UNIVERSIDADE FEDERAL DE LAVRAS

DOUGLAS LAMOUNIER FARIA

RESÍDUOS LIGNOCELULÓSICOS COMO ALTERNATIVA DE REFORÇO EM COMPÓSITOS CIMENTÍCIOS EXTRUDADOS

LAVRAS - MG
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Tese apresentada à Universidade Federal de Lavras, como parte das exigências do Programa de Pós-Graduação em Engenharia de Biomateriais, área de concentração Compósitos e Nanocompósitos Lignocelulósicos, para a obtenção do título de Doutor.

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LIGNOCELLULOSIC WASTE AS A REINFORCEMENT ALTERNATIVE IN EXTRUDED CEMENT COMPOSITES

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(São Tomás de Aquino)

RESUMO

Os produtos de origem cimentícia são caracterizados por seu comportamento quebradiço e frágil. Por esta razão, a utilização de materiais de reforço auxilia na absorção de energia pelo compósito, propiciando maior resistência aos esforços de tração e deformação. O amianto e as fibras poliméricas são comumente usados para modificar a natureza frágil dos materiais à base de cimento, mas não representam uma solução ecológica, e o amianto causa doenças pulmonares. Meios alternativos são evidenciados pelo aumento das pesquisas avaliando os benefícios do uso de fibras vegetais. Contudo, o desafio principal é a compatibilidade entre a fibra e a matriz, devido à natureza hidrofílica da fibra, e, também, devido ao ambiente alcalino da matriz cimentícia, que degrada a fibra vegetal. Uma alternativa para contornar esse desafio é a carbonatação acelerada, que age reduzindo a alcalinidade da matriz cimentícia, propiciando um ambiente favorável para melhor adesão entre fibra e matriz. Desta forma, esta pesquisa teve como objetivo avaliar as propriedades físicas, mecânicas e microestruturais de fibrocimentos extrudados e compostos com fibras residuais da cultura do sorgo ou da seringueira, como alternativa de reforço e o aproveitamento correto desses materiais de descarte. A pesquisa consistiu em duas propostas de uso de fibras naturais. Na primeira proposta, compósitos utilizando fibras de sorgo foram produzidos pelo processo de extrusão. Na segunda proposta, a fim de se reduzir a influência dos extrativos na hidratação do cimento, as fibras de seringueira passaram por tratamento alcalino com hidróxido de sódio (NaOH) (10% m/v), além disso, a influência da cura dos compósitos por carbonatação acelerada foi avaliada. Ambos os tipos de compósitos permaneceram em processo de cura em ambiente saturado por 2 dias, após esse período, os compósitos produzidos com fibras de seringueira passaram por carbonatação acelerada por um período de 12 h, depois permanecerem em cura úmida até completar o período de 28 dias. O emprego de fibras lignocelulósicas como reforço em matriz cimentícia se mostrou viável, pois apresentou comportamento satisfatório no que diz respeito às propriedades físicas e mecânicas dos fibrocimentos produzidos. De forma geral, o compósito cimentício com melhor desempenho mecânico foi alcançado pela substituição de cimento por fibras de sorgo na porcentagem de 1%. Nos compósitos produzidos com fibras de seringueira, a carbonatação acelerada ocasionou a redução das propriedades mecânicas, com exceção da tenacidade, repercutindo na melhoria da absorção da energia mecânica dos compósitos. Assim, a produção de compósitos cimentícios utilizando resíduos lignocelulósicos agrega valor a esses resíduos, diminuindo a poluição ambiental causada pela queima ou descarte inadequado, bem como emprego dos princípios da economia circular.

Palavras-chave: Fibrocimento. Fibras vegetais. Carbonatação acelerada. Propriedades físicas e mecânicas. Extrusão.

ABSTRACT

Cement-based materials are characterized by their brittle and brittle behavior. For this reason, the use of reinforcement materials helps in the absorption of energy by the composite, providing greater resistance to traction and deformation efforts. Asbestos and polymeric fibers are commonly used to modify the brittle nature of cement-based materials, but they do not represent an ecological solution, and asbestos causes lung disease. Alternative means are evidenced by the increase in research evaluating the benefits of using vegetable fibers. However, the main challenge is the compatibility between the fiber and the matrix, due to the hydrophilic nature of the fiber, and also due to the alkaline environment of the cement matrix, which degrades the vegetable fiber. An alternative to overcome this challenge is accelerated carbonation, which acts by reducing the alkalinity of the cement matrix, providing a favorable environment for better adhesion between fiber and matrix. Thus, this research aimed to evaluate the physical, mechanical and microstructural properties of extruded fiber-cements compounds with sorghum or rubberwood fibers, as an alternative for reinforcement and the correct use of these waste materials. The research consisted of two proposals for the use of natural fibers. In the first proposal, composites using sorghum fibers were produced by the extrusion process. In the second proposal, in order to reduce the influence of extractives on cement hydration, the rubberwood fibers underwent alkaline treatment with sodium hydroxide (NaOH) (10% m/v), in addition, the influence of curing composites by accelerated carbonation was evaluated. Both types of composites remained in the curing process in a saturated environment for 2 days, after this period, the composites produced with rubberwood fibers underwent accelerated carbonation for a period of 12 h, then remained in wet curing until completing the period of 28 days. The use of lignocellulosic fibers as reinforcement in cement matrix proved to be viable, as it presented satisfactory behavior with regard to the physical and mechanical properties of the fiber-cements produced. In general, the cementitious composite with the best mechanical performance was achieved by replacing cement with sorghum fibers in the percentage of 1%. In the composites produced with rubberwood fibers, the accelerated carbonation caused the reduction of the mechanical properties, with the exception of the tenacity, resulting in the improvement of the mechanical energy absorption of the composites. Thus, the production of cement composites using lignocellulosic waste adds value to these wastes, reducing environmental pollution caused by burning or improper disposal, as well as employing the principles of the circular economy.

Keywords: Fiber-cement. Vegetable fibers. Accelerated carbonation. Physical and mechanical properties. Extrusion.

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1 INTRODUÇÃO

O emprego de materiais sustentáveis na fabricação de elementos construtivos é uma possibilidade para reduzir o impacto ambiental gerado pela construção civil, que contém indicadores expressivos referentes ao consumo de recursos não renováveis, gasto energético e geração de resíduos sólidos (PEREIRA et al., 2013; ONUAGULUCHI; BANTHIA, 2016).

Atualmente, os compostos reforçados de fibras ou partículas naturais são considerados entre os materiais estruturais mais promissores em tecnologias de engenharia (BELAADI et al., 2013). As fibras/partículas vegetais existem em grande quantidade, e são utilizadas por apresentarem um custo reduzido, caráter renovável e boa disponibilidade. Na maioria das vezes como resíduos e sem uma adequada destinação, as fibras/partículas se tornam um problema ambiental. Por essa razão, estudos estão sendo desenvolvidos em busca de soluções que sejam economicamente viáveis, sustentáveis, e eficientes no que diz respeito às aplicações tecnológicas (SILVA et al., 2015).

O compósito cimentício denominado fibrocimento é um material que vem sendo estudado por diversos pesquisadores. A utilização deste tipo de compósito em forma de componentes pré-fabricados, telhas, e placas divisórias, pode contribuir de maneira significativa para o rápido crescimento da infraestrutura do país (SAVASTANO JUNIOR et al., 2009; SHIRAKAWA et al., 2022).

A matriz cimentícia é geralmente reforçada com fibras sintéticas como carbono, vidro, polipropileno ou aramida. Apesar de suas vantagens, o custo elevado desses materiais, o alto consumo de energia durante a produção, e o seu impacto ambiental negativo, despertaram o interesse de pesquisadores no estudo de obtenção de reforços em matrizes cimentícias a partir da utilização das fibras/partículas vegetais, que são amplamente disponíveis no meio ambiente, são biodegradáveis, não abrasivas, não há preocupação com a saúde e segurança durante seu manuseio e obtidas por processos de baixo custo (MERTA; TSCHEGG, 2013; TESSARO et al., 2015).

Há grande interesse em estudar o uso de fibras vegetais como reforço em matriz cimentícia, pelo fato destas apresentarem altas propriedades mecânicas, como resistência à tração (200 MPa) e módulo de elasticidade (4 GPa) (SANJAY et al., 2018), além de reduzir a dependência de materiais convencionais para fabricação do concreto, mas também o impacto no meio ambiente (SAWSEN, 2015; BUI et al., 2021). As fibras/partículas naturais melhoram o comportamento de tração, resistência à flexão e dureza e ductilidade dos materiais à base de cimento. O aumento da resistência à fratura é causado pela própria fibra e pelo vínculo entre a fibra e a matriz (WEI et al., 2016).

Contudo, um desafio na utilização de fibras vegetais em fibrocimento se deve à sua instabilidade dimensional, em virtude de seu caráter hidrofílico e, também, da degradação alcalina da fibra proporcionada pela matriz cimentícia. A degradação de materiais cimentícios modificados pela adição de fibras vegetais ocorre principalmente em virtude da elevada alcalinidade da água presente nos poros da matriz do cimento Portland (com pH superior a 13) (RODRIGUES et al., 2013). Como consequências desses fatores, ocorre uma perda da aderência na interface fibra-cimento e aumento de micro e macrofissuras, o que contribui para a diminuição da resistência e da durabilidade do compósito de fibrocimento com fibras celulósicas (TONOLI et al., 2013).

A cura sob carbonatação acelerada de materiais cimentícios é uma possível solução a fim de mitigar a mineralização da fibra vegetal e a consequente redução da durabilidade do compósito. Este método acelerado consiste em criar um ambiente em que a concentração de CO_2 seja consideravelmente superior à concentração ao ambiente natural com condições experimentais selecionadas, ou seja, condições ambientais. Estas inclui a concentração de CO_2, temperatura, umidade relativa e a duração da exposição, que desempenham um papel crucial no processo de carbonatação e têm efeitos significativos na carbonatação de materiais compósitos cimentícios (BUI et al., 2021).

Dessa forma, a presente pesquisa tem por hipótese que a adição/substituição de fibras vegetais de sorgo ou seringueira, em diferentes teores, não resultem diferenças significativas no desempenho físico e mecânico dos compósitos cimentícios produzidos por extrusão em relação aos compósitos produzidos sem fibras.

2 OBJETIVOS

2.1 Objetivo geral

Avaliar os efeitos de fibras lignocelulósicas provenientes dos resíduos das culturas do sorgo e da seringueira nas propriedades físicas, mecânicas e microestruturais de fibrocimentos extrudados.

2.2 Objetivos específicos

- Avaliar a influência das fibras de sorgo e de seringueira na inibição da hidratação do cimento.
- Avaliar o teor ideal de fibras de sorgo em compósitos cimentícios produzidos por extrusão.
- Avaliar o efeito da cura sob carbonatação acelerada em compósitos de fibrocimento reforçados com fibras de seringueira.
- Avaliar o efeito do tratamento superficial com hidróxido de sódio (NaOH) das fibras de seringueira na adesão com a matriz cimentícia.

3 REFERENCIAL TEÓRICO

3.1 Fibrocimento

O fibrocimento é constituído basicamente por diferentes porcentagens de cimento Portland, calcário, fibras vegetais ou sintéticas. O cimento Portland é o componente principal, tem a função aglomerante, podendo representar valores superiores a 80% da massa dos materiais secos que formam o compósito. O calcário é usado como substituição parcial do cimento Portland, com o objetivo de reduzir custos de produção do fibrocimento, sendo considerado um produto de enchimento, representando a segunda maior porcentagem que forma o compósito em questão.

As matrizes cimentícias são compostas de aglomerantes minerais, dando origem a pastas ou, quando elaboradas contendo agregados dão origem as argamassas ou concretos. As matrizes mais utilizadas são à base de cimento Portland e, em menor escala, aquelas à base da cal e do gesso. A adição de fibras nas matrizes pode melhorar as suas propriedades mecânicas, como a resistência à tração, à flexão e ao impacto. Além disso, altera seu comportamento após fissuração diminuindo os efeitos de uma ruptura brusca da matriz cimentícia (TONOLI et al., 2012).

O fibrocimento é o material de construção civil mais utilizado nos sistemas de cobertura das edificações brasileiras. Considerando o mercado total de coberturas, o fibrocimento é aplicado em 49% das edificações, seguido pelas telhas cerâmicas (35%) e de aço (11%) (SILVA; ETULAIN, 2010).

O fibrocimento é um compósito cimentício, ou seja, material à base de cimento Portland, composto por duas fases: matriz cimentícia e fibras (Figura 1). A matriz cimentícia é uma pasta composta por cimento e adições minerais de escória, pozolânicas e/ou calcíticas, sem agregados. Para aumentar a resistência mecânica final e a tenacidade do compósito, e para viabilizar o processo produtivo, o fibrocimento possui fibras distribuídas discretamente pela matriz, que normalmente são de celulose, amianto ou fibras sintéticas de poliacetato de vinila (PVA), polipropileno (PP) ou poliacrilonitrila (PAN).

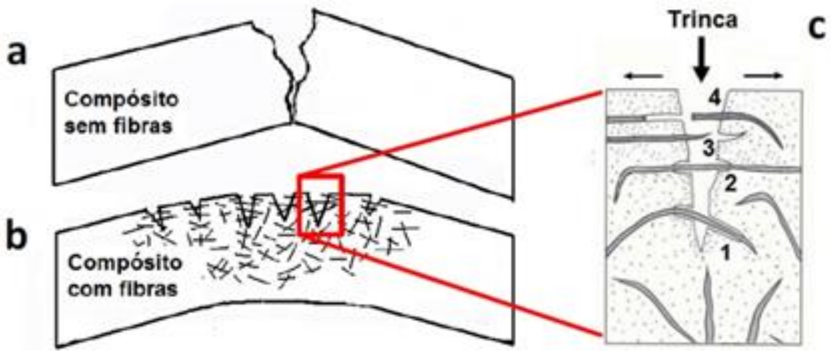
Figura 1 - Materiais que compõem o fibrocimento.

Fonte: Do autor (2022).

As matrizes cimentícias apresentam comportamentos frágeis, quebradiças e formam fissuras quando estão submetidas a esforços, ou seja, fraturam-se sem deformação plástica sob esforços de tração e cargas dinâmicas (Figura 2a) (TONOLI et al., 2010).

