos futuros.

Investigou-se a influência da composição do pó reciclado de concreto na fixação de CO₂ e no desempenho de argamassas quando usado como material cimentício suplementar (MCS) ao cimento Portland. Foram estudados pós reciclados obtidos da cominuição de concretos dosados em central, e em laboratório pela combinação entre duas relações água/cimento (0.55 e 0.65) e dois tipos de cimento (IV, V).

Os pós, pré-hidratados com 12% de umidade, foram submetidos à mineralização por carbonatação acelerada em câmara de carbonatação a temperatura de 60 ± 2 °C, umidade relativa de $60 \pm 10\%$, taxa de CO₂ de 15% em diferentes tempos (15, 30, 45 e 120 minutos). As características físico-químicas dos pós reciclados antes e após a carbonatação foram avaliadas por meio de técnicas analíticas e a fixação de CO₂ foi quantificada por termogravimetria (TG). O uso dos pós mineralizados foi avaliado conforme a NBR 7215 pelo método de determinação da resistência à compressão de argamassas, considerando 15%, 25% e 40% de substituição do cimento Portland.

A fixação de carbono nos pós reciclados variou entre 7 a 25 kg CO₂/t com potencial de contribuir de maneira eficaz para a redução das emissões de gases de efeito estufa na indústria cimenteira quando substituído até 25% do cimento Portland. Esses valores foram influenciados principalmente pela granulometria e pela composição química dos concretos que deram origem aos pós, sendo observada menor fixação nos pós produzidos com cimento CP IV e menor porosidade. O tempo de mineralização impacta na fixação de carbono, embora a relação não seja linear.

Apresenta-se, a seguir, o artigo, cuja submissão a periódico indexado na base Scopus ocorrerá quando as análises dos resultados estiverem concluídas.

CO₂ MINERALIZATION OF RECYCLED CONCRETE POWDERS FROM MULTIPLE SOURCES FOR USE AS A SUPPLEMENTARY CEMENTITIOUS MATERIAL IN PORTLAND CEMENT

Highlights

- CO₂ mineralization by the gas–solid process in recycled concrete powders (RCP) containing 17.04% to 28.99% CaO.
- Fixation of 7 to 25 kg of CO₂ per ton of RCP mineralized for 30 minutes.
- Improvement of environmental indicators by replacing up to 25% of Portland cement (CP I) with RCP mineralized for 30 minutes.
- Potential use of carbonated powders as supplementary cementitious materials (SCMs) in low-emission cementitious matrices.

Abstract

This study investigates the influence of recycled concrete powder composition on CO₂ fixation and performance when used as a supplementary cementitious material (SCM) in Portland cement matrices. Four recycled powders were obtained by crushing laboratory-produced concretes with controlled water-to-cement ratios (0.55 and 0.65) and cement types (CP IV and CP V), along with one powder of uncontrolled origin (RCP), derived from the recycling of concretes from a batching plant, which was also ground (RCPg). The powders, pre-hydrated with 12% moisture, were subjected to accelerated CO₂ mineralization in a carbonation chamber (60 ± 2 °C, relative humidity of 60 ± 10%, and CO₂ concentration of 15%) for different durations (15, 30, 45, and 120 min). The physical and chemical characteristics before and after carbonation were determined by density, pH, specific surface area, and SEM-EDS analyses, while carbon fixation was quantified by thermogravimetric analysis (TGA). The performance of the carbonated powders as SCMs was evaluated by compressive strength tests of mortars at 28 and 91 days, replacing 15%, 25%, and 40% of Portland cement by mass. Carbon fixation ranged from 7 to 25 kg CO₂/t, demonstrating the potential of recycled powders to significantly contribute to the reduction of greenhouse gas emissions. The rapid CO₂ mineralization (30 min) led to efficient CO₂ fixation and mechanical performance equivalent to mortars produced with non-carbonated powders. The particle size distribution of the powders and the type of cement used in the original concrete were identified as key factors influencing the CO₂ fixation capacity and the potential use of recycled powders as SCMs. In the context of the decarbonization of the cement industry, these findings highlight the potential of recycled concrete powders as large-scale generators of carbon credits.

Keywords: Mineral carbonation; CO₂ uptake, Carbon capture, utilization and storage (CCUS).

1 INTRODUCTION

Portland cement is one of the most widely used materials in the world, essential for infrastructure and housing construction. With an estimated global production of about 4 billion tons in 2024 [1], its use has a significant environmental cost: approximately 1.56 billion metric tons of carbon dioxide emissions [2], which represent between 5% and 8% of the world's CO₂ emissions [3,4]. The consequences of CO₂ emissions include various environmental and climatic impacts, including increased global temperatures, changes in rainfall intensity and distribution, more frequent and severe extreme weather events, sea-level rise, and ecosystem disturbances, among others [5,6].

Considering this scenario and the growing pressure to reduce emissions, in 2022, the global cement and concrete industry committed to achieving carbon neutrality by 2050. Among the strategies considered by the sector, Carbon Capture, Utilization and Storage (CCUS) technologies are estimated to account for 37% of the CO₂ emitted, about 1.37 billion tons [7]. Geological injection into underground reservoirs and carbon mineralization are two possible routes for long-term carbon storage [8,9]. The first technique, which is more mature, tends to offer a greater contribution to stored volume, while the second stands out for its stronger security [10,11].

Mineral carbonation (MC) was proposed 30 years ago as a way to fix CO₂ [12,13] permanently and involves the formation of solid carbonate minerals with rocks rich in calcium or magnesium (wollastonite, olivine,

serpentine, pyroxenes) [8,9,14], mine tailings [15] and industrial by-products such as steel slag [16] and fly ash [17]. The process can be done in situ by injecting into geological formations, or ex situ by transporting materials to an industrial plant for gas treatment in reactors [9,18].

Ex-situ MC offers the advantage of valorizing or stabilizing waste, making this technology attractive [8,18,19]. Various applications are registered for carbonate minerals, such as fillers in paper and pulp production, pH-reducing agents in the pharmaceutical sector, constituents of fertilizers for agricultural use, and raw materials in the manufacturing of refractory metals and cements [14]. Furthermore, due to their reduced particle size and availability, industrial wastes are presented as ideal materials for mineral carbonation [14,20].

In the construction industry, about 3 billion tons of construction and demolition waste (CDW) are generated [21]. During their recycling and transformation into aggregates, about 20% of a residual powder is generated, with particles smaller than 0.15 mm [22,23]. Compared to Portland cement, they have high levels of SiO₂ (>30%), CaO (<17%), alumina (>6%), and iron oxide (>4%) [24], whose content varies according to the origin of the waste and the type of aggregate [25–27], and which, after mechanical [28,29], thermal [30] and/or chemical treatments [31,32], have the potential to replace up to 30% of the mass of Portland cement [23,33].

In a more recent approach, mineral carbonation has been studied not only to fix CO₂ but also to improve the performance of these powders as SCMs [34,35] through the precipitation of calcium carbonate (CaCO₃) and the formation of the silica-alumina gel [22,36,37], reported in the literature as having a pozzolanic behavior [38,39].

Various studies on temperature, pressure, particle size, reaction time, reactor type, solid-liquid ratio, CO₂ concentration, diffusivity, and other factors have been conducted to understand the kinetics of the carbonation reaction and maximize CO₂ fixation [36,40], however, it was observed that most works are based on waste with high CaO contents. Xuan et al. [41] for example, after 7 days in a hermetic stainless steel container, verified the fixation of 110 kg CO₂/t of concrete slurry waste (CSW) (CaO=64.51%, SiO₂=19.57 %, MgO=1.48%). The material was dried and manually crushed, using particles smaller than 0.15 mm, while He et al. [42], through mechanochemical carbonation in 0.5 h, achieved a fixation of 221 kg.CO₂/t of CSW (CaO=44.35%, SiO₂=27.09%, MgO=2.07%). Adekunle [43], for Cement Kiln Dust (CKD), identified several studies using this material, with CaO contents in the range of 37% to 77% and notably high alkalinity, which showed a fixation capacity between 15 and 270 kg CO₂/t of CKD; however, the presence of alkalis Na₂O and K₂O are pointed out as a technical concern for their use in cementitious matrices.

The use of Recycled Cement Paste Powder (RCPP) produced in the laboratory was observed to simulate the behavior of Recycled Concrete Powder (RCP) obtained from CDW recycling [44], but without the influence of aggregates and other impurities [45,46]. Fang and Chang [47], reported RCP (CaO=61.13%, SiO₂=21.45 %, MgO=2.08%) and carbonation of powders pressed into molds fixed at about 198 kg CO₂/t. While Zhao *et al.* [46], with ground particles Ø 2 mm and humid (CaO=64.99%, SiO₂=19.36%, MgO=3.25%) after mineralization in a reactor for 24 h, fixed about 297.5 kg CO₂/t RCP.

Regarding the application of the material as SCM, Lu *et al.* [22], in RCPP (CaO=64.5%, SiO₂=21.91%, MgO=1.52%) with particles smaller than 0.75 mm and fully carbonated, verified a compressive strength about 32% higher compared to the non-carbonated powder when 20% of the cement mass was substituted in pastes. In turn, Wu *et al.* [48] observed that the water-cement (w/c) ratio and the presence of SCMs such as fly ash, silica fume, and mineral powder in the source pastes of the RCP influenced the compressive strength of mortars; the use of carbonated powder provided increases of up to 12.6% compared to the use of non-carbonated powder, when 30% of the cement was substituted. It is reported that the use of carbonated RCPP accelerates the early hydration of cement through the so-called filler effect [39,49]. Particle reactivity is associated with the amorphous nature of silica gel, as compared to metakaolin and silica fume [39].

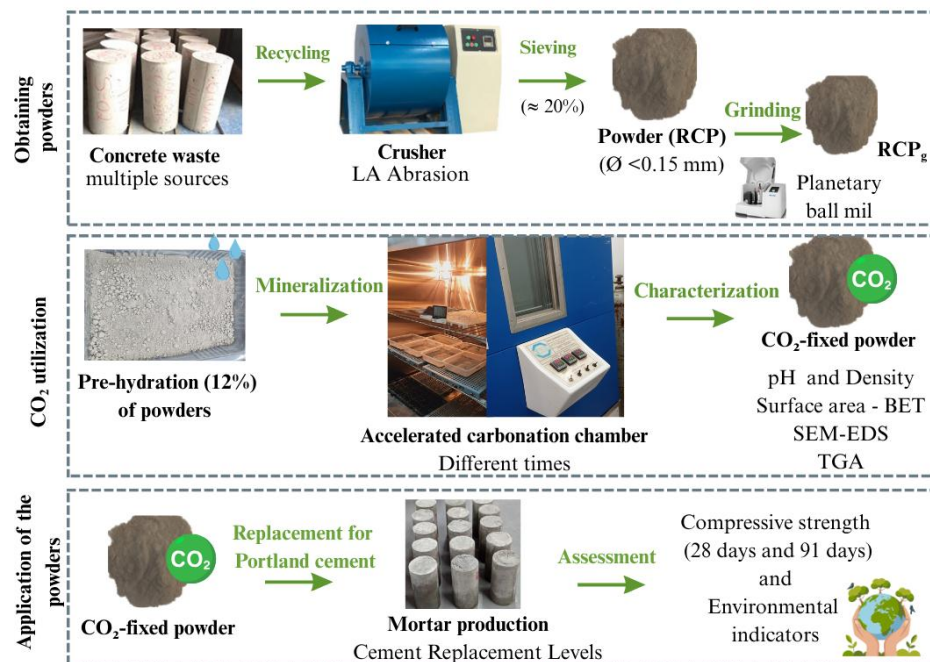
Few investigations were verified in the literature for powders obtained from CDW recycling. Kaliyavaradhan, Li and Liang [50], for RCP (CaO=32.49%, SiO₂=40.06%, MgO=2.16%) after 90 h of exposure and with w/s ratio =0.4, fixed 28.17 kg CO₂/t, achieving higher strengths than cement mortars containing 20% of carbonated RCP. Feng *et al.* [51] observed that, compared to non-mineralized RCP (CaO=38.2%; SiO₂=41.0%), mortars with 10% RCP treated by calcination at 600 °C, followed by carbonation for 24 h, showed gains of 17.6% in compressive strength and 24% in flexural strength. Quian [52], in recycled mortar powders (CaO=20.9%, SiO₂=69.6%, MgO=1.0%) after mineralization by accelerated carbonation in a reactor, verified a fixation of 20.8 kg CO₂/t, and the substitution of 10% of the cement provided a gain greater than 10% compared to the non-carbonated powder.

Although studies on the mineralization of cementitious materials have been advancing, many focus on waste with high CaO contents and controlled chemical composition. However, real construction waste presents a heterogeneous composition; thus, the objective of this work is to investigate the influence of the composition of different RCP sources on the efficiency of mineralization, expanding the understanding of the behavior of carbonated waste and contributing to integrated strategies for waste valorization and carbon fixation, with potential for generating credits in the carbon market.

2 MATERIALS AND METHODS

Different recycled concrete powders were evaluated for CO₂ fixation efficiency and mechanical and environmental performance when used as a cement substitute in mortars. The materials used are first presented, followed by their characterization. Next, the accelerated carbonation mineralization process is described, followed by the characterization tests. Finally, the molding of the mortars, the physical and mechanical tests, and the indicators used to evaluate the use of mineralized powders are presented. The study scheme is shown in Figure 1.