As fibras possibilitam o surgimento de mecanismos de tenacificação que, por sua vez, promovem comportamento mecânico pseudoplástico do compósito. Ou seja, as fibras aumentam a resistência mecânica e a capacidade do compósito absorver a energia, com a distribuição de microfissuras ao longo do material (Figura 2b).

Figura 2 - Representação esquemática do comportamento à flexão de um compósito: a) sem fibras, e b) reforçado com fibras; c) detalhe do caminhamento da trinca através do compósito reforçado com fibras: (ponto 1) e (ponto 2) *bridging* e descolamento da fibra; (ponto 3) arrancamento (*pull-out*) da fibra; (ponto 4) rompimento da fibra.



Fonte: Adaptado de Coutts (1986).

A incorporação de fibras de reforço é a principal particularidade do fibrocimento, que o torna um compósito cimentício diferenciado, em comparação com os demais materiais a base de cimento Portland. O amianto foi o material mais utilizado como reforço na produção de fibrocimento, no entanto, devido aos diversos problemas de saúde provocados nos trabalhadores do setor, essa fibra natural foi substituída por outras fibras alternativas, (AGOPYAN et al., 2005). O amianto é uma fibra natural mineral sedosa, com propriedades físico-químicas com grande potencial para ser utilizada como reforço mecânico em fibrocimento, como: alta resistência mecânica, durabilidade, flexibilidade e resistência ao ataque de ácido e álcalis. Apesar destas ótimas propriedades, o amianto apresenta inúmeros malefícios para a saúde humana, como asbestose, que pode levar à morte (BARAN et al., 2016); desta forma, teve sua extração e comércio proibidos no Brasil e em diversos outros países. As fibras vegetais, como reforço das matrizes frágeis do fibrocimento, têm despertado grande interesse dos produtores, especialmente devido seu baixo custo, grande disponibilidade, economia de energia na produção, etc.

A produção de fibrocimento sem amianto teve início no Brasil no ano de 2001, com a importação de fio de PVA. Entretanto, o alto custo da fibra de PVA e a demora com a importação, contribuíram para que a indústria acelerasse o projeto de desenvolvimento de sua própria fibra, dessa forma, no ano de 2003 iniciou-se a produção da fibra de polipropileno (DIAS et al., 2010).

O fibrocimento, aplicado no nosso país em inúmeras coberturas, é um material que contém fibras de amianto (10% a 20%) fortemente aglutinadas por cimento (PROENÇA et al., 2014).

Faruk et al. (2012) descreveram que nos últimos anos a produção de artigos e pesquisa referente a compósitos utilizando fibras vegetais aumentou consideravelmente. Entretanto, compósitos reforçados com fibras vegetais ainda estão na dependência do conhecimento de alguns fatores importantes, relacionando a sua aplicação, desempenho e durabilidade. É importante considerar que as fibras vegetais têm composição química diferente e depende do tipo de planta, da dimensão da célula cristalina, do ângulo helicoidal que a celulose faz em relação ao eixo central, defeitos superficiais, estrutura da macrofibra vegetal, propriedades físicas e mecânicas das fibras e a interação que a fibra pode fazer com a matriz do compósito.

Esse fato tem incentivado o desenvolvimento de vários produtos com essas fibras, em matrizes poliméricas ou cimentícias, que apresentam aumento da resistência mecânica, conforto térmico e tenacidade.

O uso de fibras vegetais apresenta algumas desvantagens, sendo as principais a baixa durabilidade e a mineralização das fibras. Muitas tentativas de produção de argamassas ou pastas de cimento Portland comum reforçadas com fibras vegetais fracassaram devido ao fato de os compósitos apresentarem vida útil entre 2 e 4 anos (AGOPYAN, 1991). Segundo o mesmo autor, uma das principais razões para ocorrer essa rápida degradação é a elevada alcalinidade da água presente nos poros da matriz de cimento *Portland* com pH próximo de 13, o que leva à mineralização das fibras.

O aumento da durabilidade das fibras vegetais como reforço em cimento pode ser abordado por diferentes tecnologias, por exemplo, estudo com impregnação das fibras com agentes repelentes à água (SOUZA et al., 2017), tratamentos superficiais para selar os poros dos compósitos (GRAM, 1983), redução da alcalinidade da matriz com uso de adições ou tipos alternativos de aglomerantes (AGOPYAN, 1991).

A baixa durabilidade das fibras na matriz cimentícia ocasiona a redução do desempenho do compósito de fibrocimento, pois além de ocorrer a degradação das fibras com perda de propriedades, há perda de aderência entre fibra e matriz, e, consequente redução de propriedades mecânicas do compósito.

3.2 Carbonatação acelerada do cimento

A carbonatação é um fenômeno natural que afeta os materiais cimentícios. Tal fenômeno consiste na reação dos produtos da hidratação do cimento com o dióxido de carbono (CO_2). Com o passar do tempo, o cimento absorve o CO_2 da atmosfera e esse processo ocorre, durante a vida útil dos produtos e, também, após a demolição (FILOMENO et al., 2020). A carbonatação fixa o CO_2 e altera as propriedades físico-químicas das matérias-primas. A utilização da carbonatação acelerada para mitigar a degradação das fibras vegetais, utilizada como reforço em compósitos de fibrocimento, é algo que necessita de mais estudos na área da construção civil. Isso porque a carbonatação acelerada é efetiva em condições ambientais específicas, como teor de CO_2, temperatura, umidade relativa e período de exposição.

O processo de carbonatação é complexo e envolve a difusão do CO_2 atmosférico através dos poros insaturados presentes na matriz cimentícia (LO et al., 2016; MARTINS et al., 2018). Quando o dióxido de carbono (CO_2) reage com o hidróxido de cálcio ($Ca(OH)_2$), forma-se o carbonato de cálcio ($CaCO_3$), reação predominante no processo.

A carbonatação acelerada se deve à reação de dissolução do CO_2 em água, em que, é transformado em ácido carbônico (H_2CO_3) nos poros insaturados da matriz de cimento, ocorrendo a dissociação de íons HCO_3^- e CO_3^- (Equação 1 e Equação 2). Concomitantemente, há dissociação do $Ca(OH)_2$ em íons Ca^{2+} e OH no meio (Equação 3), que finalmente forma o $CaCO_3$ (Equação 4).

CO_2 (g) + H_2O (l) ➔ H^+ (aq) + HCO_3 (aq) (1)

$Ca(OH)_2$ (aq) ➔ Ca^{2+} (aq) + CO_3^{2-} (aq) (2)

$Ca(OH)_2$ (aq) ➔ Ca^{2+} (aq) + $2HO^-$ (aq) (3)

Ca^{2+} (aq) + CO_2^{3-} (aq) ➔ $CaCO_3$ (s) (4)

A cura acelerada de materiais cimentícios em ambiente rico em dióxido de carbono (CO_2) surge como uma promessa frente à degradação destes materiais. A carbonatação acelerada mitiga a degradação das fibras vegetais utilizadas como reforço em fibrocimentos e demais materiais à base de cimento, uma vez que o pH da matriz é reduzido para um valor próximo de 8,3 nas zonas totalmente carbonatadas (SAETTA et al., 1993; FILOMENO et al., 2020). Essa redução na alcalinidade torna o cimento menos agressivo às fibras e melhora a interface do compósito (TOLEDO FILHO et al., 2003; DOS SANTOS et al., 2019). O processo de cura de materiais cimentícios sob carbonatação ocorre pela difusão do CO_2 através dos poros insaturados da matriz cimentícia (LO et al., 2016; MARTINS et al., 2018). O CO_2 reage com o hidróxido de cálcio ($Ca(OH)_2$) (Equações 1 e 2) formando carbonato de cálcio ($CaCO_3$) (Equações 3 e 4). Nos poros insaturados do cimento, o CO_2 é dissolvido em água e transformado em gás carbônico ácido (H_2CO_3), havendo dissociação de íons HCO_3^- e CO_3^-. Ao mesmo tempo há dissociação do $Ca(OH)_2$ nos íons Ca^{2+} e OH^-, formando $CaCO_3$ (FILOMENO et al., 2020). O processo de carbonatação acelerada acontece naturalmente, porém em uma taxa lenta; contudo, o processo pode ser acelerado utilizando uma câmara de carbonatação, com temperatura, umidade relativa, concentração de CO_2 e tempo de exposição padronizados.

Diversos autores observaram resultados satisfatórios utilizando a carbonatação acelerada como método de cura em compósitos cimentícios reforçados com fibras vegetais, sendo observada redução da alcalinidade da matriz cimentícia, melhor adesão fibra-matriz e, consequentemente, melhoria das propriedades mecânicas (ALMEIDA et al., 2013; PIZZOL et al., 2014; SANTOS et al., 2015; URREA-CEFERINO et al., 2017; NEVES JUNIOR et al., 2019).

3.3 Sorgo

O Brasil possui lugar de destaque frente à produção agrícola mundial, e, consequentemente, muitos resíduos de biomassa são gerados em diversas etapas da cadeia produtiva (desde a colheita até o processamento dos grãos). A maior parte destes resíduos não possui utilização adequada, representado um grave problema ambiental, uma vez que são descartados em locais e em condições inapropriadas. Dessa forma, o emprego dos resíduos deste setor como material de reforço em matriz cimentícia pode ser considerado uma excelente alternativa, no que diz respeito à destinação adequada desses resíduos, e, também, como substituição de materiais de alto custo utilizados na produção de fibrocimento. A utilização de fibras/partículas vegetais na produção de fibrocimento vem crescendo cada dia mais, uma vez que a inserção dessas fibras disponíveis não agridem o meio ambiente, reduz o custo da produção e proporcionam ao material excelentes propriedades mecânicas e bom desempenho.

O sorgo é considerado o quinto cereal de maior importância no mundo, sendo base alimentar de mais de 500 milhões de pessoas, em mais de 30 países (MACE et al., 2013). No Brasil é destinado à produção de ração animal, e seu cultivo vem crescendo tanto em área plantada quanto em produtividade. Foram 2,084 milhões de toneladas de grãos em 864,6 mil hectares de área plantada na safra de 2020/2021, com produtividade de 2,41 t/ha. Para a safra de 2021/2022, a área plantada foi estimada em 950,2 mil hectares, com uma produção de 3,041 milhões de toneladas e uma produtividade de 3,20 t/ha. A região de maior produtividade é a Centro-Sul, que possui 83,20 % (1,734 milhões de toneladas) da colheita nacional (Companhia Nacional de Abastecimento - CONAB, 2022).

O sorgo apresenta pouca necessidade de água, possuindo maior tolerância ao déficit de umidade no solo, por essa razão, pode ser cultivado em uma vasta faixa de condições de solo (VASCONCELLOS, 2001).

A incorporação das partículas de sorgo em compósitos possibilita uma nova forma de aproveitamento destes resíduos (CHEN et al., 2014; ABDEL‑MAGID et al., 2021; AIDA et al., 2022), agregando valor a um produto de descarte e possibilitando a geração de um novo produto no mercado.

3.4 Seringueira

A seringueira é natural da Amazônia, e existem dez espécies no Brasil, das onze conhecidas. Botanicamente, a seringueira é uma dicotiledônea dos gêneros *Hevea*, pertencente à família Euphorbiaceae, sendo todas elas espécies arbóreas e arborícolas (LIMA et al., 2000). Possui a *Hevea brasiliensis* (Willd. ex Adr. de Juss.) Muell.-Arg. como a espécie mais importante do gênero (GONÇALVES et al., 2002). A área plantada com seringueira cresceu consideravelmente nos últimos anos no Brasil. Em 2005 a área plantada foi de 112.396 ha e passou para 163.254 ha em 2020 (Instituto Brasileiro de Geografia e Estatística - IBGE, 2020). A expansão do plantio de seringueira no país provoca um suprimento significativo da madeira dessa cultura ao final de sua rotação (25-30 anos), o que leva ao interesse em estudos sobre essa matéria-prima. No Brasil, a madeira de *Hevea* obtida ao final do ciclo produtivo do látex é utilizada na maioria das vezes e, tradicionalmente, para uso como lenha apesar de apresentar boas características de trabalhabilidade (colagem, cravação, perfuração, entre outras) e pode ser facilmente curvada com o uso de vapor e facilmente tingida (EUFRADE JUNIOR et al., 2015).

Embora seja uma planta de origem amazônica, a parte do Brasil onde hoje mais crescem seringueiras não é mais a Amazônia, mas em áreas formadas pelo noroeste do Estado de São Paulo, o oeste do Triângulo Mineiro e o nordeste do Mato Grosso do Sul, região que alia ótimas condições climáticas, alta densidade demográfica e um grande mercado consumidor. Esta inversão que parece desafiar a noção de “vocação natural” tem causas agronômicas e econômicas (SOMAIN; DROULERS, 2016).

Hevea brasiliensis é uma planta de ciclo perene, de origem tropical, cultivada e utilizada de modo extrativo, com a finalidade de produção de borracha natural (CAMPELO JÚNIOR, 2000). A partir da saída de seu habitat passou a ser cultivada em grandes monocultivos, principalmente nos países asiáticos.

Hoje, o Estado de São Paulo é o maior produtor nacional de borracha natural. De acordo com os dados da Produção Agrícola Municipal (PAM) publicados pelo IBGE, em 2020, para uma produção nacional de borracha de 376.036 toneladas, São Paulo contribuiu com 249.393 t, ou seja, 66%, seguido pelos Estados de Minas Gerais com 28.013 t (7%); Goiás com 25.968 t (7%); Bahia com 22.872 t (6%), de Mato Grosso com 14.365 t, (4%), e, Espírito Santo com 13.744 t (4%) (IBGE, 2020).

Devido à necessária supressão das árvores para renovação do plantio de seringueira, diversos pesquisadores utilizaram a madeira de seringueira como matéria-prima em produtos de maior valor agregado, como em paineis aglomerados (IWAKIRI et al., 2018; FARIA et al., 2021) e paineis de lâminas paralelas (FARIA et al., 2019).

3.5 Tratamento superficial em fibras lignocelulósicas

As fibras naturais possuem algumas propriedades inerentes que podem servir como desvantagens em seu reforço de compósitos cimentícios, incluindo baixa resistência a ataques microbianos, baixa resistência à umidade, baixa adesão entre a superfície fibra-matriz e tendência a aglomerar durante o processamento. Essas desvantagens podem ser superadas pela modificação da superfície da fibra, que pode ser alcançada por métodos de tratamento químico, como mercerização, desparafinação, acetilação, enxerto químico, branqueamento, deslignificação e salinização e, também, métodos de tratamento físico, como descarga corona, radiação de ionização e plasma (HAYDARUZZAMAN et al., 2010; SAW et al., 2011; SUDHAKARA et al., 2013).