Figure 1- Schematic of the study



2.1 MATERIALS

All concrete powders studied (Table 1), from controlled and non-controlled sources, were obtained from the processing of cylindrical concrete specimens with dimensions (100×200 mm), which were crushed in a Los Angeles abrasion (LA abrasion) machine, using the fraction passing through the Mesh 100 sieve

($\varnothing < 0.15$ mm). The procedure was standardized by filling the machine with 30 kg of concrete, using 24 stainless steel spheres with a diameter of 48 mm and a mass varying between 390 and 445 g, operating at 30 rpm, and grinding for 2 hours.

Table 1- Information on the concretes of source of RCP

Identification	Source	Cement type	Water/cement ratio (w/c)	Compressive strength at 28 days (f_{c28}) - MPa
RCP RCPg	concrete plant (uncontrolled)		unknown	> 35*
RCP-IV.55		CP IV	0.55	17.1
RCP-IV.65	produced in the laboratory [53]	CP IV	0.65	11.5
RCP-V.55	(controlled)	CP V	0.55	27.2
RCP-V.65		CP V	0.65	24.9

* According to plant data, most concretes contain Portland cement, quartz sand, stone powder, basalt aggregate, and plasticizers.

RCP and RCPg originated from ready-mix concrete, with a non-controlled source. RCPg (Recycled Concrete Powder – grinding) was obtained by grinding the RCP in a PM 100 planetary ball mill (Retsch), equipped with a 250 cm³ agate grinding jar, rotating at 500 rpm following the protocol: filling with 60 g of powder, 50 agate balls with approximately 10 mm in diameter, using an additive in the powder and automatic reversal of the rotation direction every 15 min with a 1 min stop for cooling, considering that the mill temperature reached 37°C. Grinding was performed for 30 min with a 0.5% propylene glycol additive, as per [28]. The powders RCP-IV.55; RCP-IV.65; RCP-V.55 and RCP-V.65 were obtained from laboratory-batched concretes [53], produced with water/cement ratios of 0.55 and 0.65 and with the Brazilian cements [54] Portland-pozzolan cement (CP IV 32) and high early strength Portland cement (CP V), similar to Type IP of the ASTM C595 standard and Type III of ASTM C150, respectively.

For the production of the mortars, Ordinary Portland Cement (CP I) with 3.5% calcium sulfate (CaSO₄) [54] was used, similar to ASTM Type I cement (CEM I) in different countries [55]. The Filler Portland Cement (CP II F), equivalent to the ASTM limestone cement (IL) [56], was used as a reference, considering the high substitution rate of clinker by carbonaceous material (11%–25%) [54]. The chemical composition of the materials was determined by X-Ray Fluorescence (XRF) on a WDS Bruker S8 Tiger spectrometer, equipped with an Rh tube, and is shown in Table 2.

Table 2- Oxide composition of powders and cement (wt.%)

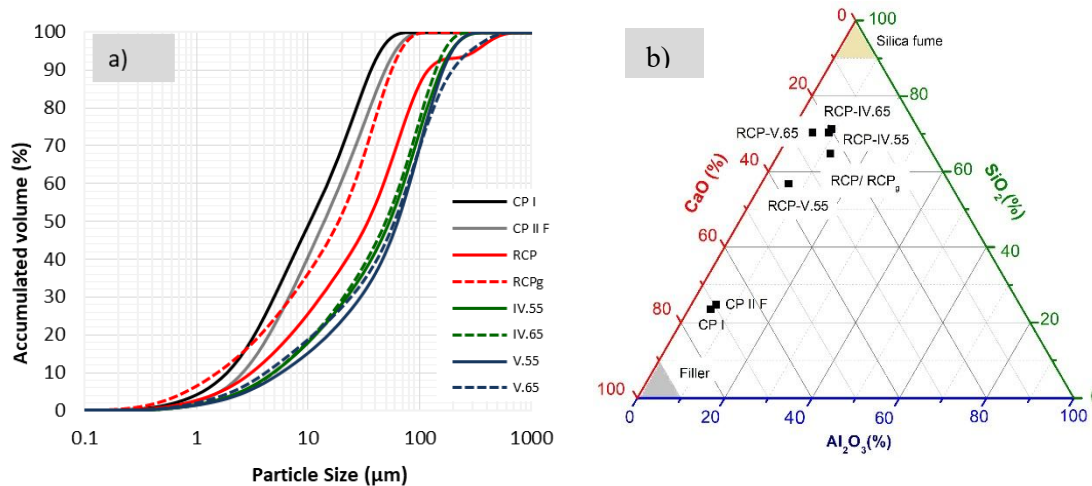
Materials	Chemical composition (%)										
	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	SO ₃	Na ₂ O	MnO	P ₂ O ₅	LOI
CP I	20.00	60.72	4.37	4.33	3.64	1.19	3.89	<LQ	<LQ	<LQ	1.95
CP II F	19.20	54.05	4.50	2.61	5.25	0.61	2.45	0.15	<LQ	<LQ	10.54
RCP/ RCP _g	48.38	17.52	8.86	9.11	4.36	0.88	1.45	0.2	1.23	0.69	6.59
RCP-IV.55	57.26	17.04	7.13	3.38	2.29	1.60	0.49	<LQ	0.41	1.04	9.33
RCP-IV.65	58.66	16.41	7.29	3.53	2.22	1.64	0.50	<LQ	0.44	1.09	8.98
RCP-V.55	44.53	28.99	4.94	3.35	1.48	1.34	0.51	0.10	0.37	1.30	13.33
RCP-V.65	59.15	20.78	4.26	2.98	1.00	1.44	0.47	<LQ	0.28	1.02	9.36

LOI: Loss on Ignition; <LQ: below the quantifiable limit.

The particle size distribution of the materials was analyzed using the Mastersizer 3000 particle size analyzer (Malvern Instruments), equipped with the Aero S accessory for dry powder dispersion [57]. Figure 2 shows the physicochemical characteristics of the materials. It is observed that the recycled powders have a high percentage of silica (SiO₂ - 60% to 65%) due to the presence of aggregates (quartz sand, basalt-origin crushed sand), calcium oxide (CaO - 16% to 30%), and alumina (Al₂O₃ - 4% to 9%), due to the presence of

partially carbonated hardened cement paste and unhydrated cement particles, as reported in the literature [27,33,58]. Although the sum of the components $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3 + \text{SiO}_2$ is greater than 50%, one of the requirements for pozzolanic materials [59], the powders may not necessarily exhibit pozzolanic activity, as some of the particles are crystalline [24,28]. The Loss on Ignition (LOI) values, which are higher than those of CP I and exceed 6%, are attributed to the decomposition of hydration products and to the amount of CaCO_3 present in the powders [29,58] and do not meet the Brazilian standard [59]. The recycling process [60] and the origin of the powders are factors that influence the compounds present in the RCP [27].

Figure 2-Physical-chemical characterization of materials: a) Particle size distribution of materials b) Ternary Diagram of SiO_2 - CaO - Al_2O_3



The specific gravity was determined by helium gas pycnometry in an Ultracyc 5000 pycnometer (Anton Paar) [61]. The surface area was determined by nitrogen adsorption (Brunauer-Emmett-Teller, BET) using a Quantachrome Nova 4200e instrument. Before the test, the powders were degassed at 40 °C for 16 h [62]. The potential hydrogen (pH) was determined in a solution containing approximately 50 ml of deionized water and 5 g of powder. After 10 minutes of mixing, the pH was measured about 30 minutes later using a digital pH meter [63]. Energy-Dispersive X-ray Spectroscopy (EDS) used with Scanning Electron Microscopy (SEM) enabled the quantitative determination of elemental ratios, such as the Calcium/Silicon (Ca/Si) ratio. The characterization of the materials, including the average particle diameter, is shown in Table 3.

The specific gravity values are lower than those of Portland cement due to the greater porosity of the concretes, which are composed of hydrated cement and silica [23,64] and ranged from 2446 to 2724 kg/m³. The pH value, higher than 12 (alkaline), classifies the RCP according to the Brazilian standard [65], as non-hazardous waste to the environment; however, the presence of heavy metals, such as aluminum (Al) and iron (Fe), would classify them as hazardous in other countries, such as Japan, Spain, and the UK [66]. Regarding the Ca/Si ratio, values ranged from 1.12 to 1.65. It is noted that the higher the strength of the source concrete (produced in the laboratory), the higher the Ca/Si ratio, following the order for the RCPs: V.55, V.65, IV.55, and IV.65. Furthermore, the concretes produced with CP V compared to CP IV showed higher Ca/Si ratios, justified by the presence of a higher CaO content in the CP V cements (See XRF of the cements in Table S1 - supplementary material). The higher Ca/Si value for the RCPg may indicate pre-carbonation during handling and grinding [67]. For the same type of cement, it was observed that the lower w/c ratio corresponded to a higher Ca/Si ratio. With less water, hydration is less complete, resulting in fewer hydration products, mainly portlandite, which affects the Ca/Si value [68].

Table 3-Material characteristics

Materials	d50 (μm)	Specific mass ρ (kg/m^3)	Surface area - BET (m^2/g)	ph	Ca/Si
CPI	11.78	3190.00	1.65	15.58	6.06
CP II F	15.80	3080.00	1.44	12.20	5.50
RCP	39.67	2723.20	3.13	12.69	1.12
RCP_g	21.78	2750.30	4.96	12.65	1.34
RCP-IV.55	63.08	2455.00	9.60	12.34	1.30
RCP-IV.65	57.65	2451.50	11.81	12.40	1.20
RCP-V.55	72.25	2446.30	5.85	12.35	1.63
RCP-V.65	67.18	2494.20	5.35	12.41	1.45

2.2 MINERALIZATION OF POWDERS AND CHARACTERIZATION

In plastic trays, approximately 250 g of the recycled concrete powders (Table 1) were pre-wetted with 12% water by spraying, followed by de-agglomeration, a process that lasted about 30 min. Then, they were placed in an accelerated carbonation chamber (Bass, UUC-RH-STD-CO₂-1000–1000/2016), under the following conditions: relative humidity of $60 \pm 10\%$, CO₂ flow of 5 L/min, CO₂ concentration of 15% (150,000 ppm), at 60°C for 15 min, 30 min, 45 min, and 120 min. The moisture content of the powders and the study temperature were defined based on a previous study [69].

After the mineralization process, the powders were placed in an oven at 40 ± 5 °C to prevent ettringite dehydration [62,70,71], and were subsequently characterized and applied as SCM in mortars. The powders were identified as follows: the non-carbonated ones were named with the source powder's acronym followed by UC (uncarbonated). For example, RCP-UC refers to the uncarbonated recycled concrete powder. The powders subjected to mineralization were identified by the letter C (carbonated), followed by the process duration. For example, RCP-C15 indicates the recycled concrete powder mineralized for 15 min.

The characterization of the mineralized powders was conducted following Brazilian standards or specific procedures reported in the literature and adopted by other authors (Table 4). Scanning Electron Microscopy (SEM) analyses were carried out using a Zeiss EVO MA10 microscope.

Table 4- Material characteristics

Characteristics	Methodology
Potential of hydrogen (pH)	Embrapa [72]
Specific mass - ρ (kg/m^3)	ASTM D5550 [61]
Specific Surface Area Brunauer-Emmett-Teller - BET (m^2/g)	Baggio <i>et al.</i> [73]
Morphology of the powders (SEM) coupled with Energy Dispersive X-Ray Spectroscopy (EDS)	Oliveira <i>et al.</i> [29]

The determination of CO₂ fixation in the powders was determined by thermogravimetric analysis, based on the difference between the peak corresponding to the decarbonation of calcium carbonate (CaCO₃), which occurs within the temperature range of 550 to 800 °C (in the form of calcite), of the carbonated and uncarbonated samples (Equations 1 and 2) [46,74]. Approximately 50 mg of the sample was placed in an open alumina crucible in the Perkin Elmer STA 8000 equipment. A nitrogen flow of 30 ml/min was applied with a heating range of 30 to 1000 °C and a heating rate of 10 °C/min. The adopted protocol was based on several other studies that applied the same technique [29,75,76]. Each sample was analyzed separately,

without assuming the same range for all samples, since the onset of thermal decomposition may vary with the degree of crystallinity of the CaCO_3 [77,78].

$$CO_2(\%) = \Delta m_{(550-800^\circ C)} \times \frac{MM_{CO_2}}{MM_{CaCO_3}} \quad \text{Equation 1}$$

$$CO_{2,fixation}(\%) = CO_2(\text{carbonated}) - CO_2(\text{uncarbonated}) \quad \text{Equation 2}$$

Where: MM_{CO_2} = molar mass of CO_2 (~44.01 g/mol), MM_{CaCO_3} = molar mass of CaCO_3 (~100.09 g/mol).

2.3 USE OF MINERALIZED POWDER

The use of powders before and after accelerated carbonation mineralization in mortars, prepared and molded according to NBR 7215 [79] similar to ASTM C1437-20 [80]. For a mass proportion of 1:3 (cement: quartz sand) and a water/cement ratio of 0.48, 25% of the CPI mass was replaced with recycled powder mineralized for 30 minutes, given their potential use as SCM [28]. For the RCP, substitution contents of 15% and 40% of the cement mass were also evaluated. The sand had a density of 2.65 kg/dm^3 and a particle size distribution defined by the standard, used in proportions of 25% for each fraction: 1.18 mm, 0.60 mm, 0.30 mm, and 0.15 mm.

After moist curing, the rectified cylindrical specimens (50 mm×100 mm) were crushed in a hydraulic press (model I-3025-B), with a loading rate of 0.25 ± 0.05 MPa/s, according to NBR 5739 [81], at 28 days and 91 days. The average of four specimens per age was adopted as the result. Analysis of Variance (ANOVA) was performed using the Sisvar software [82] at a significance level of 5% ($p \leq 0.05$) to evaluate differences between means.

To analyze the environmental benefits of mortars containing recycled concrete powders (UC or C), two indicators were evaluated: Binder Intensity (BI) and CO_2 Intensity (CI) [29,83], as defined by Equations 3 and 4, respectively. The CI indicator is used to compare the carbon emissions of mortars with different substitution levels [84,85].

$$BI \left(\frac{\text{kg}}{\text{m}^3} / \text{MPa} \right) = \frac{C_{\text{Cement}}}{f_c} \quad \text{Equation 3}$$

Where: C_{Cement} : Cement consumption (clinker, calcium sulfate, and limestone filler, kg/m^3); f_c : Compressive strength (MPa).

$$CI \left(\frac{\text{kg} \cdot \text{CO}_2}{\text{m}^3} / \text{MPa} \right) = \frac{E}{f_c} \quad \text{Equation 4}$$

Where: E: Total CO_2 emissions related to the consumption of materials (cement and powders) for the production of 1 m^3 of mortar ($\text{kg CO}_2/\text{m}^3$); f_c : Compressive strength (MPa).

The emissions from the cements and the uncarbonated powders were calculated using Equations 5 and 6. For the carbonated powders (Equation 7), in addition to the emissions associated with the processing they underwent, the carbon emissions from the electricity consumption for the operation of the carbonation chamber were added, and the CO_2 fixed in the particles (Table 5) was deducted.

$$E_{\text{cement}} \left(\frac{\text{kg} \cdot \text{CO}_2}{\text{m}^3} \right) = C_{\text{Cement}} * E_{\text{Cement}} \quad \text{Equation 5}$$

$$E_{\text{powder,NC}} = (C_{\text{powder,NC}} * E_{\text{processing}}) \quad \text{Equation 6}$$

$$E_{\text{powder,C}} = (C_{\text{powder,C}} * E_{\text{processing}}) + (C_{\text{powder,C}} * E_{\text{Carbonation chamber}}) - (CO_{2,fixation}) \quad \text{Equation 7}$$

Where: C: Material consumption (cement or powder) to produce 1 m^3 of mortar (kg/m^3); E: CO_2 emissions associated with the production of cement and powder ($\text{kg} \cdot \text{CO}_2/\text{t}$); $E_{\text{processing}}$: CO_2 emissions related to the sieving and/or grinding processes for powder production ($\text{kg} \cdot \text{CO}_2/\text{t}$); $E_{\text{Carbonation chamber}}$: CO_2 emissions

associated with the electricity consumption of the carbonation chamber ($\text{kg}\cdot\text{CO}_2/\text{t}$); $\text{CO}_{2,\text{fixation}}$: CO_2 fixation in the powder through the mineralization process ($\text{kg}\text{CO}_2/\text{t}$), obtained from Equation 2.

In the calculation of CO_2 emissions associated with the consumption of the carbonation chamber (Table 5), the average annual emission factor for 2024, equal to $\sim 46\text{ kg}\cdot\text{CO}_2/\text{MWh}$ ($0.046\text{ kg}\cdot\text{CO}_2/\text{kWh}$), reported by the Ministry of Science, Technology, and Innovation [86] was used. An equipment power of 5 kW [87] and a production capacity of about 150 kg of treated powder per duration of the mineralization process were assumed.

Table 5 - Values for carbon emissions

Data	Carbon emissions	Reference
E_{CPI}	890.29 $\text{kg}\cdot\text{CO}_2/\text{t}$ (Mean value)	[88]
E_{CPIIF}	762.70 $\text{kg}\cdot\text{CO}_2/\text{t}$ (Mean value)	
$E_{\text{powder}-\phi < 0.15\text{ mm}}$	6.36 $\text{kg}\cdot\text{CO}_2/\text{t}$	[89]
$E_{\text{powder-grinding}}$	24.5 $\text{kg}\cdot\text{CO}_2/\text{t}$ * grinding duration	[29]
$E_{\text{carbonation chamber}}$	1.53 $\text{kg}\cdot\text{CO}_2/\text{t}$ * duration of mineralization	[63]

In this study, emissions associated with the transportation of powders or gas, or with the production of CO_2 in cylinders, were not considered, as these data depend on the technological route to be adopted.

3 RESULTS AND DISCUSSION

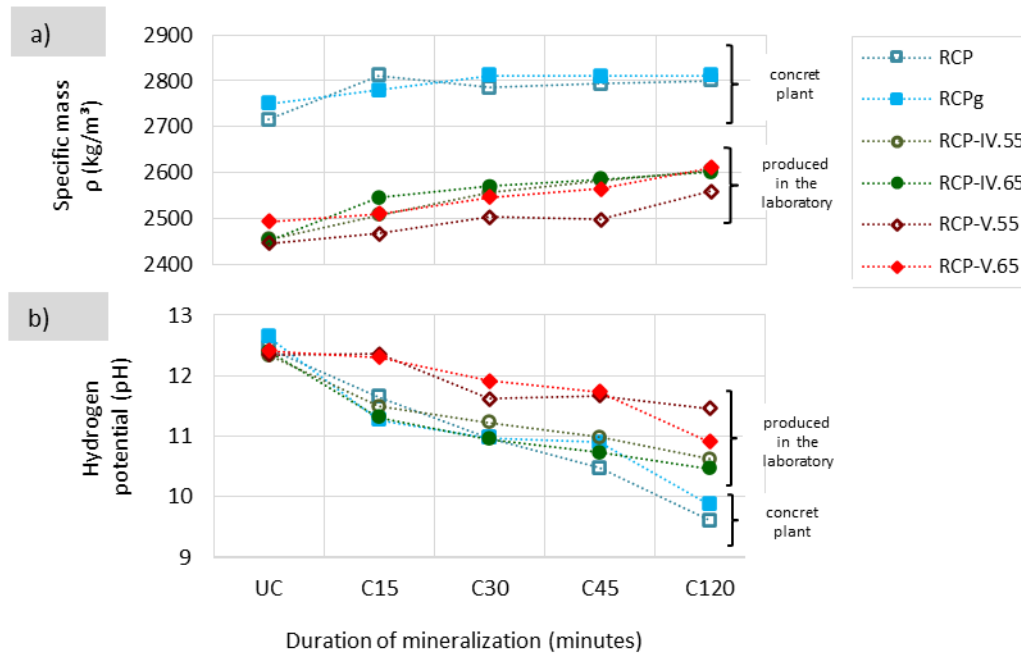
3.1 CHARACTERISTICS OF MINERALIZED RCP

3.1.1 Specific mass and pH

The accelerated carbonation mineralization caused changes in the specific gravity and pH of the recycled concrete powders, as observed in Figures 3a and 3b, respectively. For the specific gravity, the longer the process duration, the greater the increase compared to the uncarbonated (UC) powders, with values between 2714.80 to 2819.10 kg/m^3 for the RCP and RCP_g powders, which originated from the ready-mix concrete plant, and in a lower range for the other powders, with values between 2446.30 to 2610.90 kg/m^3 , from laboratory batching. Compared to the UC samples, the most significant increase was 6.1%, observed for RCP-IV.65 (C120), while the smallest values were recorded for RCP and RCP_g, with increases of 3.15% and 2.25%, respectively (C120). It is observed that the values are inverse to the f_{c28} of the source concretes of the powders (Table 1), and the reduction in particle size (RCP_g) also influenced this property. As reported in the literature, the carbonation of $\text{Ca}(\text{OH})_2$ implies a relatively larger solid volume [90–92], which may justify the increase in this property.

The potential hydrogen (pH) values of all recycled powders decreased due to the consumption of hydroxide ions (OH^-), which are responsible for alkalinity, during the carbonation reaction; however, all values remained ≥ 9 (Figure 3b). The decrease in pH to values between 8 and 9 reduces the alkaline protection that concrete offers to steel, making it susceptible to corrosion [93]. RCP and RCP_g showed the most considerable reductions for this characteristic, decreasing from 12.45 to 9.61 and from 12.45 to 9.88, respectively, after 120 minutes (C120). The smallest reduction was observed for RCP-V.55 (12.35 to 11.46), which can be attributed to the lower porosity of the source concrete, which influenced the diffusion of CO_2 and, consequently, the fixation of CO_2 [68,94]. The carbonation of portlandite generally maintains pH stability; however, the decalcification of C-S-H causes the most significant drop in pH, to values between 10 and 8.5. The subsequent decalcification of the decalcified C-S-H into a silica gel and calcium carbonate further reduces the pH [90,95].

Figure 3- Ph and Specific mass of mineralized RCP

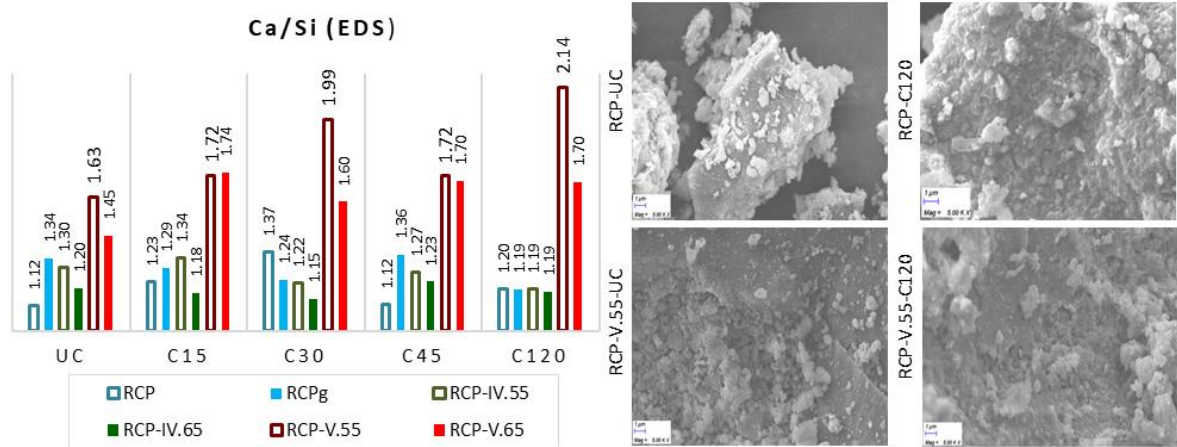


Given the mineralization of the recycled concrete powders, the specific gravity analysis indicated differences between the powders originating from the recycling plant and those produced in the laboratory. Despite these differences, both followed the same trend: mineralization increased specific gravity and reduced pH, indicating CO₂ fixation through CaCO₃ formation.

3.1.2 Ca/Si ratio and Morphology

Regardless of the powder's origin, the mineralization of recycled concrete powders resulted in changes in the Ca/Si ratio (Figure 4), with values ranging from 1.20 to 2.14 after 120 min of mineralization (C120). The RCP presented the highest value for this characteristic after 30 min of mineralization (1.37). In comparison, RCPg reached its maximum after 45 min (1.36), both showing a reduction thereafter, with values of 1.20 and 1.19, respectively, after 120 min (C120). The powders produced with CP IV cement exhibited behavior like those made at the recycling plant, with a reduction to 1.19 after 120 min of mineralization. On the other hand, the powders formulated with CP V cement did not show a decrease in the Ca/Si ratio throughout the process. Chen *et al.* [96] pointed out that a Ca/Si ratio of 1.2 marks the initial point at which calcium ions could be removed from the C-S-H interlayer during decalcification. When C-S-H is carbonated, its Ca/Si ratio decreases and it becomes quite porous, approaching amorphous silica [97], with a value close to 0.67 [98]. At the microscopic scale, morphological changes can be observed in the RCP and RCP-V.55 powders after 120 minutes of mineralization, with the precipitation of CaCO₃, resulting in rhombohedral, regular-shaped crystals, consistent with the findings of [46].