O tratamento químico de fibras é um processo de melhoria da adesão entre a superfície da fibra e a matriz cimentícia para uma adequada transferência de tensão (BETTINI et al., 2010), removendo as impurezas superficiais com consequente obtenção de uma superfície rugosa. Durante o tratamento químico é removido da superfície das fibras os extrativos, hemiceluloses e lignina, resultando em aparecimento de marcas globulares e células de parênquima.

O tratamento superficial por alcalinização utilizando solução de hidróxido de sódio (NaOH) é um dos tratamentos químicos mais usados para as diversas fibras vegetais (BELTRAMI et al., 2014). A modificação ocasionada por esse tratamento é o rompimento da ligação de hidrogênio no grupo hidroxila (OH) presente na estrutura da fibra, aumentando assim a rugosidade superficial (AMICO et al., 2005; SGHAIER et al., 2012).

4 CONSIDERAÇÕES FINAIS SOBRE A REVISÃO BIBLIOGRÁFICA

Diante do que foi apresentado nos tópicos anteriores, a reciclagem de resíduos provenientes da agroindústria é uma opção para mitigar os impactos ambientais causados pelo descarte incorreto destes materiais. A valorização de resíduos em produtos de maior valor agregado se mostra uma alternativa ecologicamente, socialmente e financeiramente viável, contribuindo para inserir as indústrias brasileiras no contexto da “economia circular” e “economia verde”. O fibrocimento é um material já estabilizado na indústria da construção civil. A produção de fibrocimentos utilizando resíduos da cultura do sorgo e de seringueira se mostra uma alternativa às fibras de amianto (atualmente sua utilização se encontra proibida) e às fibras poliméricas, as quais possui alto valor comparado às fibras vegetais.

Entretanto, a literatura carece de estudos envolvendo a compatibilidade das fibras vegetais com a matriz cimentícia, além de métodos e condições alternativas de cura dos materiais cimentícios.

Desta forma, este trabalho foi desenvolvido em dois artigos visando à caracterização física e química das fibras de sorgo e de seringueira, bem como a influência dessas fibras na matriz cimentícia, a fim de se entender o comportamento dos fibrocimentos sujeitos à cura úmida tradicional e à cura sob carbonatação acelerada.

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SEGUNDA PARTE - ARTIGOS

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Valorization of sorghum wastes as raw material for cement-based extruded composites

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Abstract

Synthetic fibers are efficient to modify the brittle nature of cement-based materials but do not depict an ecological solution, while the use of asbestos fibers is already widely banned for causing lung diseases. Alternative means are evidenced by the increase in research evaluating the benefits of using vegetal fibers. This study aimed to determine the ideal content of sorghum fibers to compose the cement-based extruded. The composites were produced using 1–5% sorghum fibers, 33% ground agricultural limestone, 1% hydroxypropylmethylcellulose, 1% additive carboxylic polyether, and 65–60% high early-strength cement. The apparent density, water absorption, and porosity, as well as the static bending properties, were evaluated. Air permeability was determined to ascertain the effect of the fibers on the interconnected porosity. The use of sorghum fibers as a substitute for cement proved to be viable to a replacement content of 1%, resulting in contents of 6.95% water absorption and 12.89% apparent porosity. For this fiber content, compared to the composite without fibers, there was a decrease in the modulus of rupture and an increase in the modulus of elasticity. Among the fiber contents tested, the best mechanical performance was obtained at 1% fiber. Thus, the use of sorghum fibers in the production of cement-based values this agricultural waste, as well as mitigating the environmental pollution caused by its burning or improper disposal.

Keywords: Cementitious matrix. Lignocellulosic material. Natural fiber. Mechanical properties. Extrusion.

1. Introduction

Asbestos was the most common reinforcement material used in fiber-cement production, and as shown by Silva et al. (2005), studies are being carried out to replace this natural fiber, which already forbidden in many countries because it is associated with several health problems in workers (Baran et al., 2016). A wide variety of fibers, including glass (Khorami et al., 2017), steel (Antonova et al., 2021), carbon (Belli et al., 2020), polymer (Li et al., 2022), and textile (Iorio et al., 2021), have been studied to replace asbestos fibers in cementitious matrices. However, due to ecological appeals allied with the high cost of these fibers production, aroused interest in the study of natural fibers to compose the fiber-cement matrices.

The use of sustainable materials in the fabrication of building components offers an opportunity to mitigate the environmental impact of civil construction, which has expressive indicators for nonrenewable resource consumption, energy consumption, and solid waste generation (Pereira et al., 2013; Onuaguluchi and Banthia 2016; Santos et al., 2017). Natural fiber-reinforced composites are currently regarded as one of the most promising materials in engineering technologies Belaadi et al. (2013). Because, while natural fibers increase some mechanical properties of composites, the environmental problems caused by the burning of straw and other residues from agricultural production can be reduced (Wang et al., 2020).

Plant fibers are abundant and are used because they are inexpensive, renewable, and readily available. As a result, studies are being conducted to identify economically viable, sustainable, and efficient solutions for technological applications, such as those based on clay, rubber, polypropylene, and polylactic matrices (Barbieri et al., 2013; Silva et al., 2015; Ngaowthong et al., 2019; Yangthong et al., 2019;). Particularly in the case of cement-based composites, the use of fibers plays a critical role in mitigating cracking caused by cement shrinkage and postcracking, and the fibers provide a bridging effect on the transfer of stresses within the cracked cementitious matrix (Raabe et al., 2018). Agricultural production generates a large amount of waste, the majority of which is discarded in inappropriate locations and conditions, posing a serious environmental problem. Thus, using waste from this sector as reinforcement material in cementitious matrices can be considered an excellent alternative, both in terms of waste disposal and as a substitute for high-cost materials used in the manufacture of fiber-cement. Numerous lignocellulosic fibers can be used as reinforcement in cementitious matrices, but factors affecting the product's properties, such as size, species, and chemical compositions, must be considered (FPL, 2010). Although fiber content affects the mechanical properties of composites, failure of bending composite elements can be attributed to matrix fiber slippage, adhesion loss, or fiber rupture (Anjos et al., 2003). Sorghum is the fifth most important cereal in the world and provides food for over 500 million people in more than 30 countries (Mace et al., 2013). It requires little water and is more tolerant of soil moisture deficits, allowing it to be grown in a wide variety of soil conditions (Kaplan et al., 2019). Sorghum is grown in Brazil for the purpose of producing animal feed, and its cultivation has increased in both planted area and yield. The 2020-2021 harvest produced 2.084×10^6 tons of grain on 864.6×10^3 hectares of the planted area, yielding 2.41 tons per hectare. For the 2021-2022 harvest, the planted area was expected to reach 950.2×10^6 hectares, yielding 3.041×10^3 tons at a rate of 3.201 tons per hectare. The South-Central region is the most productive, accounting for 83.20% (1.734×10^6 tons) of the national harvest (CONAB, 2022).

Several studies have shown the potential for using vegetable fibers as a substitute for cement in extruded cementitious composites, such as Fonseca et al. (2019) in fiber-cement with jute fibers, Teixeira et al. (2019) using curauá fibers, and cellulose pulp fibers Raabe et al. (2022). However, there is a gap involving studies using sorghum fibers in cementitious matrices. The incorporation of sorghum fibers into composites enables new applications for these wastes, thereby increasing the value of a waste product and enabling the generation of a new product on the market.

In this context, based on experimental results of physical properties and static bending mechanical performance, this study aimed to evaluate the ideal content of sorghum fibers in cement-based composites produced by extrusion.

2. Materials and methods

2.1 Obtainment of sorghum fibers

Sorghum (*Sorghum bicolor* (L.) Moench) was collected to produce fiber-cement composites on a farm in the city of Jataí, GO, a municipality located southwest of Goiás, at coordinates 17°52'53" S latitude and 51°42'52" W longitude, at an average altitude of 700 m. After collecting the material, it was ground in a hammer mill to produce sliver-type fibers. Only fibers that passed through the 0.42 mm sieve and remained in the 0.25 mm sieve were used. Prior to the production of the composites, the sorghum fibers were dried to a moisture content of 3%.

2.2 Physicochemical characterization of sorghum fibers

The physicochemical characterization of sorghum fibers was performed by moisture analysis according to E871-82 (ASTM, 2019), fiber density analysis according to the adaptation of NBR 11941 (ABNT, 2003) guidelines by Protásio et al. (2013), total extractive analysis according to NBR 14853 (ABNT, 2010) standard, insoluble lignin content according to NBR 7989 (ABNT, 2010), ash content according to NBR 13999 (ABNT, 2017), holocellulose and cellulose content according to the procedure described by Browning (1963) and Kennedy et al. (1987), respectively.

2.3 Production of fiber-cement composites

The mixing proportions in mass used in the production of composites (Table 1) were 0% fiber (named Control composite), 1%, 2%, 3%, 4%, and 5% sorghum fibers in place of cement, 33% agricultural limestone, 1% HPMC, 1% ADVA, and 65–60% high early-strength cement (Portland cement CPV-ARI, according to NBR 5733 (ABNT, 1991) and equivalent to ASTM cement type III).

Table 1. Mix design used in the production of the fiber-cement composites.

Materials	Mass (%)
Portland cement CPV-ARI	65–60
Agricultural limestone	33
Dry sorghum fiber	0–5
HPMC	1
ADVA	1
Total	100

Portland cement CPV-ARI was chosen because of the absence of additives and was therefore considered less aggressive in terms of mineralization/degradation. Additionally, the high initial strength promotes mechanical properties and mitigates the effect of extractives (Bezerra et al., 2006). Ground limestone was used in place of Portland cement to reduce the cost of the manufacturing process and, as recommended, for the composite's stabilization (as it is considered an inert ingredient) and for sustainability reasons (Fonseca et al., 2019). The additives hydroxypropylmethylcellulose (HPMC) and carboxylic polyether (ADVA) were used to aid in the rheology of the mixture. These additives were used to increase the water retention capacity, as well as to improve labor productivity and the cement stabilization process. For each additive (HPMC and ADVA), the proportion of 1.0% of the total mass of each mixing was used.

A planetary mixer was used to combine the ingredients. Cement, limestone, fibers, and HPMC were first mixed for two min at a speed of 140 rpm. After adding the ADVA and water, the mixing process was continued for five min at 285 rpm to ensure the uniform distribution of the fibers in the formed mass. The water-cement (w/c) ratio was 0.40.

The fiber-cement paste was processed in a helical extruder. The mass was passed through the extruder three times to ensure proper homogenization and orientation of the sorghum fibers for extrusion (Fig. 1). After molding, the specimens were sealed in plastic bags and stored at high humidity and room temperature for 27 days. Following a 27-day wet cure period, the composites were cut with a disk saw to achieve final dimensions of 28 mm × 18 mm × 200 mm (width, thickness, and length, respectively). After 24 h of immersion in water, the specimens were subjected to physical and mechanical tests. Each composition required the preparation of eight specimens for testing.

Fig. 1 Extruded fiber-cement molding process. (a) Specimen output through the extruder nozzle; (b) Specimens wrapped in a plastic bag containing water inside for the wet curing period

2.4 Physical and mechanical properties of composites

Physical tests for water absorption (WA), apparent porosity (AP), and apparent density (AD) were conducted following standard C 948-81 (ASTM, 2016).

Static bending tests were conducted using a universal testing machine equipped with a 20 kN load cell (EMIC DL, Brazil). To determine the mean values of modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE), and toughness, a four-point bending test was configured with a span of 150 mm, and the loading was applied at a constant crosshead speed of 1.5 mm/min.

2.5 The effect of sorghum fibers on the hydration of cement paste

The method used to determine the inhibition of sorghum fibers by cement was modified from Hofstrand et al. (1984). Fifteen grams of dry sorghum fibers, 200 g of Portland cement CP V-ARI/Plus, and 90 mL of water were combined for this test. After homogenization for 5 min, these materials were placed in Styrofoam boxes lined with aluminum foil to minimize temperature loss to the environment. Each Styrofoam box contained a temperature sensor connected to a Datalogger, which recorded data at one-second intervals over 24 h. For this test, a PicoLog data acquisition system, model TC-08, connected to a computer and type k thermocouples was used.

The box and data acquisition system were housed in an acclimatized room set to a temperature of 20 ± 1 °C and relative humidity of 60 ± 5% to further eliminate external interferences. Three replicates using sorghum fibers were performed, in which the fibers passed through a 4.9 mm sieve but were retained in a 2.4 mm sieve. The inhibition index was calculated using Equation 1 (Okino et al., 2004):

$$\mathrm{I} = \left[\frac{(\mathrm{TC} - \mathrm{TS})}{TC}\right]\left[\frac{(\mathrm{HS} - \mathrm{HC})}{HC}\right]\left[\frac{(\mathrm{SC} - \mathrm{SS})}{SC}\right] \times 100 \tag{1}$$

where I is the cement curing hydration index (%); TC is the maximum temperature of the cement paste (°C); TS is the maximum temperature of the sorghum fiber/cement paste (°C); HC is the time to reach the maximum cement hydration temperature in the cement paste (h); HS is the time to reach the maximum temperature of the cement hydration mixture in the sorghum fiber/cement paste (h); SC is the maximum temperature increment of the curve in the cement paste (°C/h); and SS is the maximum temperature increment of the curve in the sorghum fiber/cement paste (°C/h). The effect of cement curing inhibition is classified according to Table 2 (Okino et al., 2004).

Table 2. Classification of lignocellulosic material according to the hydration index obtained.

Hydration index (%)	Classification
I < 10	Low inhibition
I = 10 to 50	Moderate inhibition
I = 50 to 100	High inhibition
I > 100	Extreme inhibition

2.6 Analysis of the rupture surface of the composites

After the composites fractured in the static bending test, they were investigated by scanning electron microscopy (SEM). To obtain the micrographs, the specimens were coated with gold in an evaporator and then analyzed in a LEO EVO 40 XPV at an electrical voltage of 20 kV.