Figura 4-Ca/Si ratio (EDS) and powder morphology



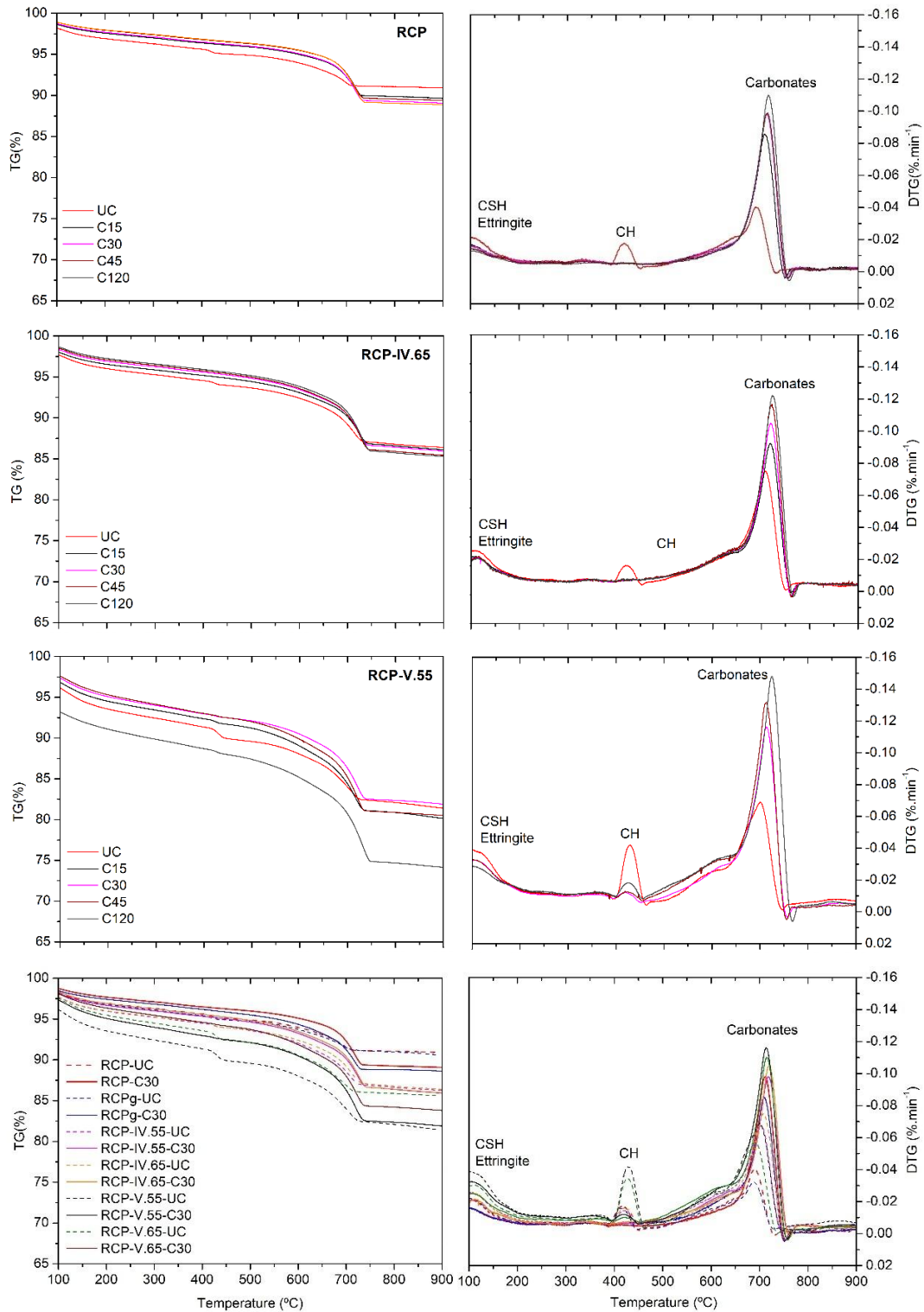
Given the times evaluated, it is recommended to consider other approaches, such as mechanochemical carbonation, as surface exposure during the mineralization process may promote the dissolution of calcium ions and favor the formation of amorphous silica [99,100]. Alternatively, it is also necessary to validate the performance of other types of powders with lower silica content, given that in this process, except for the powders produced with CP V (less porous), the Ca/Si ratio of 1.2 indicated the beginning of decalcification, but not the polymerization of C-S-H.

3.2 CO₂ FIXATION IN RECYCLED CONCRETE POWDERS

The results of the TG/DTG analysis for the powders mineralized by accelerated carbonation are shown in Figure 5. As illustrated, between 100 and 300 °C, the mass loss was due to the dehydration of hydration products (C-S-H gel and ettringite). At the same time, from 400 to 500 °C, the decomposition of calcium hydroxide (Ca(OH)₂ or CH) occurred. The determination of CO₂ fixation is based on the decarbonation of calcium carbonate (CaCO₃), which occurs between 550 and 800 °C, corroborating the literature findings [46,78].

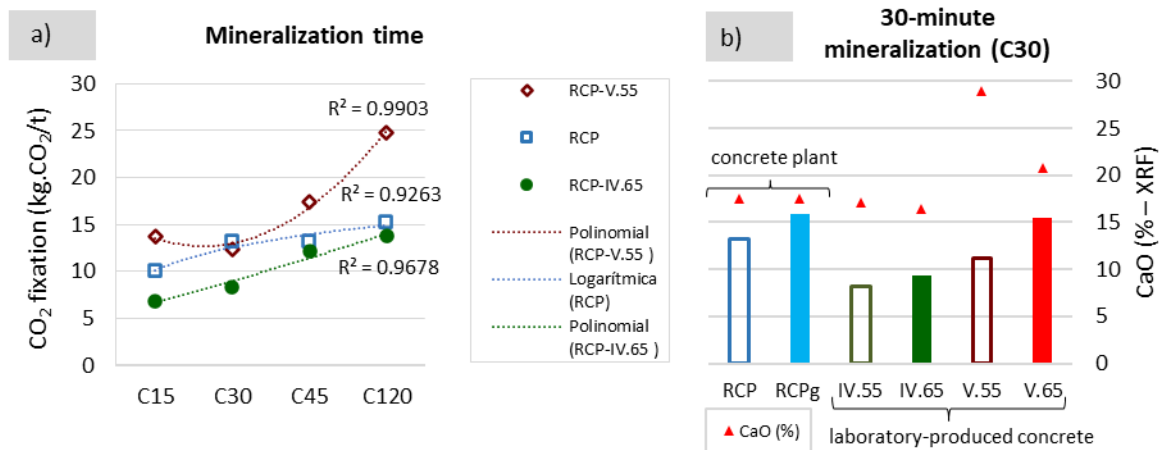
Based on TG/DTG curve analyses, carbon fixation values were quantified for the RCP powders (obtained from the recycling plant) and for RCP-IV.65 and RCP-V.55 (produced in the laboratory), which respectively have the lowest and highest CaO content (16.41% and 28.99%). The powders, mineralized for 30 minutes and used as SCM in partial substitution for cement, were also evaluated, as shown in Figure 6. It was observed that mineralization between 15 and 120 minutes fixed 7-25 kg-CO₂/t (Figure 6a), varying according to the origin of the recycled concrete powder. The RCP showed a logarithmic correlation with mineralization time, indicative of a rapid reaction between the powder's alkaline reserve and CO₂, with a tendency to stabilize after 30 minutes. In contrast, the other powders showed a second-order polynomial correlation, suggesting that they still have the potential to fix carbon without evidence of stabilization, meaning that times longer than 2 hours can still be explored, given the availability of Ca(OH)₂, as also visualized in the DTG curves (Figure 5). These results can be justified by the natural carbonation to which the concretes originating from the RCP were subjected before recycling [102], added to the carbonation that may have occurred during the collection, crushing, or handling processes of the powders [69], resulting in a smaller alkaline reserve than the powders from concretes produced in the laboratory.

Figure 5- TG and DTG curves of un-carbonated (UC) and carbonated (C)



The observed CO₂ fixation values are lower than those reported in the literature for concrete slurry waste (110 to 203 kg·CO₂/t) and cement kiln dust (86.8 to 116.8 kg·CO₂/t), both with CaO values higher than 30% [101] and similar to what was found for recycled concrete powders after 168 hours of mineralization, ~ 30 kg·CO₂/t [50].

Figura 6- CO₂ fixation in recycled concrete powders: (a) as a function of mineralization time; (b) powders mineralized for 30 minutes



Analyzing the powders mineralized for 30 minutes (Figure 6b), for the same chemical composition, the RCPg with a smaller particle size had a higher CO₂ fixation than RCP, and was like the RCP produced in the laboratory, RCP-V.65, with a value of about 16 kg·CO₂/t. In a gas-solid process, Risson *et al.* [63], reported lower water content in the powders and obtained values close to 22 kg·CO₂/t after 7 days. The reduction in particle size and the consequent increase in exposed surface area during mineralization favor the dissolution of CO₂ and the availability of CO₃²⁻ ions. With a greater quantity of available ions, the chemical reaction is accelerated, promoting the formation of calcium carbonate [67,102].

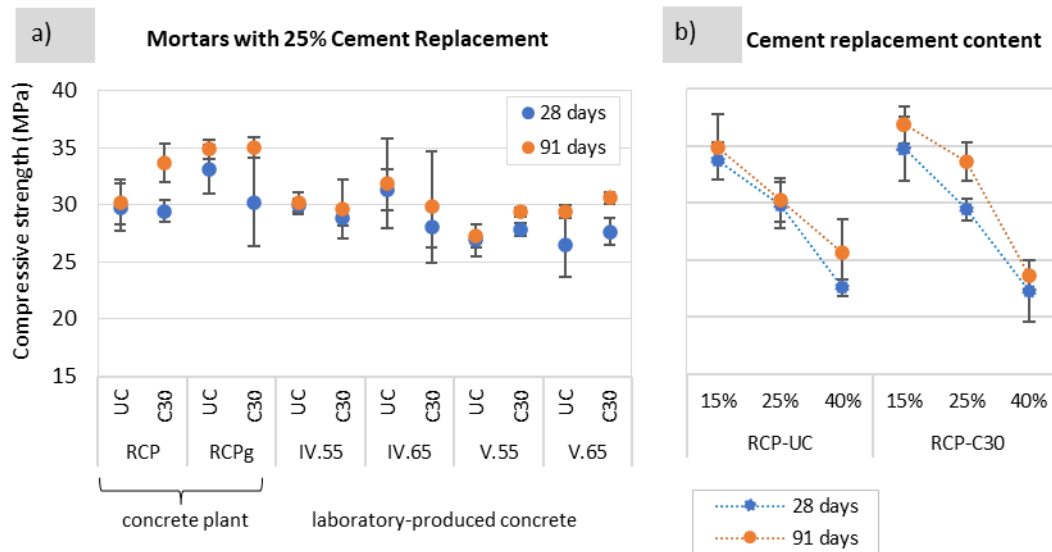
Regarding the influence of the water/cement ratio and the type of cement of the laboratory-produced concretes that originated the recycled concrete powders, it is observed that the powders originating from concretes with a lower w/c ratio (lower porosity), as pointed out by Cassel [53], showed lower CO₂ fixation. The reduced porosity hinders the diffusion of CO₂, limiting the extent of the carbonation reaction [103,104]. Regarding the type of cement, it is observed that the powders originating from concretes produced with CP V ARI (higher CaO) showed a tendency for greater CO₂ fixation compared to those with CP IV (lower CaO), a result that can be attributed to the presence of a greater amount of SCM in the composition of CP IV, reducing the alkaline reserve for the CaCO₃ reaction, as pointed out by [105]. Li and Wu (2022) [101] when analyzing various industrial wastes, verified that the efficiency of the mineralization process is more dependent on particle size, the microstructure of the materials (e.g., porosity), and the mineralization conditions than on the CaO content itself, corroborating the findings of this work, in which the powders showed the highest CO₂ fixation in the following descending order: RCPg>RCP-V.65>RCP>RCP-V.55>RCP-IV.65>RCP-IV.55.

Regarding cementitious materials, Sereng *et al.* [106], for recycled concrete aggregates in two particle size ranges (1–4 mm and 10–20 mm) and different origins, verified CO₂ fixation between 10 and 50 kg·CO₂/t after 8 days of the gas-solid process. The values varied with factors such as previous natural carbonation, temperature, aggregate size, and moisture content, with moisture content being the most influential. Cassel [53], in turn, investigated the gas-solid mineralization process for 24 hours using recycled aggregates from the same concretes that originated the powders in this work, with a particle size range of 10–20 mm. The results indicated a fixation between 1.84 and 12.81 kg·CO₂/t, with the most porous aggregate showing the highest fixation, in this case, the one originating from concrete IV.65. In the literature, aqueous carbonation processes are designed to overcome limitations of the chemical kinetics of the reactions and diffusion, which influence the speed and efficiency, especially in gas-solid processes [107,108].

3.3 COMPRESSIVE STRENGTH OF MORTARS

Figure 7a presents the compressive strength results of the mortars containing recycled concrete powders, uncarbonated (UC) and carbonated for 30 minutes (C30). Compressive strengths between 25 and 35 MPa are observed (Figure 7a), with a reduction in particle size (RCP_g) favoring the mechanical property, yielding values above 32 MPa for the UC powders at 28 days. The increase in surface area favors hydration reactions when used in sizes smaller than that of cement and in proportions less than 30%, corroborating the findings of other studies [29,109,110]. Considering the requirements of NBR 16697 [54], the mineralized powders (C30) are classified in class 25 (25 MPa) for contents up to 25% of Portland cement substitution.

Figure 7- Compressive strength of mortars: a) 25% cement replacement; b) Different cement replacement rates



In this study, the impact of CO₂ mineralization on compressive strength was less evident than usually reported with the use of hydrated cement paste powders, especially at 28 days. However, in 91 days, strength gains were observed with 25% cement substitution by the carbonated powders (C30), reaching up to 11% for RCP and 8% for RCP-V.55, compared to the mortars with uncarbonated powders. For substitution contents of 30% and 35%, gains of approximately 12.8% [48] and 12% [111], respectively. For a lower content (20%), Kaliyavaradhan, Li and Ling [50] reported an 8% gain using carbonated recycled concrete powders. The improvement in performance is attributed to the nucleation sites provided by CaCO₃ and Si-gel, which contribute to the accelerated hydration of cement [22] as well as to the filler effect, which refines the pores of the cementitious matrix [112]. Furthermore, the CaCO₃ with tricalcium aluminate (C₃A) in the cement increases the calcium carboaluminate content, which can also contribute to increased strength [113,114]. The later strength gain (at 91 days) indicates slower hydration kinetics at early ages, as also verified in the literature for recycled concrete powders [23,29].