2.7 Air permeability tests

Permeability parameters were determined using experimental data and fitting of Forchheimer's equation (Equation 2), a well-established empirical relationship that expresses the parabolic dependence of pressure drop (ΔP) with the resulting superficial or face velocity

(v_s) of the fluid through the medium (Innocentini et al., 2017; Innocentini et al., 2019; Innocentini et al., 2005; Fioroni et al., 2020; Corradetti et al., 2016):

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s + \frac{\rho}{k_2} v_s^2 \tag{2}$$

where L denotes the medium length or thickness along the macroscopic flow direction and μ and ρ denote the fluid's viscosity and density, respectively. The parameters k_1 and k_2 are referred to as Darcian and non-Darcian permeability coefficients, respectively. These coefficients are dependent on the porous structure and are used in Equation (2) to balance the effects of viscous and inertial losses on the total pressure drop. For compressible flow, ΔP in Equation (2) must be determined using Equation (3):

$$\Delta P = \frac{{P_i}^2 - {P_o}^2}{2P} \tag{3}$$

where P_i and P_o are the inlet and outlet absolute gas pressures, respectively. P denotes the absolute pressure at which v_s, μ and ρ are measured or calculated (in this work $P = P_o$).

The permeability coefficients k_1 and k_2 were determined experimentally using a laboratory-built device, with testing conducted in a steady-state regime with dry airflow at ambient temperature (T = 23-29 °C, $P_{atm} = 94.7$ kPa, $\mu = 1.88 \times 10^{-5}$ Pa.s; $\rho = 1.1$ kg m^{-3}) on three specimens of each batch. Further details of the method and the experimental setup are given elsewhere (Innocentini et al., 2017; Innocentini et al., 2019; Innocentini et al., 2005).

The samples for the air permeability tests were named S0 (control), S3 (3% sorghum fiber), and S5 (5% fiber content), and the apparatus's design and methodology are described elsewhere (Innocentini et al., 2019) and illustrated in Fig. 2.

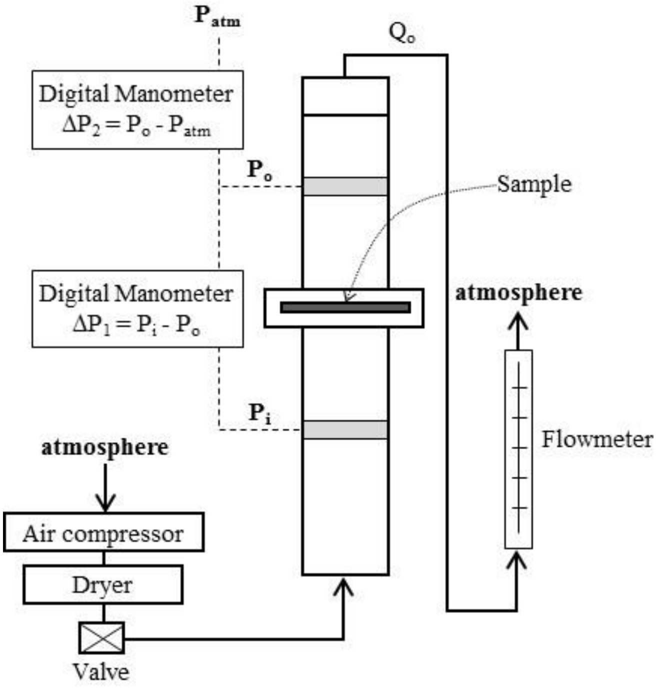


Fig. 2 Scheme of the airflow permeation apparatus

3. Results and discussion

3.1 Sorghum fiber properties

Table 3 summarizes the mean values and standard deviations for the chemical characteristics of sorghum fibers. This constituent distribution demonstrates a high extractive content, and as Chafei et al. (2014) demonstrated, the amount of total extractives present in the lignocellulosic material can have a significant effect on the setting time and cement hydration, thereby impairing the mechanical strength of the composites.

Table 3. Chemical composition of sorghum fibers (%, dry basis).

Chemical constituents	Content (%)
Total extractives	13.21 ± 1.24*
Insoluble lignin	17.19 ± 0.94*
Cellulose	34.27 ± 2.78*
Hemicelluloses	32.86 ± 2.13*
Ash	2.47 ± 0.06*

*Standard deviation.

The chemical composition of the lignocellulosic material under investigation may be the primary impediment to manufacturing cementitious composites (Mori et al., 2007). Simatupang et al. (1978) asserted that the extractives found in wood and lignocellulosic materials are responsible for cement setting inhibition, with their active principles being phenolic compounds and free carbohydrates. This behavior occurs because extractives can preclude Portland cement from hardening, preventing the composite from reaching the temperature required for the cement hydration reaction to occur (Marques et al., 2016). Additionally, the authors assert that the addition of vegetable fibers to the cement mass alters the composite's thermal equilibrium and the intensity of hydration reactions.

According to Bledzki and Gassan (1999), a high lignin content decreases the mechanical performance of the composite. This is because the lignin found in plant fibers has an amorphous structure that is highly soluble in an alkaline environment, resulting in increased alkaline attack by the cement (Correia et al., 2015). On the other hand, cellulose contributes significantly to the mechanical strength of cementitious composites owing to its microfibril structure, which results in a high tensile strength and high crystallinity.

Lignocellulosic materials are considered incompatible with cementitious paste due to the presence of soluble hemicellulose, which is degraded into simple sugars in alkaline solutions, interfering with the cement hydration process (Sutigno, 2000; Macêdo et al., 2012; Pelaez-Samaniego et al., 2013).

The cellulose and lignin contents of sorghum fibers determined in this study were slightly higher than those determined by Pedreira et al. (2003), who found 25.3–31.2% cellulose and 3.6–5.5% lignin for eight sorghum hybrids. For hemicelluloses, the values of the present work are within the range observed by the author of 24.8–34.3%. This discrepancy in results can be explained by the variety of species, the location of cultivation, the season, and the method of harvesting, all of which affect the chemical composition of lignocellulosic materials. These chemical characteristics should be analyzed because they directly affect the quality of cementitious composites, as they are responsible for the bond with the cement matrix and the degradation of natural materials used as reinforcement in cementitious composites (Lee et al., 1987).

The average density of sorghum fiber was 0.16 g cm^{-3} at a moisture content of 10.89%. The use of low-density fibers as reinforcing material can have a significant effect on the extrusion process, as it increases the number of fibers scattered throughout the cementitious matrix, reducing the mass's extrudability due to fiber cohesion. Additionally, a low density can hinder the homogenization of the mixture due to the presence of balls, clogging the extruder.

3.2 Physical properties of composites

The content of sorghum fiber used had a statistically significant effect on the physical properties of water absorption (WA), apparent porosity (AP), and apparent density (AD). Fig. 3 illustrates the average values obtained for the water absorption of cementitious composites, showing that the increase in sorghum fiber content increased water absorption by the composite.

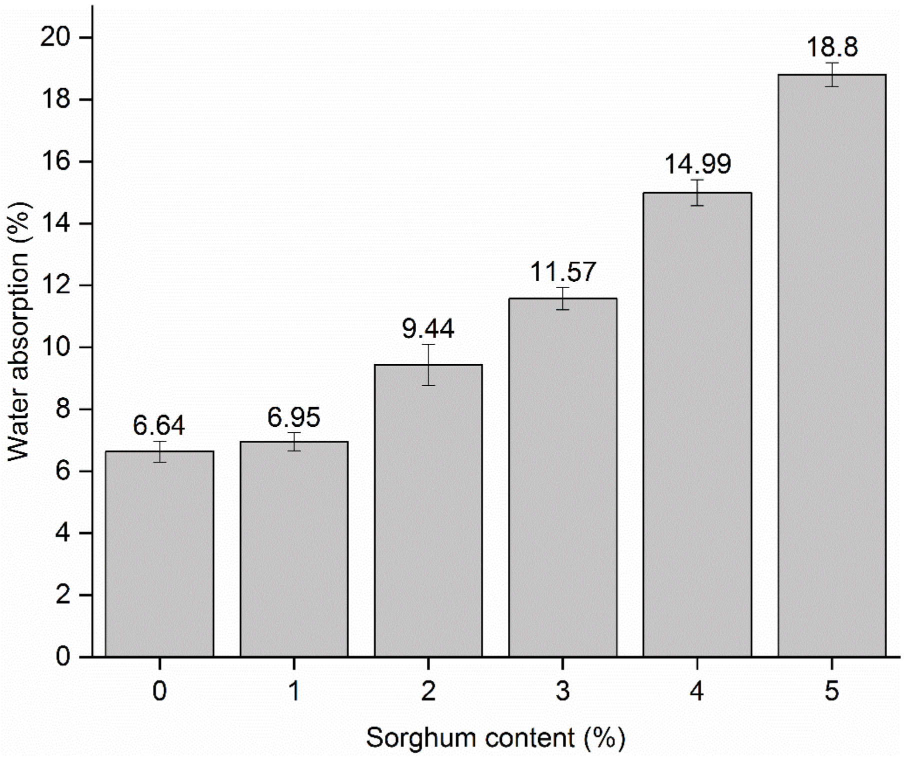


Fig. 3 Water absorption of fiber-cement produced with different contents of sorghum fibers

Fig. 4 shows that the average values for apparent porosity as a function of sorghum fiber content followed a similar trend to water absorption. In both cases, the effect of fiber addition in comparison with the fiber-free composite is clear.

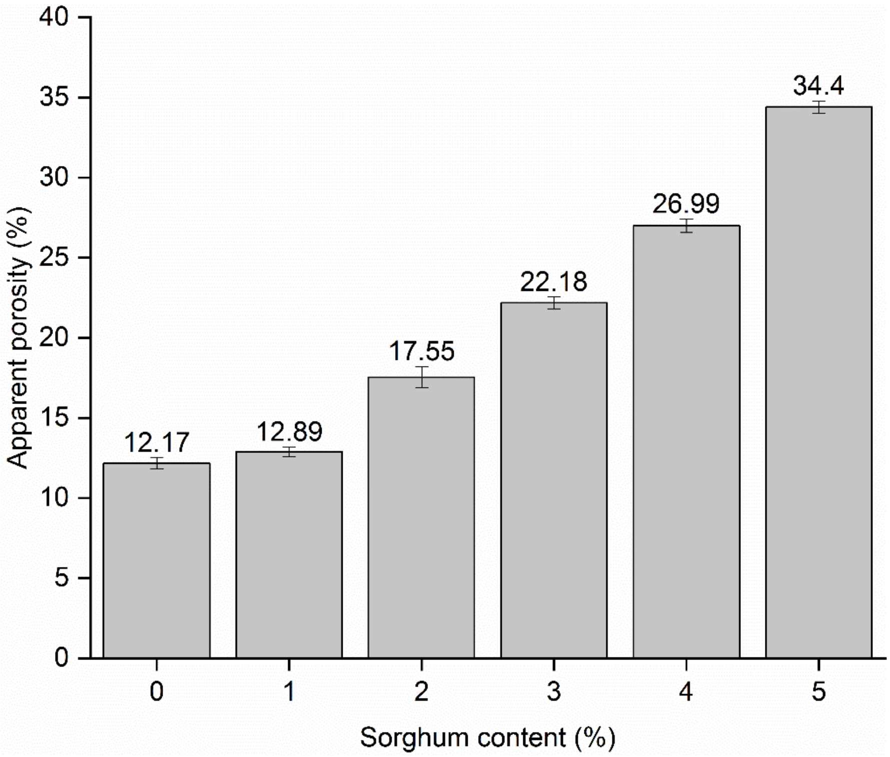


Fig. 4 Apparent porosity of fiber-cement produced with different contents of sorghum fibers

Fig. 5 shows that the apparent density of the composites decreased significantly for the two highest fiber contents (4% and 5%). This effect, associated with a density ratio (sorghum fibers/cement paste) of 0.106, could be attributed to a poorer packing density of the cement matrix caused by the presence of too many fibers. Fig. 3 and Fig. 4 show that apparent porosity and water absorption increased faster for these fiber contents, which is consistent with the change in packing density caused by fibers.

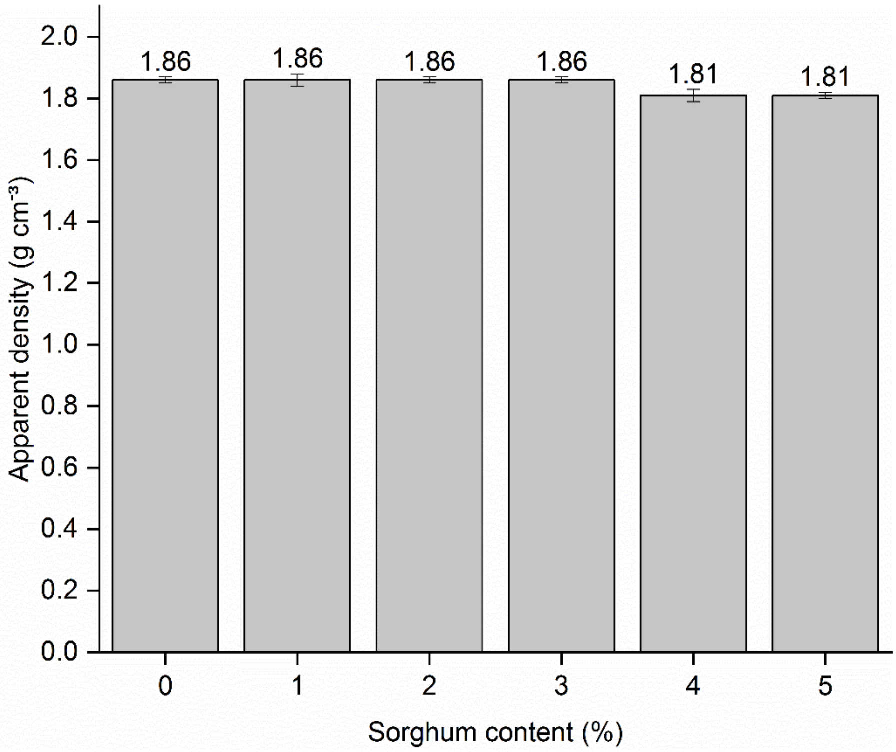


Fig. 5 Apparent density of fiber-cement produced with different contents of sorghum fibers

The fact that apparent density remained constant up to 4% and 5% fiber content, despite the increased porosity of the composite, is because the fibers are lighter than cement paste. Composites produced with 5% sorghum fibers exhibited the highest apparent porosity and water absorption. This may be a disadvantage due to the hydrophilic nature of the sorghum fibers, which increases the appearance of defects in the composite microstructure because of their insertion into the cement matrix (Fig. 6). Therefore, the increased apparent density and decreased apparent porosity indicate that the matrix appears to be more compact and contains fewer defects (Tonoli et al., 2010).