Analyzing the influence of particle size distribution (RCP, RCP_g), the effect of CO₂ on the particles (UC and C30), and the curing age (28 and 91 days) (Table S2 – supplementary material), it was observed that both particle size and curing age significantly affected the compressive strength of mortars with 25% Portland cement replacement. Grinding increased the specific surface area of the particles (RCP_g), making them closer to that of Portland cement and thereby enhancing the reactivity in the mixtures when compared to unground RCP [110,115]. Furthermore, mortars produced with recycled concrete waste tend to exhibit higher increases in compressive strength at later ages, since under normal curing conditions, the reactions of the blended cement occur more slowly than those of Portland cement [116,117]. Thus, the combination of increased surface area and finer particle size of the recycled powders enhances hydration reactions, contributing to strength development over time, even for powders containing CO₂.

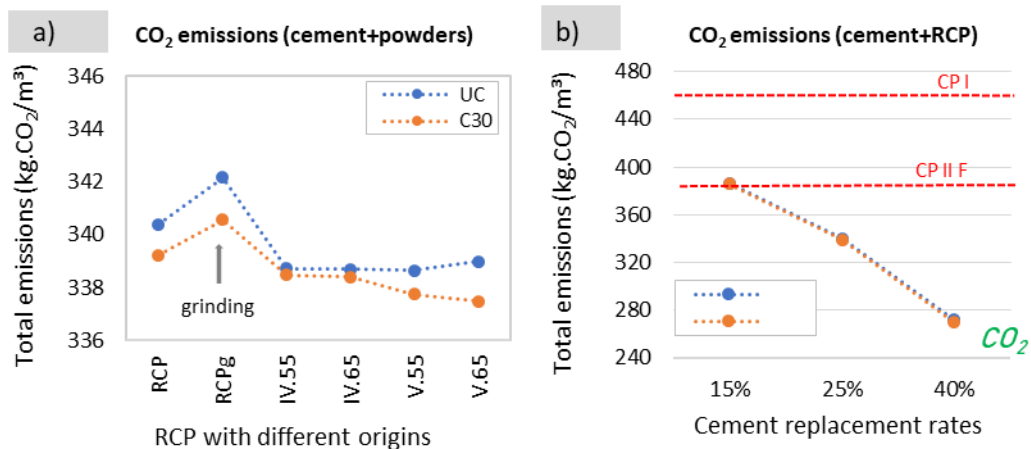
Analyzing the influence of the cement type and the w/c ratio of the concrete from which the recycled powders originated (Table S3 – supplementary material) on the compressive strength of the mortars, it was observed that the cement type of the original concrete significantly affected the mortar strength (p -value < 0.05). Recycled powders derived from concrete containing CP IV, due to the presence of pozzolanic material in its composition, tend to produce more durable products in the long term compared to those derived from CP V, which may explain the observed differences in compressive strength when used as SCM in mortars [106,116].

Regarding the cement substitution contents, it is observed that the UC and C30 powders showed similar behavior, with a reduction in compressive strength compared to CP I, whose values at 28 and 91 days were 38.50 MPa and 43.80 MPa, respectively, as a function of the increase in the substitution content, reaching about 23% and 42% at 28 days for 25% and 40% substitution, respectively. Substitution contents of up to 20% of the cement maintain the porosity of the cementitious matrix constant by pore refinement. In comparison, higher contents (30%-45%) had adverse effects [115–117] corroborating the findings of this work.

3.4 ENVIRONMENTAL INDICATORS

The use of mineralized powders reduces CO₂ emissions, with the reduction becoming more significant as the cement substitution content increases (Figure 8). For the evaluated powders, after mineralization, the total CO₂ emissions (cement + powders) varied between 337 and 340 kg·CO₂/m³ of mortar (25% substitution), values lower than CP II F (388.34 kg·CO₂/m³) and CP I (455.96 kg·CO₂/m³). The environmental benefits can be even more significant when considering the reductions associated with the extraction of raw materials, and when the costs of disposing of the powders in landfills are taken into account [85,118]. It is observed that CO₂ emissions are strongly influenced by the electrical energy used in the grinding process [119]. However, the use of RCPg in the mortar resulted in higher emissions than with the other powders (342 kg·CO₂/m³); the smaller particle size favored CO₂ fixation (15.92 kg·CO₂/t).

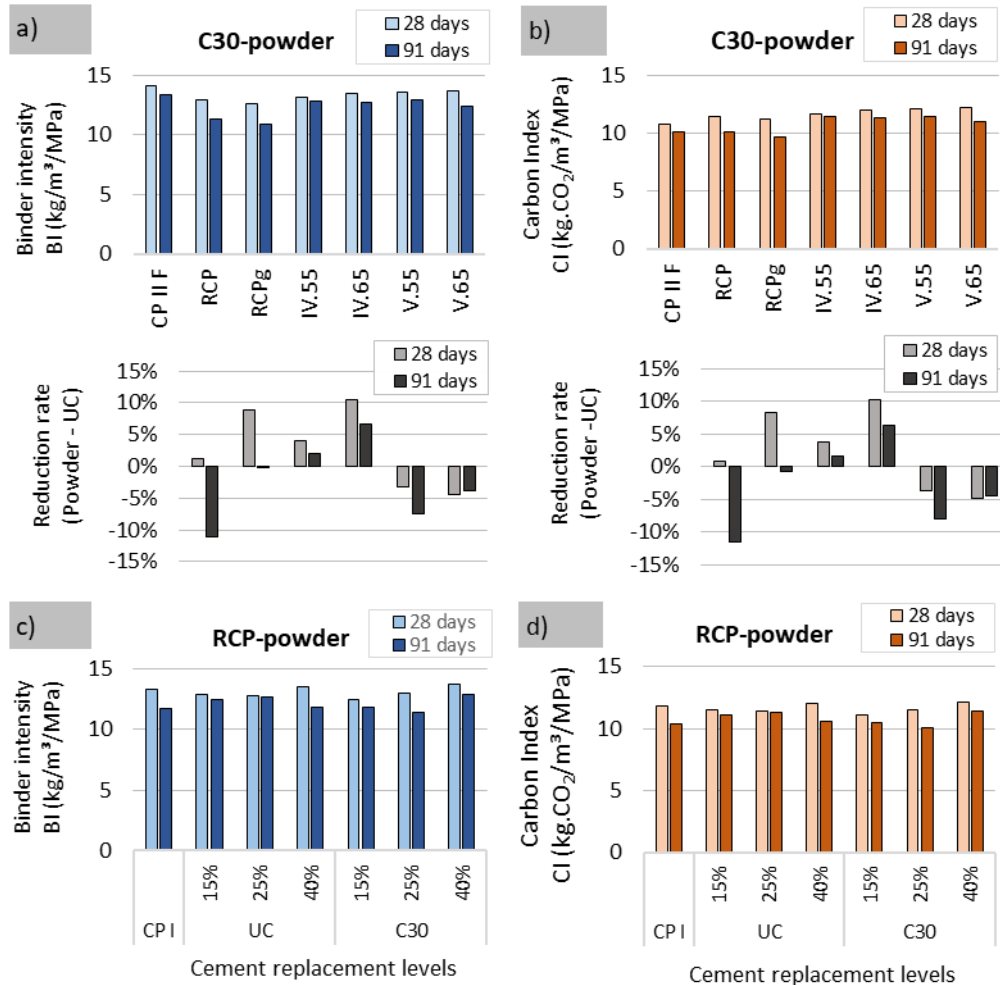
Figure 8-Total CO₂ emissions to produce 1m³ of mortar: a) RCP from different sources; b) Different levels of cement replacement by RCP



With the increase in the cement substitution content by the powders (Figure 8b), there was a significant reduction in CO₂ emissions. Compared to CP I, reductions in CO₂ emissions (kg·CO₂/m³) of 18%, 34%, and 68% were observed for the 15%, 25%, and 40% Portland cement substitution contents, respectively. CO₂ emissions were also lower than those of CP II F, which reinforces the relevance of CCUS technology associated with the use of supplementary cementitious materials as an effective decarbonization strategy. Although the mineralized powders showed reductions in CO₂ emissions to produce 1 m³ of mortar, these

reductions should not occur at the expense of the mortars' compressive strength. Thus, in Figure 9, the environmental indicators, the Binder Index (BI) and Carbon Index (CI), are shown.

Figure 9- Environmental indicators for mortars: a) Binder Intensity (BI) and Carbon Index (CI)



It was verified that the powders presented values between 12.65 and 13.75 at 28 days and 10.92 and 12.83 kg/m³/MPa at 91 days, except for RCP-IV.55; the other powders presented values lower than CP II F (14.17 and 13.33 kg/m³/MPa, respectively), indicating better environmental performance. The best results were obtained with the RCP and RCP_g powders, with reductions relative to CP II F of 1.2 and 1.5 kg/m³/MPa, respectively. Although the values obtained in this study are lower than those reported by [105], with a reduction from 8 to 5 kg/m³/MPa in recycled powders of hydrated pastes composed of different SCMs (ground granulated blast furnace slag, fly ash, and recycled waste glass powder) [105], it is essential to highlight that the results of this study were obtained from a material already exposed to the natural environment, with a higher silica content compared to powders originating from laboratory-hydrated cement pastes. For the CI indicator, values between 11.52 and 12.21 were observed at 28 days, and 9.73 to 11.49 kg·CO₂/m³/MPa at 91 days, with 25% cement substitution using different recycled concrete powders. Analyzing the origins of the powders, it is observed that the powders from the recycling plant concretes (RCP and RCP_g) presented Binder Indices lower than CP II F 32 at 91 days.

Based on the compressive strength at 91 days, the best results were obtained with RCP, with reductions of 11.08% in BI and 11.60% in CI compared to untreated (UC) powders. Next, RCP-V.55 stands out, which presented reductions of 7.53% in BI and 7.95% in CI. Longer exposure times for recycled powders from

laboratory-batched concretes should be analyzed, given the potential for improvements in environmental indicators observed after 30 minutes of mineralization. For the cement substitution contents (Figure 9c and 9d), the mortars containing 15% and 25% substitution showed a reduction in the BI and CI indicators compared to CP I at 28 days, and this reduction was further improved with the use of carbonated powders (C30) at 91 days.

The findings of this work provide additional information on the behavior of recycled concrete powders of different origins, mineralized via a gas-solid process. Optimizing the mortar mix, considering particle characteristics (e.g., particle packing and water-reducing admixtures), can be used to ensure compliance with the C32 strength class (32 MPa at 28 days), without necessarily reducing the cement substitution content.

The main limitation of this study continues to be the absence of available data in the literature on CO₂ fixation in recycled concrete powders, rather than in powders originating from hydrated cement pastes or other cementitious materials, as well as the lack of detailed information on the preparation of the materials, especially regarding the pre-hydration of the particles. Variables such as agglomeration, water homogenization within particles, and pre-hydration time, among others, are still little explored. Although mineralization was tested on a laboratory scale, the results presented in this study contributed to advancing industrial-scale application, integrating with the cement industry's decarbonization agenda through the adoption of CCUS technologies and, in parallel, advancing the circular economy.

4 CONCLUSIONS

Aiming to reduce CO₂ emissions associated with cement use and to achieve total recycling of cementitious construction and demolition waste, this study evaluated the mineralization of recycled concrete powders with different physicochemical characteristics and used them as a supplementary cementitious material at different substitution levels. The conclusions of the study are:

- The density of the recycled concrete powders increased with mineralization time due to the formation of carbonates, especially calcite. Exposure for 200 minutes resulted in average density increments of 2.7% for powders from uncontrolled-origin concretes and 5.5% for those produced in the laboratory (controlled origin), with different w/c ratios (0.55 and 0.65) and cement types (CP IV and CP V ARI). This effect is attributed to the higher porosity of the latter and the absence of prior natural carbonation in the controlled-origin powders.
- Mineralization affected the pH, Ca/Si ratios, and morphology of the recycled concrete powders. For materials derived from ready-mixed concretes, a pH reduction to values below 10 was observed, while the others maintained values above 10 under the same conditions. Except for powders produced with CP V ARI cement, C–S–H decalcification was verified after 120 minutes, indicated by Ca/Si ratios close to 1.2, although without the formation of silica gel. Morphologically, the presence of CaCO₃ crystals confirmed CO₂ fixation in the particles.
- Based on thermogravimetric analyses, recycled powders pre-hydrated with 12% moisture and mineralized at 60 °C for up to 120 minutes fixed between 7 and 25 kg CO₂/t. These values were mainly influenced by particle size distribution and the chemical composition of the parent concretes, with lower fixation observed for powders produced with CP IV cement and lower porosity. Mineralization time also affected carbon fixation, although the relationship was not linear. CO₂ fixation was higher during the initial exposure periods, showing a tendency to decrease over time until the exhaustion of carbonatable compounds or the loss of CO₂ permeability.
- The impact of CO₂ mineralization on the compressive strength of mortars was less evident at 28 days; however, at 91 days, strength gains of 11% for RCP and 8% for RCP-V.55 were observed compared with their respective non-carbonated powders. The combination of increased surface area and higher fineness of the recycled powders enhances hydration reactions, contributing to strength development over time, even for CO₂-containing powders.