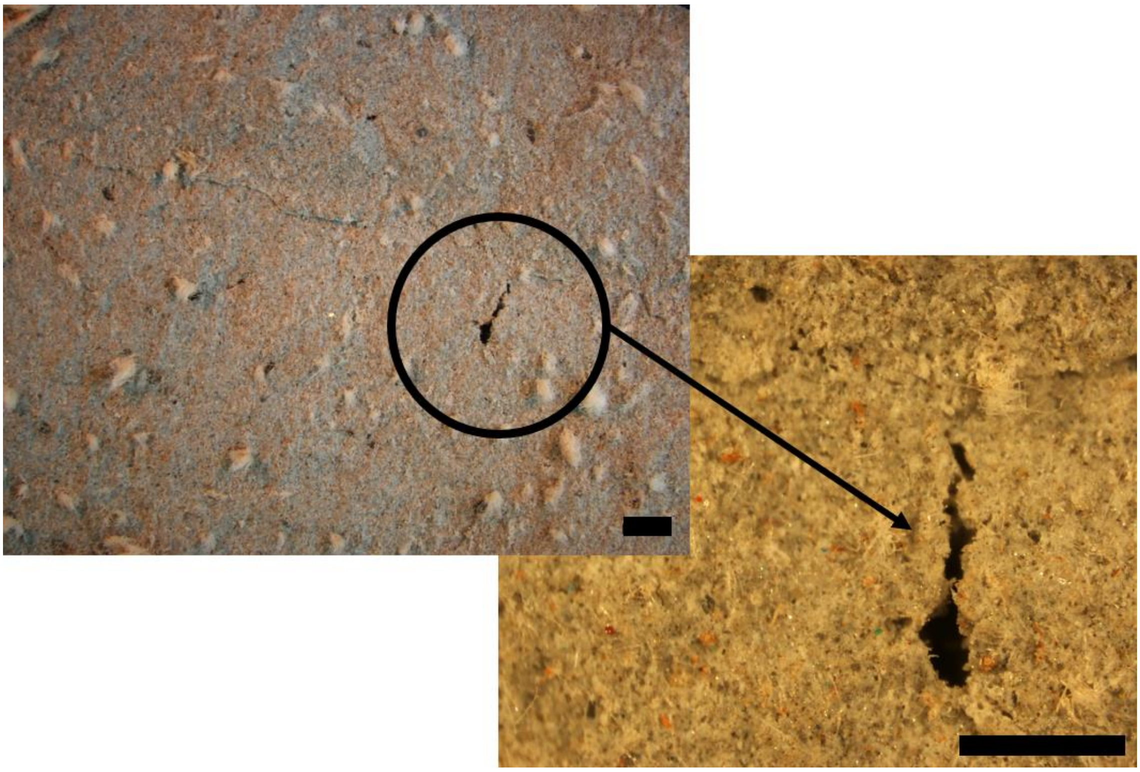


Fig. 6 Cracks on the surface of composites produced with 5% sorghum fibers. The scale unit shown in each part is equal to 1000 μm

To the best of the authors' knowledge, there are no examples in the literature of fiber-cement materials produced with sorghum fiber replacement. However, Fonseca et al. (2016) observed the effect of varying contents of eucalyptus nanofibrils on the physical-mechanical properties of fiber-cement after 28 days of cure. Brasileiro et al. (2013) conducted a study that found behaviors consistent with the findings of this research. The purpose of the study (Brasileiro et al., 2013) was to investigate the use of coconut fiber in the manufacture of cementitious composites and the effect of sand addition on physical and mechanical properties.

In general, the physical properties obtained were satisfactory. The formulations used achieved acceptable results, exhibiting physical property values consistent with those specified in NBR 7581-2 (ABNT, 2014), which stipulates a maximum water absorption value of 37% for fiber-cement produced without asbestos.

3.3 Mechanical properties of composites

The typical curve of normal bending stress and specific deformation (Fig. 7) presents the mechanical behavior of different contents of substitution from sorghum fiber composites.

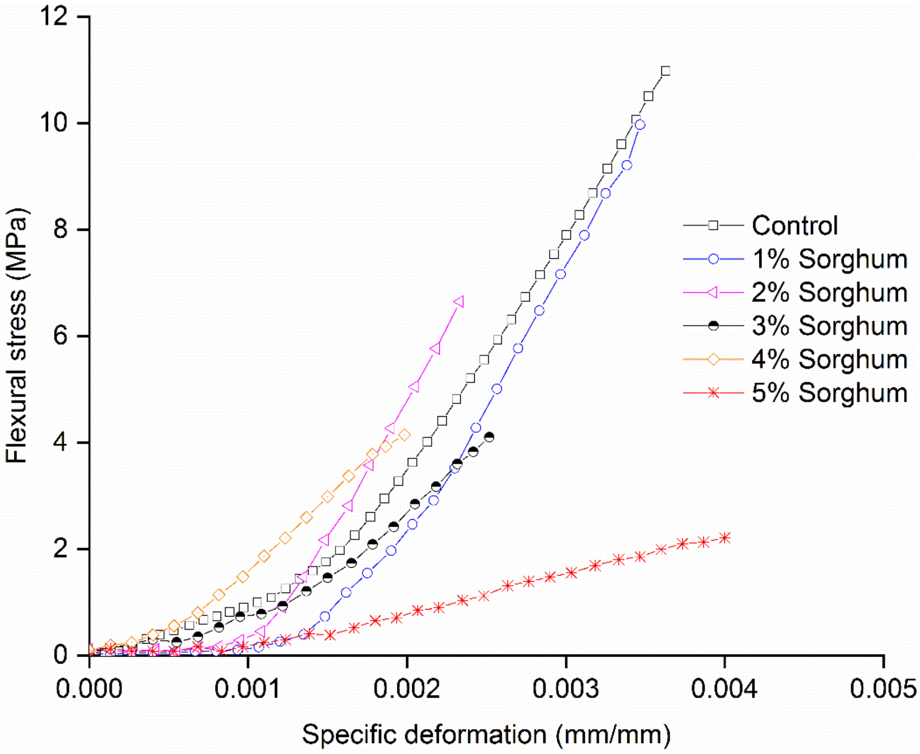
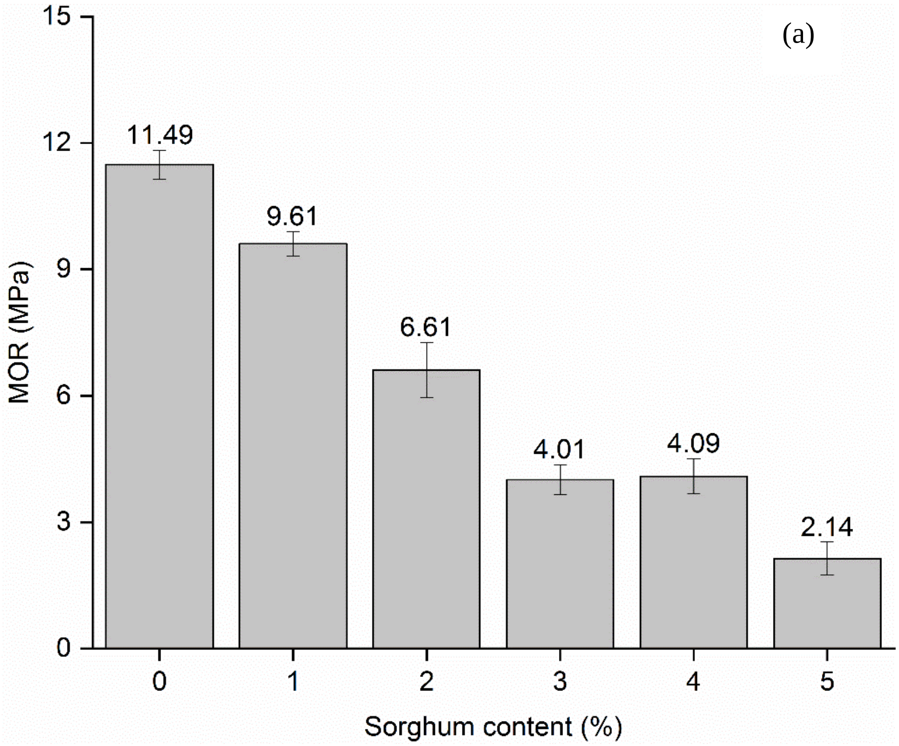
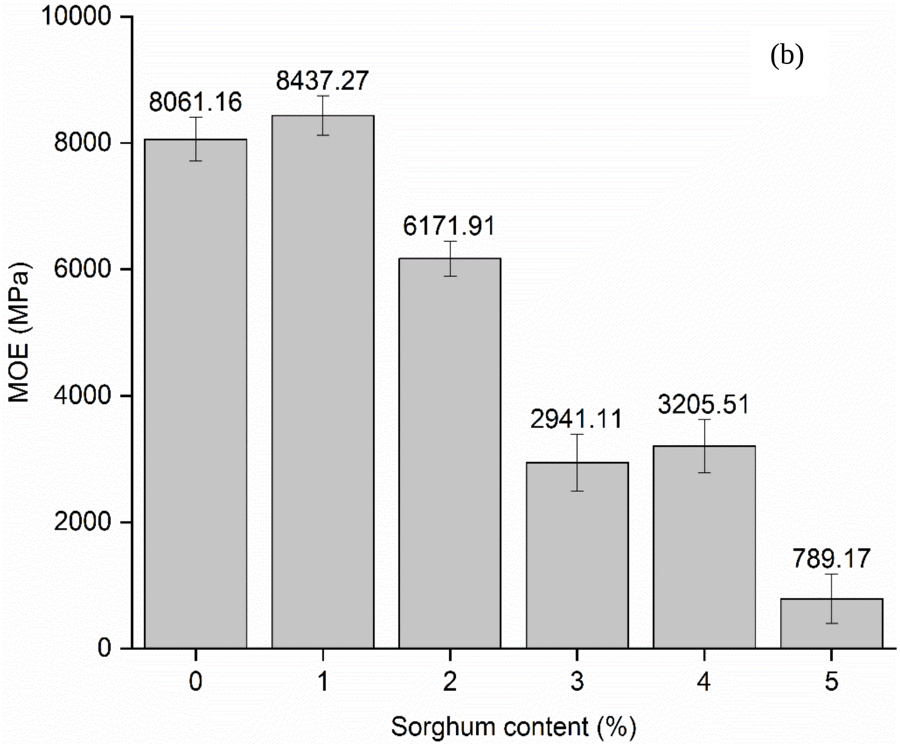


Fig. 7 Normal bending stress vs. specific deformation of fiber-cement composites

The composites produced without the addition of sorghum fibers (control) and containing 1% fibers reached a maximum stress significantly superior to the other composites. Due to the nature of the composites, it is observed that all of them had a brittle fracture, presenting low values of specific deformation. For the specimens with 5% fibers, a less fragile behavior was observed than the others; however, there was an abrupt decrease in the normal bending stress.

The mean values and standard deviations for the modulus of rupture, modulus of elasticity, and limit of proportionality are shown in Fig. 8 (a-c).





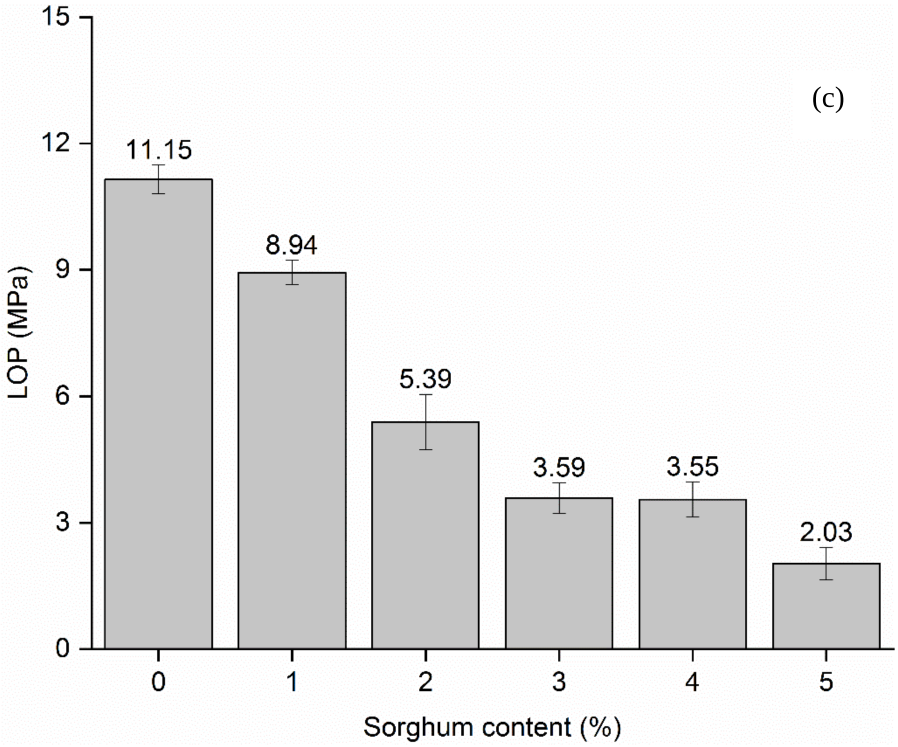


Fig. 8 Static bending testing of specimens produced with different contents of sorghum fibers, (a) MOR; (b) MOE; (c) LOP

As observed in Fig. 8 and Fig. 9, there was a significant effect of sorghum fiber content on the mechanical properties of the composites. There was no significant difference in the average MOE value (Fig. 8b) with only 1% fiber added, followed by a significant decrease in the results with the other fiber contents. The decrease in MOE (Fig. 8b) as the fiber content increases can be attributed to the high apparent porosity of fiber-reinforced composites in comparison to the control specimen. Due to the increased number of voids, this high porosity can result in a decrease in the modulus of elasticity. Additionally, fibers have a lower modulus of elasticity than cement paste.