- For a CO₂ mineralization time of 30 minutes, CO₂ fixation enabled the production of mortars with compressive strength equivalent to that of mortars produced with non-carbonated powders at 28 and 91 days, when 25% of Portland cement mass was replaced. Moreover, particle size distribution and the type of parent cement were identified as key factors influencing both CO₂ fixation capacity and the performance of the powders when used as SCMs.
- RCP mineralized for 30 minutes exhibited the best environmental indicators compared with CP I and CP II F-32 cements at replacement levels of 15% and 25%. This powder also showed a logarithmic correlation with mineralization time, suggesting a rapid reaction between its alkaline reserve and CO₂, with a tendency toward stabilization after 30 minutes. In contrast, the other powders exhibited a second-order polynomial correlation, indicating that they still have potential for CO₂ fixation even after 120 minutes.

Considering the challenges inherent in the decarbonization of the cement industry and the wide availability of construction and demolition waste, the potential for CO₂ fixation in the particles observed in this work, combined with the use of these materials as supplementary cementitious additions (SCM), positions recycled powders as potential generators of carbon credits on a large scale. Strategic choices in configuring the mineralization system, such as particle size, mineralization method, and transport distance, must be carefully evaluated to assess the economic viability of the process. Investments from the private sector, as well as continuous advances in public and private research and development (R&D), are fundamental for the consolidation and expansion of these technologies.

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SUPPLEMENTARY MATERIAL

Table S1 presents the oxide compositions of the cements used in the production of the laboratory concretes that originated the RCP-IV.55, RCP-IV.65, RCP-V.55, and RCP-V.65 powders.

Table S1- Oxide composition of cements (wt%) obtained by XRF

Materials	Chemical composition (%)											
	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	SO ₃	TiO ₂	Na ₂ O	BaO	P ₂ O ₅	LOI
CP IV	36.16	35.81	10.17	3.59	4.39	1.33	4.21	0.54	0.15	0.13	0.06	3.40
CP V	63.18	18.43	3.53	2.76	2.80	0.86	4.23	0.24	<LQ	0.17	0.12	3.60

LOI: Loss on Ignition; <LQ: below the quantifiable limit.

Source: Cassel (2024).

Table S2 presents the ANOVA parameters for the compressive strength of mortars, considering first the powder granulometry (RCP, RCPg), followed by the type of powder, taking into account CO₂ mineralization (UC, C30), and the age at the compressive strength test of mortars containing 25% replacement of Portland cement with the powders.

Tabela S2- ANOVA parameters for compressive strength of mortars with 25% replacement of Portland cement with powders of uncontrolled source

Mortars Produced with 25% Cement Replacement by Recycled Powders of uncontrolled source						
Variables	Sum of squares	df	Mean Square	F-value	p-value	Remarks
Particle size distribution (RCP, RCPg)	50.8536	1	50.8536	12.5510	0.0017	significant
Powder (UC or C30)	0.0435	1	0.0435	0.0110	0.9183	non-significant
Age (28 or 91 days)	62.8881	1	62.8881	15.5220	0.0060	significant
Particle size distribution*Powder	16.8781	1	16.8781	4.1660	0.0524	non-significant
Particle size distribution*Age	1.8818	1	1.8818	0.4640	0.5021	non-significant
Powder*Age	22.6464	1	22.6464	5.5890	0.0265	significant
Particle size distribution*Powder*Age	0.2775	1	0.2775	0.0680	0.7958	non-significant
error	97.2391	25	4.0516			

df: degrees of freedom

Table S3 presents the ANOVA analysis parameters for the compressive strength of the mortars, considering all recycled concrete powders used as a replacement for Portland cement (from controlled and uncontrolled origins).

Tabela S3-ANOVA parameters for the compressive strength of mortars produced with recycled concrete powders used as a replacement for Portland cement (from controlled and uncontrolled source)

Mortars Produced with 25% Cement Replacement by Recycled Powders						
Variables	Sum of squares	df	Mean Square	F-value	p-value	Remarks
Source (RCP, RCPg, RCP-IV.55, RCP-IV.65, RCP-V.55, RCP-V.65)	294.3822	5	58.8764	81.8860	0.0000	significant
Powder (UC or C30)	0.2400	1	0.2481	0.3450	0.5588	non-significant
Age (28 or 91 days)	85.6959	1	82.6959	115.0140	0.0000	significant
Source*Powder	265.3822	5	58.8764	81.8860	0.0000	significant
Source*Age	26.3969	5	5.2794	7.3430	0.0000	significant
Powder*Age	15.6332	1	15.6332	21.7430	0.0000	significant
Origin*Powder*Age	26.3969	5	5.2794	7.3430	0.0000	significant
error	51.7686	72	0.7190			
Influence of cement and water/cement ratio of original concrete						
Variables	Sum of squares	df	Mean Square	F-value	p-value	Remarks
Cement (IV or V)	43.7113	1	43.7113	15.0280	0.0006	significant
w/c ratio (0.55 or 0.65)	0.0010	1	0.0010	0.0000	0.9852	non-significant
Powder (UC or C30)	2.4961	1	2.4976	0.8590	0.3620	non-significant
error	81.4436	28	2.9087			
Mortars produced with different cement replacement levels using RCP powders (UC and C30)						
Variables	Sum of squares	df	Mean Square	F-value	p-value	Remarks
Powder (UC or C30)	4.6066	1	4.6066	1.0720	0.3073	non-significant
Replacement content (15%, 25%, 40%)	1082.3373	2	541.6866	125.9760	0.0000	significant
Age (28 or 91 days)	51.5638	1	51.5638	12.0030	0.0014	significant
Powder*Replacement content	19.0835	2	9.5417	2.2210	0.1231	non-significant
Powder*Age	2.7889	1	2.7889	0.6490	0.4257	non-significant
Replacement content *Age	1.0412	2	0.5206	0.1210	0.8862	non-significant
Powder*Replacement content*Age	15.1015	2	7.5508	1.7580	0.1869	non-significant
error	154.6486	36	4.2958			

df: degrees of freedom

4 CONCLUSÕES DA TESE

Frente aos desafios ambientais associados à geração de resíduos de construção e demolição (RCD) e às emissões de CO₂ da indústria do cimento, este trabalho buscou investigar soluções práticas integrando a mineralização de CO₂ em pós reciclados de RCD com diferentes origens, composições químicas e granulometrias, e o uso como material cimentício suplementar (MCS) ao cimento Portland.

Em relação à preparação (cominuição) e caracterização das partículas (absorção de água) para uso como MCS atendendo ao objetivo específico "i", verificou-se:

- com 30 minutos de moagem e o uso de 0,5% de propilenoglicol foi possível reduzir o diâmetro médio das partículas (d_{50}) e aumentar a área superficial- BET, sem alterar a cristalinidade do pó reciclado de concreto (RCP).
- a redução do tamanho das partículas para diâmetros inferiores a 0,15 mm resultou em aumento da demanda de água, o que comprometeu a trabalhabilidade e a resistência à compressão das argamassas avaliadas conforme a NBR 7215, quando o material foi utilizado como material cimentício suplementar (MCS).
- a TGA mostrou ser uma ferramenta eficiente para avaliação da absorção de água em pós reciclados de RCD com menor variabilidade nos resultados de um modo geral, uma vez que a determinação da condição superfície saturada seca é realizada de forma gráfica após a realização do ensaio, sem envolver manipulação da amostra.

Em relação ao uso de CO₂ em pós de RCD, atendendo aos objetivos específicos "ii", "iii" e "iv", os resultados experimentais indicaram:

- o potencial de fixação de CO₂ em pós reciclados de concreto é superior aos de origem mista, com maior fixação em partículas menores e mais porosas.
 - pós de concreto mineralizados a 20 °C por 2 h a 168 h, fixaram entre 14,7 e 24,1 kg. CO₂/t enquanto pós mistos no máximo 5,4 kg. CO₂/t (pré-hidratação por 24 horas com 6% de umidade).

- pós processados no moinho planetário por 30 minutos (RCPg), mineralizados a 60°C por 0,5 h, fixaram 15,92 kg.CO₂/t, valor superior ao RCP nas mesmas condições, 13,2 kg.CO₂/t (pré-hidratação por 30 minutos com 12% de umidade).
 - pós reciclados de concretos produzidos com CP V (maior teor de CaO) e relação a/c de 0,65 (mais porosos), mineralizados a 60 °C por 0,5 h de exposição ao CO₂, fixaram 15,50 kg.CO₂/t, valor superior ao obtido com o CP IV e a/c de 0,55, que fixaram 8,17 kg.CO₂/t.
- Em relação às condições de mineralização de CO₂, os resultados experimentais indicaram:
 - a faixa ótima de umidade para a fixação de CO₂ situa-se entre 6% e 18%, à temperatura de 60 °C, com fixação entre 10,15 até 13,30 kg.CO₂/t em 0,5 h de exposição ao CO₂.
 - quanto maior o tempo de exposição, maior foi a fixação de CO₂, com valores de 29,47 kg.CO₂/t nos RCP mineralizados por 24 horas com 12% de umidade à 40 °C. No entanto, a correlação entre essas variáveis depende da composição química do pó. Por exemplo, o RCP apresentou uma correlação logarítmica com tendência à estabilização em 2 horas, enquanto o RCP (IV.65 e V.55) apresentaram correlação polinomial.

Em relação ao uso dos pós com CO₂ mineralizados como MCS, atendendo o objetivo específico “v”, verificou-se:

- a mineralização de CO₂ promoveu melhoria das propriedades físicas dos pós como o aumento entre 2 a 3% da massa específica, áreas superficiais entre 3,1 a 6 m²/g chegando a valores próximos a 14 m²/g com a exposição por 24 horas ao CO₂ (12% de umidade à 40 °C).
- para os pós reciclados de concreto, a mineralização de CO₂, resultou em alterações na relação Ca/Si de 0,97 a 1,57 e redução do pH para valores acima de 9. No entanto, não foi observada a formação da sílica gel.
- sob as mesmas condições de mineralização (60 °C, 0,5 h, 12% de umidade) e substituição de 25% do cimento, foram observadas

variações na fixação de CO₂ (10,95 kgCO₂/t e 13,30 kgCO₂/t) e, consequentemente, na resistência à compressão aos 28 dias (32,05 MPa e 29,45 MPa). Isso indica que a heterogeneidade do pó, o preparo dos materiais ou a variação no controle de umidade na câmara de carbonatação ($\pm 10\%$) podem impactar diretamente a fixação de CO₂ e na performance do material.

- os pós mineralizados com CO₂ atuaram como fillers, resultando em aumento da resistência mecânica em idades avançadas (56 e 91 dias) quando comparados aos pós não carbonatados. A substituição de até 25% do cimento Portland por esses pós mineralizados assegurou indicadores ambientais inferiores aos do CP I, sob condições controladas de umidade (12%), temperatura (60 °C) e tempo de mineralização (0,5 h).

Considerando os desafios relacionados a heterogeneidades dos materiais e a pré-carbonatação pela exposição natural, e as diversas variáveis que envolvem a mineralização por carbonatação acelerada, os resultados apresentados nesta tese representam um avanço pioneiro no contexto brasileiro, ao demonstrar a viabilidade técnica da mineralização de CO₂ em pós reciclados de concreto e uso como material cimentício suplementar ao cimento Portland. No entanto, para que essa rota seja consolidada e ampliada, são necessários novos esforços em pesquisa aplicada, políticas de incentivo e investimentos industriais que promovam a integração desses materiais e processos ao setor produtivo, ampliando o alcance das soluções de baixo carbono no setor da construção.

4.1 SUGESTÕES PARA TRABALHOS FUTUROS

Com base nas observações realizadas no decorrer do desenvolvimento desta tese, foram elaboradas algumas sugestões para trabalhos futuros:

- (i) Analisar a influência do processo de reciclagem na fixação de CO₂ e relacionar com as possíveis rotas tecnológicas para potencializar o uso da tecnologia CCUS.
- (ii) Explorar os processos de carbonatação mecanoquímica e aquosa em pós reciclados de concreto;

- (iii) Analisar a durabilidade frente à penetração de íons cloreto e carbonatação de materiais cimentícios contendo os pós mineralizados;
- (iv) Ampliar as discussões a respeito do potencial de fixação de carbono dos pós reciclados de concreto no mercado de carbono.
- (v) Quantificar a contribuição para a circularidade na construção civil do uso do pó reciclado de concreto mineralizado.

4.2 PREMIAÇÕES E PUBLICAÇÕES ASSOCIADAS À TESE

4.2.1 Premiações

1. **UNIVERSIDADE FEDERAL DA INTEGRAÇÃO LATINO-AMERICANA (UNILA)**. Artigo premiado – Menção Honrosa ao trabalho “Influência do tempo de saturação do pó (75 μm) de concreto na captura de carbono”. Prêmio concedido durante o 4º *Seminário Internacional do Programa de Pós-Graduação Interdisciplinar em Energia e Sustentabilidade*, 2023, Foz do Iguaçu, PR.
2. **ASSOCIAÇÃO NACIONAL DE TECNOLOGIA DO AMBIENTE CONSTRUÍDO (ANTAC)**. Melhor artigo do ENARC 2023 ao trabalho intitulado: “Concretos permeáveis com agregados reciclados: avaliação da difusibilidade de CO_2 ”, Grupo de Trabalho de Resíduos. Prêmio concedido durante o 8º *Encontro Nacional de Aproveitamento de Resíduos na Construção – ENARC*, 2023, Foz do Iguaçu. Modalidade presencial, 30 out. – 1 nov. 2023.
3. **ANTAC - ASSOCIAÇÃO NACIONAL DE TECNOLOGIA DO AMBIENTE CONSTRUÍDO**. Equipe vencedora do 2º Desafio ENTAC no Eixo Processo da produção do ambiente construído com o trabalho intitulado: “Cimento Bioativado com Pó Reciclado de RCD com CO_2 Fixado | PCR BIO FIX, 2024”. Prêmio concedido durante o *Encontro Nacional de Tecnologia do Ambiente Construído (ENTAC)*, 2., 2024, Maceió, AL.
4. **ASSOCIAÇÃO NACIONAL DE TECNOLOGIA DO AMBIENTE CONSTRUÍDO (ANTAC)**. Prêmio Excelência em Ciência do Cimento – Yushiro Kihara (1º lugar) ao pôster intitulado: “Tratamento do pó de concreto com ácido tânico para aplicação como material cimentício suplementar”, Grupo de Trabalho de Cimentos. Prêmio

concedido durante o *Simpósio Brasileiro de Ciência do Cimento – SBCC*, 1., 2023, São Paulo, SP. Escola Politécnica da USP, 5–6 nov. 2023.

4.2.2 Publicações associadas à tese

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2. **RISSON**, K. D. B. S.; PROENÇA, M. P.; OLIVEIRA, D. R. B.; CASSEL, V. S.; DAL MOLIN, D. C. C.; ZARA, K. R. F.; POSSAN, E. Pós de resíduos da construção e demolição tratados com CO₂: potencial de uso como material cimentício suplementar. In: *Simpósio Brasileiro de Ciência do Cimento*, 2023. Anais [...]. São Paulo: ANTAC, 2023. v. 1, p. 1–5. Disponível em: <https://eventos.antac.org.br/index.php/sbcc/article/view/3691>. Acesso em: 23 abr. 2025.
3. SOARES, R. Y. E.; **RISSON**, A. V.; SANDOVAL, G. F. B.; POSSAN, E.; **RISSON**, K. D. B. S. Concretos permeáveis com agregados reciclados: avaliação da difusibilidade de CO₂. In: *Encontro Nacional de Aproveitamento de Resíduos na Construção*, 8., 2023, Foz do Iguaçu. Anais [...]. Foz do Iguaçu: ENARC, 2023. Disponível em: <https://doi.org/10.46421/enarc.v8i00.3005>. Acesso em: 23 abr. 2025.
4. CASSEL, V. S.; **RISSON**, K. D. B. S.; DAL MOLIN, D. C. C.; PUNHAGUI, K. R. G.; POSSAN, E. Potencial de captura de CO₂ de agregados graúdos reciclados de concreto no cenário nacional. *Concreto & Construção*, [S. l.], n. 116, p. 10–45, 2024. Disponível em: <https://concretoeconstrucoes.org.br/index.php/revista/edicao-116-artigo-002>. Acesso em: 23 abr. 2025.
5. PROENÇA, M. P.; OLIVEIRA, D. R. B.; **RISSON**, K. D. B. S.; POSSAN, E. CDW powder activated by mechanical, thermal and tannic acid treatment: an option for circularity in construction. *Waste and Biomass Valorization*, p. 1–24, 2024. DOI: 10.1007/s12649-024-02802-y.
6. OLIVEIRA, D. R. B.; LEITE, G.; PROENÇA, M. P.; **RISSON**, K. D. B. S.; POSSAN, E.; MARQUES FILHO, J. Concrete waste as a substitute for cement: evaluation of

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 8. PROENÇA, M.; **RISSON**, K. D. B. S.; OLIVEIRA, D.; POSSAN, E. Potencial pozolânico do resíduo misto de construção e demolição ativado termicamente: ensaios físico-mecânicos. In: *Congresso Sul-Americano de Resíduos Sólidos e Sustentabilidade*, 6., 2023, Foz do Iguaçu. Anais [...]. Foz do Iguaçu: CONRESOL, 2023. Disponível em: <http://dx.doi.org/10.55449/conresol.6.23.VII-012>. Acesso em: 23 abr. 2025.
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3. **RISSON**, K. D. B. S.; SANDOVAL, G. F. B.; PINTO, F. S. C.; CAMARGO, M.; MOURA, A. C.; TORALLES, B. M. *Molding procedure for pervious concrete specimens by density control*. *Case Studies in Construction Materials*, [S. l.], v. 15, e00619, dez. 2021. Disponível em: <https://doi.org/10.1016/j.cscm.2021.e00619>. Acesso em: 1 maio 2025.

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APÊNDICE A – RESULTADOS DA BUSCA NA BASE SCOPUS (2014-2024)

Portfólio	Classificação	Título	Referência
✓	Tratamento de partículas com carbono	Phase assemblance evolution during wet carbonation of recycled concrete fines	Shen <i>et al.</i> (2022)
X	Artigo de revisão	Roles of carbonated recycled fines and aggregates in hydration, microstructure and mechanical properties of concrete: A critical review	Zhang, T. <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	CO ₂ -fixing and recovery of high-purity vaterite CaCO ₃ from recycled concrete fines	Mehdizadeh; Mo; Ling (2023)
✓	Tratamento de partículas com carbono	Kinetics of enforced carbonation of cement paste	Zajac <i>et al.</i> (2020)
X	Artigo de revisão	Total recycling of concrete waste using accelerated carbonation: A review	Poon <i>et al.</i> (2023)
X	Artigo de revisão	Valorization of waste powders from cement-concrete life cycle: A pathway to circular future	Kaliyavaradhan; Ling; Mo (2020)
✓	Tratamento de partículas com carbono	Effects of carbonated hardened cement paste powder on hydration and microstructure of Portland cement	Lu <i>et al.</i> (2018)
✓	Tratamento de partículas com carbono	Enhancing the microstructure and surface texture of recycled concrete fine aggregate via magnesium-modified carbonation	Jiang <i>et al.</i> (2022)
X	Reforço de agregado miúdo	Synergistic enhancement effect of recycled fine powder (RFP) cement paste and carbonation on recycled aggregates performances and its mechanism	Bian <i>et al.</i> (2022)
✓	Tratamento de partículas com carbono	Quantifying the effects of wet carbonated recycled cement paste powder on the properties of cement paste	Mao <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Enhancing the treatment efficiency of recycled concrete fines with aqueous carbonation	Jiang, Y. <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Surface characterization of carbonated recycled concrete fines and its effect on the rheology, hydration and strength development of cement paste	Ouyang <i>et al.</i> (2020)
✓	Tratamento de partículas com carbono	A novel upcycling technique of recycled cement paste powder by a two-step carbonation process	Fang <i>et al.</i> (2021)
X	Cura carbônica	Preparation and carbonation hardening of low calcium CO ₂ sequestration materials from waste concrete powder and calcium carbide slag	Liu <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Mechanism underlying early hydration kinetics of carbonated recycled concrete fines-ordinary portland cement (CRCF-OPC) paste	Peng <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Mechanism of carbonating recycled concrete fines in aqueous environment: The particle size effect	Jiang <i>et al.</i> (2022)

Portfólio	Classificação	Título	Referência
X	Outros	Fabrication of negative carbon superhydrophobic self-cleaning concrete coating: High added-value utilization of recycled powders	Jiang, L. <i>et al.</i> (2023)
X	Cura carbônica	High-performance belite rich eco-cement synthesized from solid wastes: Raw feed design, sintering temperature optimization, and property analysis	Lyu <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Effect of wet carbonated recycled cement paste powder on the rheology of cement paste	Mao <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Effect of carbonation treatment on fracture behavior of low-carbon mortar with recycled sand and recycled powder	Tang <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Concrete improvement incorporating recycled powder and aggregates treated via a combination of calcination and carbonation: The impact behaviors	Feng <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Effect of conditions on wet carbonation products of recycled cement paste powder	Mao <i>et al.</i> (2023)
X	Reforço de agregado miúdo	Turning concrete waste powder into carbonated artificial aggregates	Shi <i>et al.</i> (2019)
X	Ensaio de Carbonatação	Carbonation of cement paste with GGBFS: Effect of curing duration, replacement level and CO ₂ concentration on the reaction products and CO ₂ buffer capacity	Vanoutrive <i>et al.</i> (2022)
✓	Tratamento de partículas com carbono	Triethanolamine-promoted separation of calcium from recycled concrete fines during aqueous carbonation	Teune; Schollbach (2024)
✓	Tratamento de partículas com carbono	Response surface methodology for the optimization of CO ₂ uptake using waste concrete powder	Kaliyavaradhan; Li; Ling (2022)
X	Ensaio de Carbonatação	Influence mechanism of Nano-SiO ₂ on geopolymer recycled concrete: Change mechanism of the microstructure and the anti-carbonation mechanism	Yang <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	High-temperature CO ₂ for accelerating the carbonation of recycled concrete fines	Wu, Y. <i>et al.</i> (2022)
✓	Tratamento de partículas com carbono	The influence of carbonation on the formation of calcium oxychloride	Ghantous <i>et al.</i> (2016)
✓	Tratamento de partículas com carbono	Effects of pretreated recycled powder substitution on mechanical properties and microstructures of alkali-activated cement	Zhang, B. <i>et al.</i> (2023)
X	Reforço de agregado miúdo	Re-cementation effects by carbonation and the pozzolanic reaction on LWAs produced by hydrated cement paste powder	Tang <i>et al.</i> (2022)
✓	Tratamento de partículas com carbono	Bonding mechanism of botanical concrete: Microstructural changes between waste concrete powder and wood	Wei <i>et al.</i> (2022)
X	Artigo de revisão	The utilization of recycled concrete powder as supplementary cementitious material in cement-based materials: A systematic literature review	Rocha; Toledo Filho (2023)

Portfólio	Classificação	Título	Referência
X	Ensaio de carbonatação	Pore structure and durability of green concrete containing recycled powder and recycled coarse aggregate	Wu, Yiwen <i>et al.</i> (2022)
X	Ensaio de carbonatação	Quality properties of self-consolidating concrete mixed with waste concrete powder	Kim (2017)
✓	Tratamento de partículas com carbono	Carbonation Behaviors of Recycled Concrete Fines Containing Supplementary Cementitious Materials: Compositions, Microstructures, and Mechanisms	Jiang <i>et al.</i> (2024)
X	Cura carbônica	Carbonation-cementation of recycled hardened cement paste powder	Zhu; Fang; Wei (2018)
✓	Tratamento de partículas com carbono	Properties and CO ₂ -curing enhancement of cement-based materials containing various sources of waste hardened cement paste powder	Wu <i>et al.</i> (2021)
X	Ensaio de carbonatação	Durability of waste concrete powder-based geopolymer reclaimed concrete under carbonization and freeze–thaw cycles	Yang <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Highly reactive carbonated recycled concrete fines prepared via mechanochemical carbonation: Influence on the early performance of cement composites	Zhao <i>et al.</i> (2024)
✓	Análise ambiental	Carbon emission analyses of concretes made with recycled materials considering CO ₂ uptake through carbonation absorption	Xiao <i>et al.</i> (2021)
✓	Tratamento de partículas com carbono	Production of sustainable lime-based brick using carbonated recycled concrete fines: Mechanical, mineralogical and microstructure properties	Ouyang <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Upcycling of waste hydrated cement paste containing high-volume supplementary cementitious materials via CO ₂ pre-treatment	Mehdizadeh <i>et al.</i> (2022)
X	Artigo de revisão	A systematic comparison of performance of recycled concrete fine aggregates with other alternative fine aggregates: An approach to find a sustainable alternative to river sand	Rifa <i>et al.</i> (2023)
✓	Análise ambiental	A comparative life cycle assessment of recycling waste concrete powder into CO ₂ -Capture products	Kravchenko <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Recycling and valorization of hydrated cement blends in mortars via semi-dry carbonation – The role of waste glass, granulated blast furnace slag and fly ash	Liu; Tang; Wang (2023)
✓	Tratamento de partículas com carbono	Corrosion mechanism of recycled mortar prepared from CO ₂ -treated hardened cement paste powder	Pan <i>et al.</i> (2023)
X	Ensaio de carbonatação	Sustainable utilization of hybrid recycled powder and recycled polyethylene terephthalate fiber in mortar: Strength, durability and microstructure	Hou <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Development of sustainable alkali-activated slag bricks with high content of carbonated recycled concrete fines: Strength, microstructure and environmental benefit	Ouyang <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Evaluation of carbonation conversion of recycled concrete fines using high-temperature CO ₂ : Reaction kinetics and statistical method for parameters optimization	Mehdizadeh <i>et al.</i> (2023)