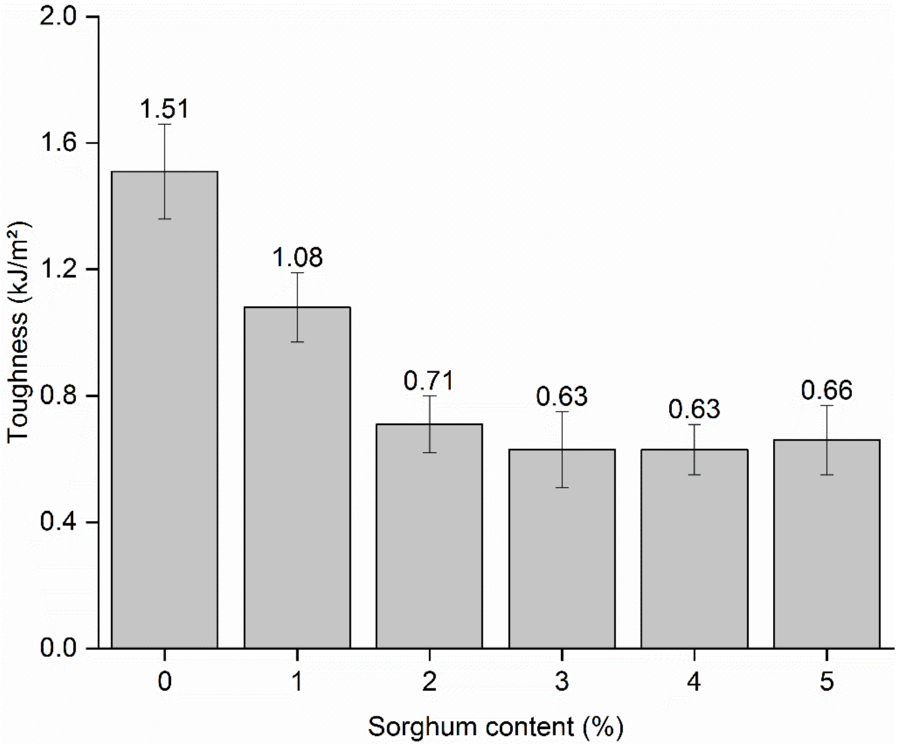


Fig. 9 Toughness of fiber-cement produced with different contents of sorghum fibers

The values of MOR (Fig. 8a), LOP (Fig. 8c), and toughness (Fig. 9) decreased with increasing sorghum fiber content. Composites with 3%, 4%, and 5% fiber had lower values compared to those with lower fiber additions. These findings are explained by the inhibition of cement curing around the fibers (Fig. 10), which is caused by the presence of extractives and hemicelluloses on the fiber surfaces, resulting in a low interaction between the cement matrix and fiber. The presence of lignin, with its nature of susceptibility for dissolution in an alkaline medium, is considered to contribute to the reduction of the tensile strength of vegetable fibers (Almeida et al., 2013).

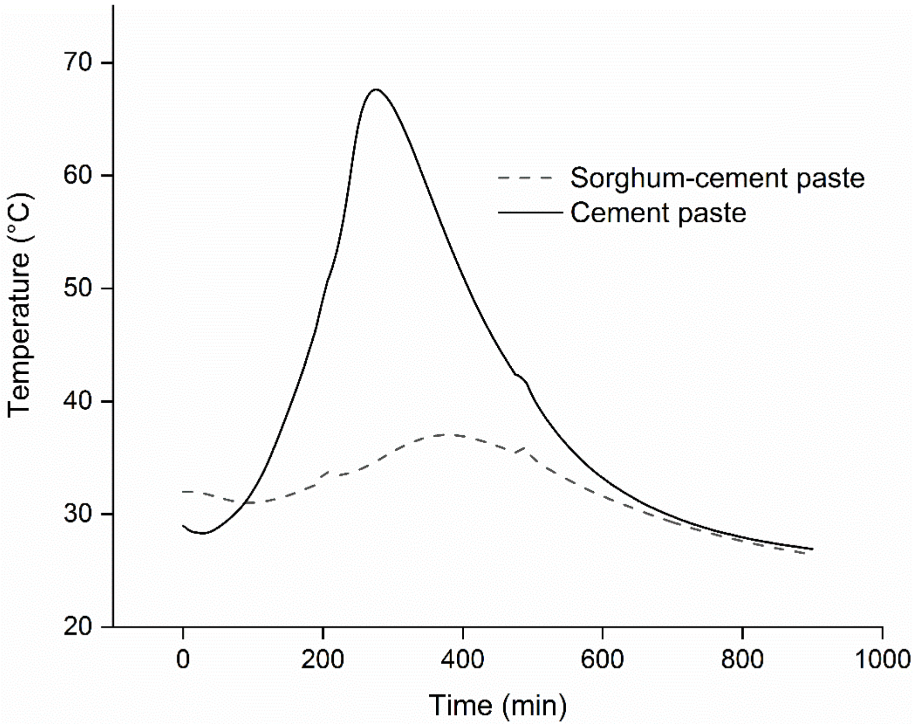


Fig. 10 Hydration curve of cement paste and sorghum-cement paste

As shown in Fig. 11 and by Equation 1, the cement paste containing sorghum waste had a hydration index of 15.53% and, following Okino et al. (2004), could be classified as medium inhibition. In Fig. 10, the maximum temperature reached by the sorghum-cement paste was approximately 37 °C, far from the maximum temperature of the cement paste, which was approximately 67 °C. Notably, for the sorghum-cement paste, the maximum temperature occurred at a hydration time longer than the time required for the cement paste, confirming the high inhibition index and the incompatibility between the cement and the sorghum fiber.

The decrease in the average values of the mechanical properties evaluated can be attributed to the sorghum fibers' weak linkage with the cementitious matrix. Fig. 11 illustrates the presence of pores in the cementitious matrix caused by sorghum fiber detachment.

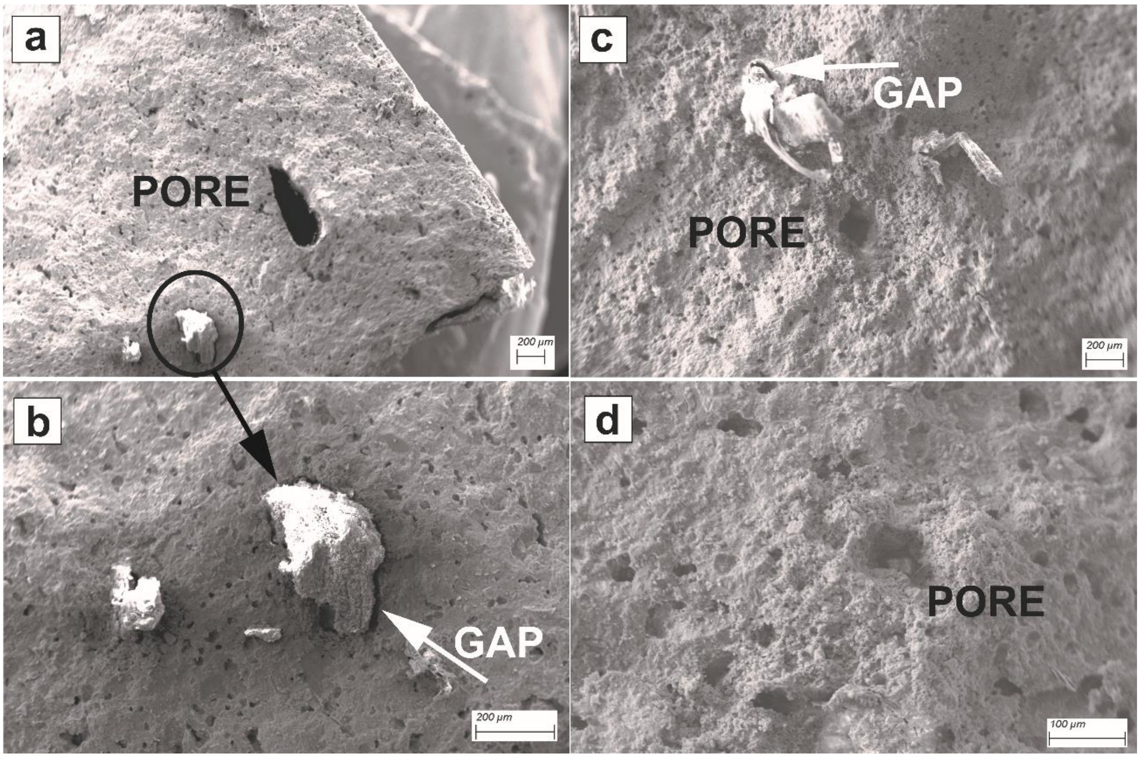


Fig. 11 Electron micrograph surface of composites produced with 3% sorghum fiber content (a, b) and 5% sorghum fiber content (c, d)

Analysis of the electron micrograph (Fig. 11) showed voids around the fibers that did not detach from the matrix, indicating a discontinuity in the stress flow between the sorghum fibers and the cementitious matrix. The strength-reducing effect of cement-based products is a consequence attributed to the formation of capillary pores (0.01-10 μm) and macropores (≥ 0.05 μm) (Almeida et al., 2013). The void spaces between the fiber and matrix are mainly attributed to the presence of a high concentration of extractives on the fiber surface, but they can also be related to the strong pressure between the matrix and fiber caused by the extrusion process. Finally, this gap between the fiber and matrix can also be caused by the contraction of the vegetable fiber due to the drying necessary to prepare the sample and the vacuum that the sample is subjected to inside the observation chamber of the electron microscope.

In addition, for composites produced without the addition of sorghum fibers, the rupture surface does not present large pores, caused by detachment by the fiber in the cement matrix, as shown in Fig. 12.

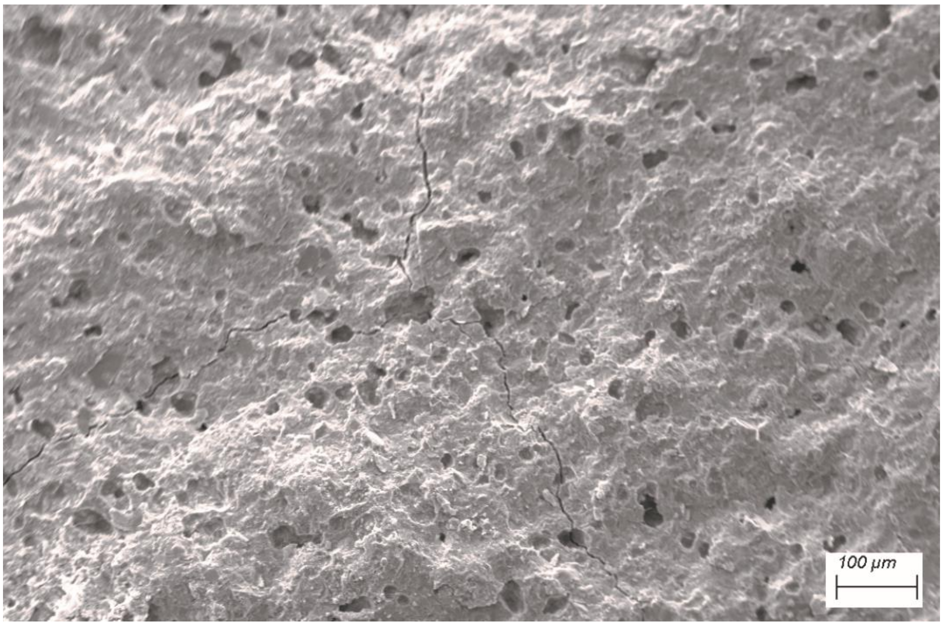


Fig. 12 Electron micrograph surface of the composite control (0% sorghum fiber)

In studies on the production of composites with vegetable fiber, Chakraborty et al. (2013), Wei and Meyer (2015), Teixeira et al. (2019), and Fonseca et al. (2019) demonstrated that because of this component, there is an alteration in the hydration behavior of cement. Additionally, they demonstrated that the high alkalinity of the cementitious matrix causes the weakening of fibers, with consequent mineralization.

According to Tonoli et al. (2013), increased LOP values indicate improved fiber-matrix adhesion; however, this was not the case in the current study, possibly due to poor fiber scattering in the matrix or a deficiency in stress transfer between fiber and cementitious matrix (Tonoli et al., 2013).

Increased toughness values indicate that fibers contribute more to delaying the initiation of crack propagation (Correia et al., 2015), which was not the case in this study, as toughness decreased as the content of fibers increased. The poor adhesion between the fiber and the cement matrix, confirmed by SEM images, caused this behavior.

As previously stated, there are no studies in the literature on fiber-cement produced with sorghum fibers. Nonetheless, investigating the effect of carbonation on extruded vegetable fiber-cement composite, which resulted in an MOR equal to 14-19 MPa, Santos et al. (2015) reported similar behaviors to those found in this study.

Another study by Lertwattanaruk and Suntijitto (2015) used coconut shell fibers and palm fibers as substitutes for cement in fiber-cement with contents of 5, 10, and 15%. The authors observed that as the content of fibers was increased, MOR values decreased in the range from 3.71-21.89% when compared to fiber-cement without fibers. The decrease in mechanical properties of composites observed in this study may be due to the mineralization of sorghum fibers because of cement hydration products migrating to porous surfaces, a phenomenon also described in the pine fiber-cement composite production study (Tonoli et al., 2012).

Overall, the mean mechanical properties obtained in this work were similar to or inferior to those reported in the literature to produce cement composites with other types of plant fibers. Composites produced with up to 1% sorghum fibers were classified as category 3 by standard NBR 15498 (ABNT, 2016), which considers an MOR greater than 7 MPa. According to the same standard, composites containing 2%, 3%, and 4% were classified as category 2, indicating an MOR greater than 4 MPa. Composites produced with 5% sorghum fibers were classified as category 1, having an MOR less than 4 MPa. This result is due to the fiber's low adhesion to the matrix, which results in a greater number of pores and, consequently, lower MOR values.

3.4 Air permeability of composites

The permeation curves for all tested samples are given in Fig. 13. The parabolic relationship between pressure drops and superficial air velocity was confirmed through the high-quality fitting of Forchheimer’s equation (correlation of determination $R^2>0.99$ in all cases), observed by the dashed lines in Fig. 13.

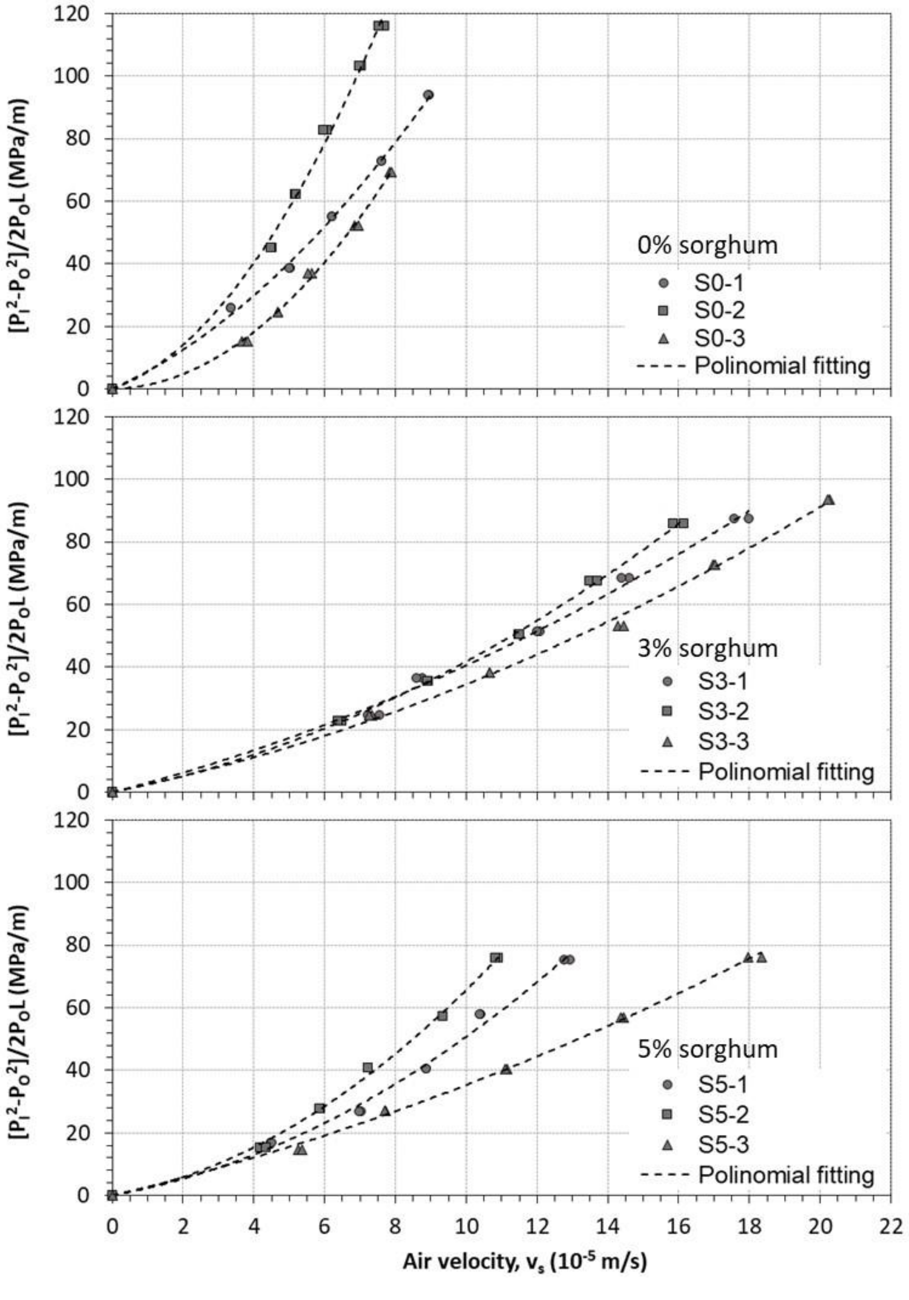


Fig. 13 Air permeation curves of samples with different contents of sorghum fibers

The permeability coefficients k_1 and k_2 were obtained from the fitted data and are given in Fig. 14.

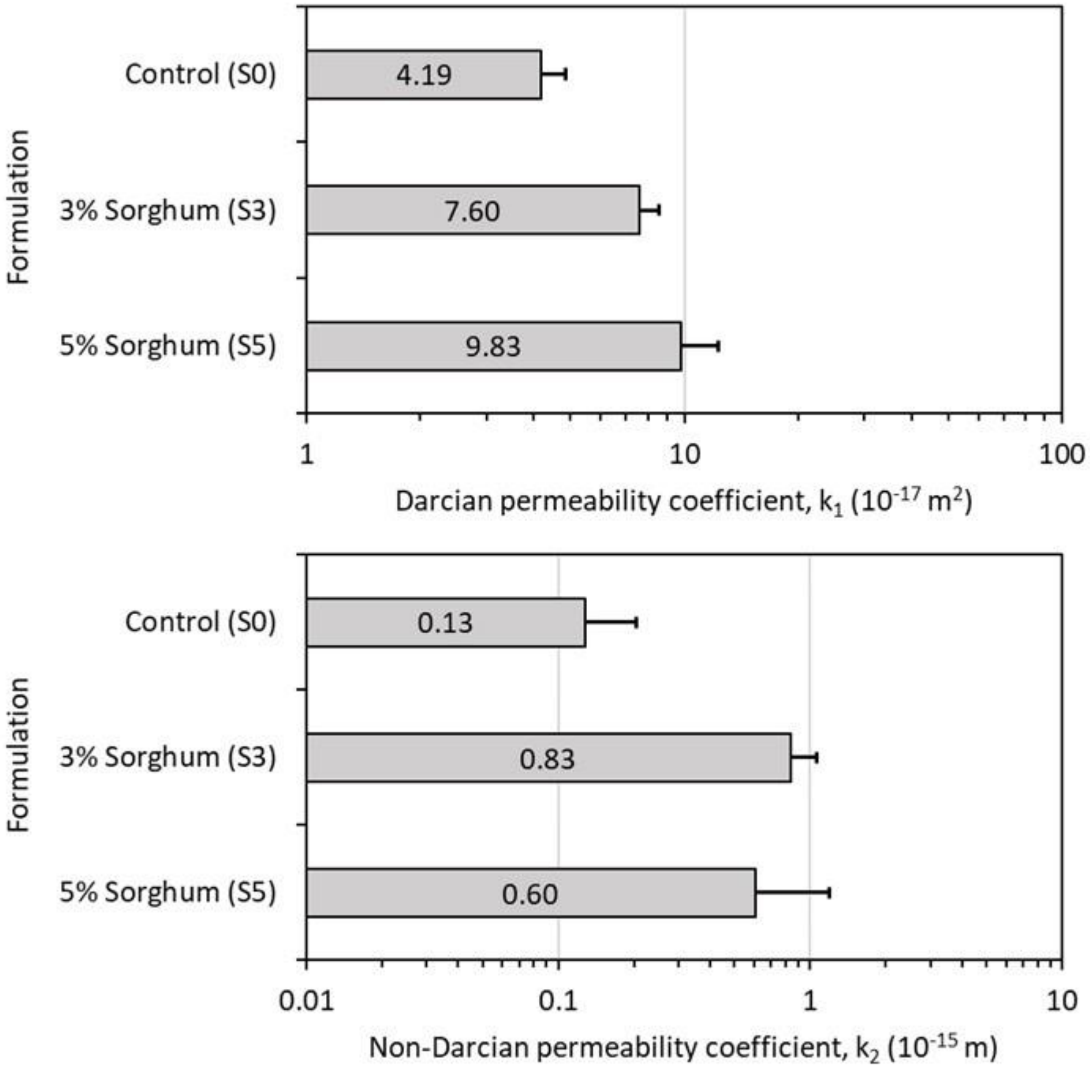


Fig. 14 Permeability coefficients of tested samples (average and deviation)

Increased apparent porosity had a significant effect on the mechanical properties of the composites, lowering MOR, MOE, and LOP values. As illustrated in Fig. 14, increasing the amount of sorghum fiber incorporated into the composites increased the permeability coefficients k_1 and k_2. This behavior is explicable because the primary permeation pathway is located at the fiber-matrix interface (Fig. 11), not within the matrix (Fioroni et al., 2020). As a result of the increased fiber content, the interconnections at the fiber-matrix interfaces become larger, increasing permeability.

The data on the permeability of the tested samples can also be compared to those of other materials using the map shown in Fig. 15 (Innocentini et al., 2005; Fioroni et al., 2020). The map is based on thousands of k_1 and k_2 data extracted from the literature and has been extensively used to classify the permeability of a wide variety of porous materials used in a variety of applications. The fiber-cement composites exhibit the same level of permeability as concrete, castables, and other fiber-cement materials. Additionally, the increase in permeability is caused by the increase in fiber content from 0% to 5%.

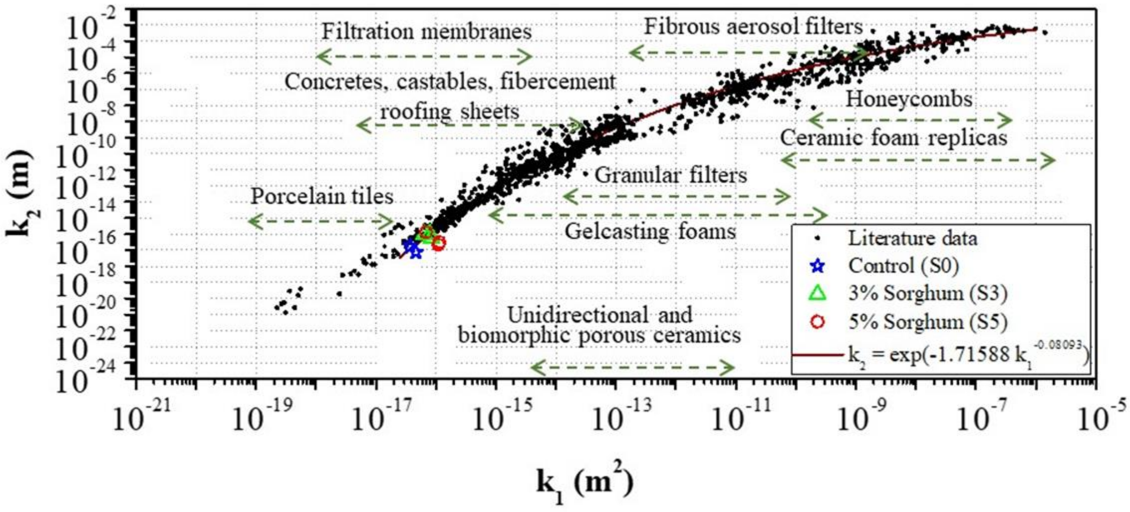


Fig. 15 Permeability map proposed in the literature (Innocentini et al., 2005; Fioroni et al., 2020) and location of samples tested in this work

4. Conclusion

The evaluation of the physical and mechanical performance of cement-based composites produced with sorghum waste fibers, with contents ranging from 1-5%, showed the feasibility of using this vegetable fiber to produce the extruded composite.

The composite produced with a fiber content equal to 1%, compared to the non-reinforced composite, showed that the WA and AP properties were little affected using fibers.

On the other hand, with fiber contents from 2-5%, these properties were abruptly increased. However, for all fiber composites, the water absorption was below the maximum of 37% established by the standard NBR 7581-2 (ABNT, 2014). In turn, the AD property was only affected by fiber contents from 4%, but with a reduction of only 2.7% compared to composites with lower fiber contents.

Due to the use of 1% sorghum waste fibers, concerning the control composite, the mechanical property of MOE was increased by 4.7%, while the MOR and toughness were reduced by 16.4 and 28.48%, respectively. For composites with 5% fiber content, the values of MOR, toughness and MOE were reduced by 81.37, 56.29 and 90.21%, respectively. These reductions in mechanical properties were due to the inhibition of cement curing and were then assigned to the sorghum fiber extractive content. In addition, extractives caused gaps between the fibers and the cement matrix, increasing the porosity of the composites. Based on the analysis of the permeability coefficients k1 (Darcian) and k2 (non-Darcian), obtained from the air permeability tests, the fiber-cement composites produced exhibit the same level of permeability as concrete, castables, and other fiber-cement materials.

Considering these results, the best mixture to produce the composite was obtained by the replacement of 1% cement with sorghum fibers. However, the deterioration of this composite due to exposure to weathering agents must be evaluated in future research. The results of this research encourage the use of sorghum waste fibers in the production of fiber-cement. For, while the consumption of cement used in the production of cement-based extruded composites is reduced, a major environmental contribution is given by the better destination of this agricultural waste.

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Declaration of conflicting interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Availability of data and material

The datasets supporting the conclusions are included in the manuscript. Furthermore, the datasets analyzed in this study are available from the corresponding author upon request.

Code availability

Not applicable.

Author contributions

Douglas Lamounier Faria and Daiane Erika Lopes Faria: Conceptualization, Investigation, Data Curation and Writing – Original Draft. Murilo Daniel de Mello Innocentini and Julio Soriano: Methodology, Investigation and Writing – Original Draft. Lourival Marin Mendes and José Benedito Guimarães Junior: Funding acquisition, Supervision, Resources, Project administration.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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The technology of accelerated carbonation on cementitious composites produced with rubberwood fibers: a new material for engineering

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Abstract

The abundance and high tensile strength of vegetable fibers make them an interesting option to modify the fragile nature and properties of cement-based products. However, a major challenge is to prevent cement matrix mineralization. Accelerated carbonation appears to be an alternative to this process, promoting a reduction in matrix alkalinity and an increase in the fiber-matrix interface. However, the type of vegetable fiber exerts a great influence on the final properties of the composite, mainly due to its anatomical characteristics. Thus, this research aimed to evaluate the effect of curing under accelerated carbonation on the physical, mechanical, and microstructural properties of extruded fiber-cement composed with rubberwood fibers replacing the traditionally used polymeric fibers. The specimens were produced with a mixture of 60% high early-strength cement, 33% ground agricultural limestone, 5% rubberwood fibers, 1% hydroxypropylmethylcellulose, and 1% carboxylic polyether. The results showed that accelerated carbonation increased the apparent porosity (17.51% to 28.18%) and decreased the mechanical properties (11.64 MPa to 8.38 MPa for modulus of rupture, and 5891.64 MPa to 2057.36 MPa for modulus of elasticity), except for toughness, which increased abruptly by 86.20% and made the product less fragile, with the best preserved tenacity mechanisms in the fracture process of carbonated composites, such as pull-out and bridging forces. It was concluded that rubberwood fibers are an interesting alternative to producing fiber-cement composites and that accelerated carbonation reduces the aggressiveness of lignocellulosic fibers and increases the energy absorption of composites.

Keywords: Fiber-cement. Lignocellulosic material. *Hevea brasiliensis*. Mechanical properties. Extrusion.