Portfólio	Classificação	Título	Referência
✓	Análise ambiental	Value stream assessment of the sustainable concrete recycling process with sequestration of CO ₂ from flue gases	Kravchenko; Besklubova (2024)
✓	Tratamento de partículas com carbono	Effects of pretreated recycled fine aggregates on the mechanical properties and microstructure of alkali-activated mortar	Xie <i>et al.</i> (2024)
X	Cura carbônica	Production of artificial aggregates by granulation and carbonation of recycled concrete fines	Kursula <i>et al.</i> , (2022)
✓	Tratamento de partículas com carbono	Revisiting the carbonation of recycled concrete fine: A pH-cycle carbonation method	Qian <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Semi-dry carbonated recycled concrete paste as alternative to limestone and its reactivity in LC3	Jin <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Beneficiation of recycled concrete fines through accelerated carbonation	Gholizadeh-Vayghan; Snellings (2022)
X	Cura carbônica	Excellent carbonation behavior of rankinite prepared by calcining the C-S-H: Potential recycling of waste concrete powders for prefabricated building products	Wang; Ren; Yang (2018)
X	Ensaio de carbonatação	Carbonation Behavior of Mortar Made from Treated Recycled Aggregates: Influence of Diammonium Phosphate	Gómez-Cano <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Valorisation of Recycled Cement Paste: Feasibility of a Short-Duration Carbonation Process	Silva <i>et al.</i> (2022b)
✓	Tratamento de partículas com carbono	Parametric Study towards Optimization of a Short Duration Carbonation Process of Recycled Cement Paste	Silva <i>et al.</i> (2022a)
X	Cura carbônica	Carbonation of Recycled Concrete Aggregates for New Concrete and Concrete Fines to Make Cement-Free Hollow Blocks	Bergmans <i>et al.</i> (2024)
X	Outros	Boosting CO ₂ Uptake from Waste Concrete Powder Using Artificial Intelligence and the Marine Predators Algorithm	Rezk <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Wet-carbonation-based Mineral Extraction and CO ₂ Sequestration Using Concrete Waste Fines at a Low Temperature	Bui <i>et al.</i> (2023)
✓	Tratamento de partículas com carbono	Effect of the Pretreatment on the Properties of Cement-Based Recycled Powder	Li <i>et al.</i> (2024)
✓	Tratamento de partículas com carbono	Influences of Carbonated Recycled Concrete Fines on Cement Hydration	Zhang, J. <i>et al.</i> (2023)
✓**	Tratamento de partículas com carbono	Accelerated carbonation of regenerated binding material prepared with waste cement paste powder	Lu, L. <i>et al.</i> (2023)
X	Outros	Verification Method of Direct Air Capture by Cementitious Material Using Carbon Isotopes	Wang <i>et al.</i> (2023)

Portfólio	Classificação	Título	Referência
✓	Tratamento de partículas com carbono	Mechanical Property and Microstructure of Cement Mortar with Carbonated Recycled Powder	Ding <i>et al.</i> (2024)
X	Cura carbônica	Development of green cement from solid wastes	Zhang, S. <i>et al.</i> (2023)
X	Ensaio de carbonatação	Corrosion of rebars in recycled glass concrete under tropical marine environment	Hoil-Canul <i>et al.</i> (2022)
✓	Tratamento de partículas com carbono	Performance and Carbon Footprint Evaluation of Cement-Based Materials Incorporating Carbonated Recycled Fine Powder	Mo <i>et al.</i> (2023)
X	Artigo de revisão	Research Progress on Carbonation Technologies For Valorising Waste Concrete: A Review	Jiang, Y. <i>et al.</i> (2023)
✓**	Tratamento de partículas com carbono	Fundamental Problems and Applications of Recycled Fine Powder Derived from Waste Concrete	Xiao <i>et al.</i> (2023)
X	Cura carbônica	Micro-mechanical Characterization of Non-carbonation and Decalcification Carbonation Zones of Cement Paste	Zhang; Li; Wang (2023)
X	Outros	Effect of Carbonization Treatment on Behavior and Mechanism of Pb ²⁺ Adsorption by Recycled Concrete Fines	Wang, K. <i>et al.</i> (2024)
✓**	Tratamento de partículas com carbono	Influence law of physical and chemical properties of the recycled hardened cement powder by mineral carbonation with low concentration of CO ₂	Liu, Q. <i>et al.</i> (2022)

Legenda: ✓ SIM; X NÃO; ✓** SIM, mas não foram analisados (indisponíveis).

APÊNDICE B - PORTFÓLIO ANALISADO – REVISÃO SISTEMÁTICA DA LITERATURA

FI	Year	Ci	InOrdinatio	Título	Referência
19,3	2022	83	399	Evolução da montagem de fases durante a carbonatação úmida de finos de concreto reciclado	Shen <i>et al.</i> (2022)
17,9	2023	32	339	Fixação do CO ₂ e recuperação de vaterite CaCO ₃ de alta pureza de finos de concreto reciclado	Mehdizadeh; Mo; Ling (2023)
19,3	2020	117	335	Cinética da carbonatação forçada da pasta de cimento	Zajac <i>et al.</i> (2020)
10,6	2018	214	278	Efeitos do pó de pasta de cimento endurecido carbonatado na hidratação e microestrutura do cimento Portland	Lu <i>et al.</i> (2018)
19,3	2022	28	262	Melhorando a microestrutura e a textura da superfície de agregados finos de concreto reciclado por meio de carbonatação modificada com magnésio	Jiang <i>et al.</i> (2022)
19,3	2024	9	238	Quantificação dos efeitos do pó de pasta de cimento reciclado carbonatado húmido nas propriedades da pasta de cimento	Mao <i>et al.</i> (2024)
19,3	2023	8	233	Aumentando a eficiência do tratamento de finos de concreto reciclado com carbonatação aquosa	Jiang, Y. <i>et al.</i> (2023)
13,3	2020	81	230	Caracterização da superfície de finos de concreto reciclado carbonatado e seu efeito na reologia, hidratação e desenvolvimento da resistência da pasta de cimento	Ouyang <i>et al.</i> (2020)
15,8	2021	44	229	Uma nova técnica de reciclagem de pó de pasta de cimento reciclado através de um processo de carbonatação em duas fases	Fang <i>et al.</i> (2021)
13,3	2023	18	223	Mecanismo subjacente à cinética de hidratação precoce de pastas carbonatadas de concreto reciclado- pasta de cimento Portland normal (CRCF-OPC)	Peng <i>et al.</i> (2023)
13,3	2022	30	207	Mecanismo de carbonatação de finos de concreto reciclado em ambiente aquoso: o efeito do tamanho das partículas	Jiang <i>et al.</i> (2022)
19,3	2024	0	193	Efeito da pasta de cimento reciclado carbonatada úmida em pó na reologia da pasta cimentícia	Mao <i>et al.</i> (2024)
13,3	2023	12	193	Efeito do tratamento de carbonatação no comportamento à fratura de argamassa de baixo carbono com areia reciclada e pó reciclado	Tang <i>et al.</i> (2023)
15,8	2023	6	188	Melhoria do concreto incorporando pó reciclado e agregados tratados através de uma combinação de calcinação e carbonatação: Os comportamentos de impacto	Feng <i>et al.</i> (2023)
13,3	2023	11	188	Efeito de condições em produtos de carbonatação úmida de pó de pasta de cimento reciclado	Mao <i>et al.</i> (2023)
17,9	2024	0	179	Separação de cálcio de finos de concreto reciclado promovida pela trietanolamina durante a carbonatação aquosa	Teune; Schollbach (2024)

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10,6	2022	29	177	Metodologia de superfície de resposta para a otimização da absorção de CO ₂ utilizando resíduos de pó de concreto	Kaliyavaradhan; Li; Ling (2022)
6,4	2022	44	173	CO ₂ a alta temperatura para acelerar a carbonatação de finos de concreto reciclado	Wu, Y. <i>et al.</i> (2022)
13,3	2016	72	169	A influência da carbonatação na formação de oxiclureto de cálcio	Ghantous <i>et al.</i> (2016)
10,6	2023	12	166	Efeitos da substituição de pó reciclado pré-tratado nas propriedades mecânicas e microestruturas do cimento ativado com álcalis	Zhang, B. <i>et al.</i> (2023)
15,8	2022	1	159	Mecanismo de ligação do concreto botânico: alterações microestruturais entre resíduos de concreto em pó e madeira	Wei <i>et al.</i> (2022)
14,5	2024	0	145	Comportamento de Carbonatação de Finos de Concreto Reciclado com Materiais Cimentícios Complementares: Composições, Microestruturas e Mecanismos	Jiang <i>et al.</i> (2024)
6,4	2021	49	143	Propriedades e melhoria da cura com CO ₂ de materiais à base de cimento contendo várias fontes de pó de pasta de cimento endurecido de resíduos	Wu <i>et al.</i> (2021)
13,3	2024	0	133	Finos de concreto reciclado carbonatados altamente reativos preparados por carbonatação mecanoquímica: Influência no desempenho inicial dos compósitos de cimento	Zhao <i>et al.</i> (2024)
4,6	2021	48	123	Análises de emissão de carbono de concretos feitos com materiais reciclados levando em consideração a absorção de CO ₂ através da carbonatação	Xiao <i>et al.</i> (2021)
10,6	2024	3	121	Produção de tijolos sustentáveis à base de cal utilizando finos de concreto carbonatado: Propriedades mecânicas, mineralógicas e microestruturais	Ouyang <i>et al.</i> (2024)
6,4	2022	23	120	Reciclagem de resíduos de pasta de cimento hidratada contendo materiais cimentícios suplementares de grande volume através de pré-tratamento com CO ₂	Mehdizadeh <i>et al.</i> (2022)
11,4	2024	1	119	Uma avaliação comparativa do ciclo de vida da reciclagem de resíduos de pó de concreto em produtos de captura de CO ₂	Kravchenko <i>et al.</i> (2024)
10,6	2023	2	116	Reciclagem e valorização de misturas de cimento hidratado em argamassas por carbonatação semi-seca - O papel dos resíduos de vidro, das escórias granuladas de alto-forno e das cinzas volantes	Liu; Tang; Wang (2023)
10,6	2023	2	116	Mecanismo de corrosão de argamassa reciclada preparada a partir de pó de pasta de cimento endurecido tratado com CO ₂	Pan <i>et al.</i> (2023)
10,6	2024	0	106	Desenvolvimento de tijolos sustentáveis de escória ativada por álcalis com elevado teor de finos de concreto reciclado carbonatado: Resistência, microestrutura e benefícios ambientais	Ouyang <i>et al.</i> (2024)

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7,7	2023	4	97	Avaliação da conversão por carbonatação de finos de concreto reciclado utilizando CO ₂ a alta temperatura: Cinética de reação e método estatístico para otimização de parâmetros	Mehdizadeh <i>et al.</i> (2023)
8,1	2024	0	81	Avaliação do fluxo de valor do processo sustentável de reciclagem de concreto com sequestro de CO ₂ dos gases de combustão	Kravchenko; Besklubova (2024)
5,2	2024	5	77	Efeitos de agregados finos reciclados pré-tratados nas propriedades mecânicas e microestrutura de argamassas ativadas por álcalis	Xie <i>et al.</i> (2024)
6,4	2023	2	74	Reverso a carbonatação de finos de concreto reciclado: Um método de carbonatação com ciclo de pH	Qian <i>et al.</i> (2023)
7,4	2024	0	74	Pasta de concreto reciclado carbonatado semi-seco como alternativa ao calcário e sua reatividade em LC ³	Jin <i>et al.</i> (2024)
5,7	2022	7	73	Beneficiação de finos de concreto reciclado através de carbonatação acelerada	Gholizadeh-Vayghan; Snellings (2022)
4,7	2022	4	56	Valorização de pastas de cimento recicladas: Viabilidade de um processo de carbonatação de curta duração	Silva <i>et al.</i> (2022b)
4,7	2022	3	53	Estudo paramétrico para otimização de um processo de carbonatação de curta duração de pasta de cimento reciclada	Silva <i>et al.</i> (2022a)
2,9	2023	3	44	Extração de minerais por carbonatação húmida e sequestro de CO ₂ utilizando finos de resíduos de concreto a baixa temperatura	Bui <i>et al.</i> (2023)
3,9	2024	1	44	Efeito do pré-tratamento nas propriedades do pó reciclado à base de cimento	Li <i>et al.</i> (2024)
3,8	2023	1	43	Influência dos finos de concreto reciclado carbonatado na hidratação do cimento	Zhang, J. <i>et al.</i> (2023)
3,3	2023	0	33	Carbonatação acelerada de material ligante regenerado preparado com resíduos de pasta de cimento em pó	Lu, L. <i>et al.</i> (2023)
1,8	2024	0	18	Propriedades mecânicas e microestruturas de argamassas de cimento com pó reciclado carbonatado	Ding <i>et al.</i> (2024)
1,1	2023	0	11	Avaliação do desempenho e da pegada de carbono de materiais à base de cimento que incorporam pó fino reciclado carbonatado	Mo <i>et al.</i> (2023)
0,8	2023	0	8	Problemas fundamentais e aplicações do pó fino reciclado derivado de resíduos de concreto	Xiao <i>et al.</i> (2023)
0,1	2022	0	0	Lei de influência das propriedades físicas e químicas do pó de cimento endurecido reciclado por carbonatação mineral com baixa concentração de CO ₂	Liu, Q. <i>et al.</i> (2022)