1. Introduction

The use of sustainable materials to produce civil construction components and structural elements has the potential to reduce the environmental impact, as evidenced by the alarming indicators of consumption of nonrenewable resources, energy expenditure, and solid waste generation (Onuaguluchi and Banthia 2016). Concerning the reinforcement of cement-based matrices, carbon, glass, polypropylene, or aramid fibers are commonly used; however, the high cost and high energy consumption in the production of these synthetic fibers are recognized.

On the other hand, vegetable fibers are obtained by a low-cost process, an advantage added to the biodegradable material that does not bring concern for health and safety in handling (Merta and Tschegg 2013; Tessaro et al. 2015). The valorization of biomass by its incorporation into construction materials has become more prevalent due to the necessity of solutions that are economically viable, environmentally sustainable, and able to improve the mechanical properties of materials (Silva et al. 2015).

There is considerable interest in examining the feasibility of using vegetable fibers as reinforcement in cementitious matrices due to their high mechanical properties (Sawsen et al. 2015). Natural fibers improve the tensile and flexural strength, as well as the hardness and ductility of cement-based materials (Mejia-Ballesteros et al. 2021). The bond between the fiber and the matrix contributes to the increase in fracture strength (Wei et al. 2016). Composites reinforced by natural fibers or particles are considered one of the most promising structural materials in engineering technologies (Belaadi et al. 2013). The use of vegetable fibers in cement-based composites to produce roofing tiles, prefabricated components, and boards can significantly contribute to the rapid growth of the country's infrastructure (Savastano Junior et al. 2009).

The incorporation of vegetable fibers in fiber-cement presents challenges, mainly due to their dimensional instability, hydrophilic nature, and alkaline degradation caused by the cement matrix. Cementitious materials composed of vegetable fibers degrade primarily due to the high alkalinity of the water contained in the pores of the Portland cement matrix (Rodrigues et al. 2013). Because of this, there is a loss of adhesion at the fiber-cement interface and an increase in cracks, which contributes to the fiber-cement composite decreasing strength and durability (Tonoli et al. 2013).

Accelerated curing of cementitious materials in a high carbon dioxide (CO_2) environment appears to safeguard against these materials' degradation. Accelerated carbonation slows the degradation of vegetable fibers used in cement-based materials by decreasing the pH of the matrix to near 8.3 in fully carbonated areas (Saetta et al. 1993; Filomeno et al. 2020). This decrease in alkalinity renders the cement less aggressive toward the fibers and improves the interface of the composite (Dos Santos et al. 2019). The curing process of cementitious materials under carbonation occurs by the diffusion of CO_2 through the unsaturated pores of the cementitious matrix (Lo et al. 2016; Martins et al. 2018). CO_2 reacts with calcium hydroxide ($Ca(OH)_2$), forming calcium carbonate ($CaCO_3$). In the unsaturated pores of cement, CO_2 is dissolved in water and transformed into acidic carbon dioxide (H_2CO_3), and there is dissociation of HCO_3^- and CO_3^-. At the same time, there is a decoupling of $Ca(OH_2)$ in the ions Ca^{2+} and OH^-, forming $CaCO_3$ (Filomeno et al. 2020). The use of accelerated carbonation as a curing method in cementitious composites reinforced with vegetable fibers showed satisfactory results, with a reduction in the alkalinity of the cement matrix, better fiber matrix adhesion and consequently improvement in mechanical properties (Almeida et al. 2013; Santos et al. 2015; Neves Junior et al. 2019). Vegetable fibers promote improved carbonation due to the high air content (the fibers act an air-entraining admixture), which induces CO_2 penetration becomes easier (Buy et al. 2021). In this way, the results of accelerated carbonation in cementitious composites reinforced with vegetable fibers are related to the species used, and, therefore, research with new species is necessary.

The use of fibers from rubberwood was investigated in the literature, for example, for particleboard production (Faria et al. 2021) and activated carbon (Ma et al. 2019). The rubber tree is native to the Amazon, and Brazil is home to ten of the 11 known species. The rubber tree is a dicot of the genus *Hevea*, which belongs to the family *Euphorbiaceae*, all of which are arboreal species (Lima et al. 2000). In recent years, the area planted with rubber trees in Brazil has increased significantly. In 2005, the planted area was 112,396 ha, which increased to 163,254 ha in 2020 (IBGE 2020). The country's expansion of rubber plantations results in a significant supply of wood from this crop at the end of its rotation (25-30 years), sparking interest in research on this raw material. Despite having good workability characteristics (gluing, driving, drilling, among others) and being easily folded with the use of steam, rubberwood obtained at the end of the latex production cycle is traditionally used as firewood in Brazil (Eufrade Junior et al. 2015).

The purpose of this study was to assess the effect of curing under accelerated carbonation on fiber-cement composites reinforced with rubberwood fibers, concerning the physical, mechanical, and microstructural properties of the extruded composites.

2. Materials and methods

2.1. Obtaining and chemical treatment of rubberwood fibers

To produce fiber-cement composites, rubberwood (clone Pb235) aged 28 years was collected from the campus of the Federal University of Lavras (UFLA) located in the municipality of Lavras in the southern region of Minas Gerais, Brazil; the coordinates are 21°14′45″ S, 44°59′59″ W, and the altitude is 920 m.

After collecting the material, it was ground in a hammer mill to produce sliver-type fibers. Only fibers that passed through the 0.42 mm sieve and remained in the 0.25 mm sieve were used.

Rubberwood fibers were alkaline treated with a solution of NaOH prior to composite production, following a similar procedure described by Suardana et al. (2011). The fibers were placed in a glass beaker with a capacity of 2.5 L NaOH solution was weighed and added to the container at a concentration of 10% by mass, and the beaker was then placed in a heater set to 95 °C for 1 h. The fibers were washed with deionized water until the pH of the fibers' dripping water reached approximately 7.0, followed by drying in an oven at 105 ± 3 °C for 24 h. The mass loss of dry fibers was determined to confirm the efficiency of the treatment.

2.2. Characterization of rubberwood fibers

The characterization of rubberwood fibers was performed by fiber density analysis according to the adaptation of NBR 11941 (ABNT 2003) guidelines by Protásio et al. (2013). Total extractive analysis was carried out in accordance with the NBR 14853 (ABNT 2010) standard, insoluble lignin content according to NBR 7989 (ABNT 2010), ash content according to NBR 13999 (ABNT 2017) and holocellulose by the difference between the other constituents. The moisture content of the fibers (MC) was evaluated using Equation (1).

$$\mathrm{MC} = \frac{(\mathrm{m}_f - \mathrm{m}_i)}{\mathrm{m}_i}.100 \qquad (1)$$

where MC is the moisture content (%), m_f is the saturated mass (g) and m_i is the dry mass of fibers (g).

2.3. Production of fiber-cement composites

Composites were produced with rubberwood fibers treated and without surface treatment in substitution of 0% (named composition control) or 5% in mass to the cement based on a previous study performed by Raabe et al. (2022), 33% agricultural limestone, 1% HPMC, 1% ADVA, and 60% high early-strength cement (Portland cement CPV-ARI, equivalent to ASTM cement type III). Portland cement CPV-ARI was chosen because it lacked additives and was therefore considered less aggressive in terms of mineralization and degradation. Additionally, the high initial strength favors mechanical properties and mitigates the effect of extractives (Bezerra et al. 2006). Grinded limestone was used in place of Portland cement to reduce the cost of the manufacturing process. It is necessary for the composite's stabilization (as it is considered an inert ingredient) and for sustainability reasons (Fonseca et al. 2019). The additives hydroxypropylmethylcellulose (HPMC) and carboxylic polyether (ADVA) were used to aid in the rheology of the mixture and increase the capacity of the cement to retain water, as well as to increase labor productivity and the process of cement stabilization. A proportion of 1.0% of the total mass of each mixing was used for each additive (HPMC and ADVA).

A planetary mixer was used to homogenize the ingredients. Cement, limestone, fibers, and HPMC were first mixed for two minutes at a speed of 140 rpm. After adding the ADVA and water, the mixing process was continued for five minutes at 285 rpm to ensure the uniform distribution of the fibers in the formed mass.

The fiber-cement composite was processed in a helical extruder. The mass was passed by the extruder three times to ensure proper homogenization and orientation of the rubberwood fibers for extrusion (Fig. 1). After molding, the specimens were sealed in plastic bags and stored at high humidity and room temperature for two days, whose procedure was based on Tonoli et al. (2019).

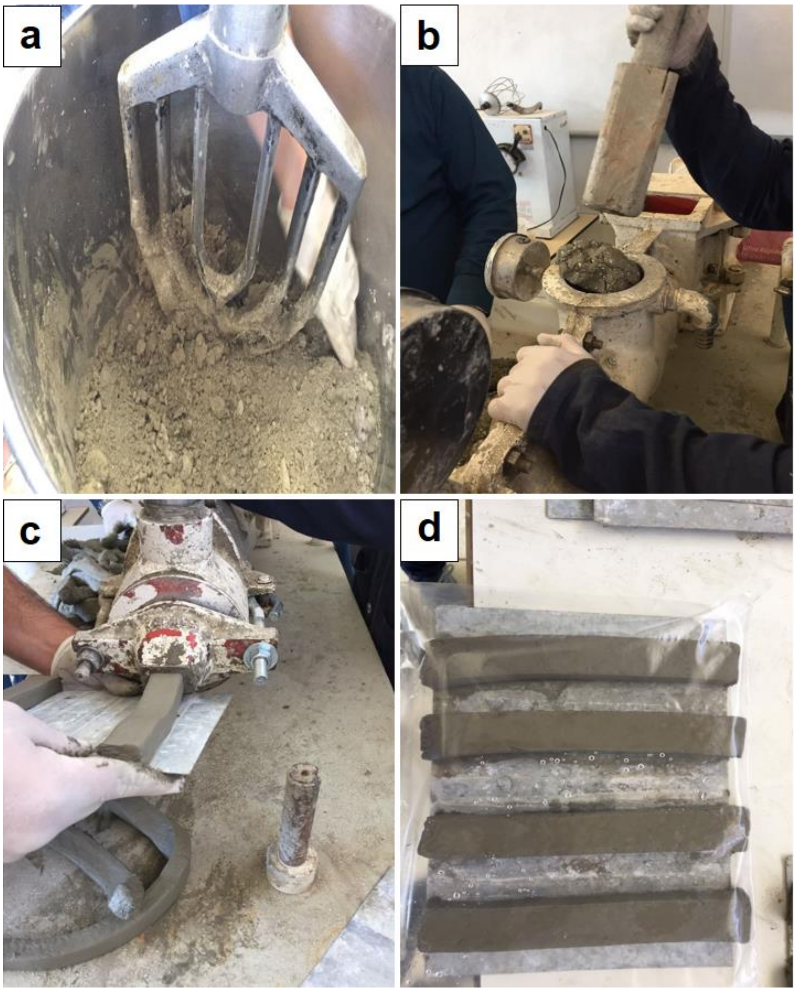


Fig. 1 Extruded fiber-cement molding process. (a) Mixture of materials; (b) Homogenization and orientation of fiber-cement composite; (c) Specimen output through the extruder nozzle; (d) Specimens wrapped in a plastic bag containing water inside for the wet curing period

2.4. Accelerated carbonation

Two days after the extrusion of the specimens, accelerated carbonation was performed using a proprietary methodology in which the autoclave was connected by a hose to an industrial-grade CO_2 cylinder with a purity of 99%. A constant pressure of 0.75 kgf/cm² was maintained, and no temperature was applied. The composites remained in a CO_2-rich environment for 12 h and were subsequently sealed in a saturated condition at a temperature of 25 °C for 26 days to complete the curing cycle (28 days).

The occurrence of carbonation was initially evaluated by titration with a 1% phenolphthalein solution, usually used as a pH indicator. This phenolphthalein solution is applied to the fracture surface of the composite, showing a violet color when in contact with basic elements (pH >7), such as the alkaline products of cement hydration, and when the pH is acidic (pH<7), the material becomes colorless, evidencing the carbonation process.

After a 27-day wet cure period, the extruded composites were cut with a disk saw to achieve the final dimensions of 50 mm × 16 mm × 200 mm (width, thickness, and length, respectively), producing eight specimens for each composition. After 24 h of immersion in water, the specimens were subjected to physical and mechanical tests.

2.5. Thermogravimetric analysis (TGA) and carbonation degree (CD) of the fiber-cement composites

To prove the occurrence of the carbonation process, thermogravimetric analysis was performed, in which the specimens were immersed in isopropyl alcohol for 1 h, following the recommendations described by Snellings et al. (2018). The specimens were dried in an oven at 70 °C for 24 h and kept in sealed plastic bags until the characterization tests. Thermogravimetric analysis (TGA) was recorded with a Q500 TA Instruments using a dynamic nitrogen stream (flow rate = 60 mL/min) at a heating rate of 10 °C/min to 900 °C. The samples (approximately 10 mg) were previously ground to a particle size below 37 μm to reduce the influence of the eventual heterogeneity of the material (Tonoli et al. 2019; Matsushita et al. 2000).

The amount of absorbed CO_2 was determined by the mass loss at temperatures ranging from 550 to 1000 °C and then multiplied by the residual mass after 900 °C, resulting in nonvolatile values expressed as a percentage (Tonoli et al. 2019). The inherent carbonates present in the noncarbonated fiber-cement were deducted, and the carbonation degree (CD) was determined according to Equation (2) (Matsushita et al. 2000).

$$\mathrm{CD}\ (\%) = \frac{(\mathrm{C} - \mathrm{C}_o)}{(\mathrm{C}_{max} - \mathrm{C}_o)} . 100 \qquad (2)$$

where C_{max} is the required CO_2 to react with the available oxides in the cement to form $CaCO_3$ (Equation 3). C is the amount of CO_2 in the fiber-cement composites, and C_0 is the amount of CO_2 in noncarbonated fiber-cement composites (Huntzinger et al. 2009).

$$\mathrm{C}_{\max}(\%) = 0.785(\mathrm{CaO} - 0.56\mathrm{Ca}(\mathrm{CO}_3) - 0.7 * \mathrm{SO}_3) + 1.091\ \mathrm{MgO} + 0.71\mathrm{NaSO} + 0.468\mathrm{K}_2\mathrm{O} \qquad (3)$$

Equation (3) presupposes that the entire mixture of Na_2O, CaO, K_2O, and MgO formed in the cementitious matrix reacts with CO_2 to form carbonates (Huntzinger et al. 2009). It does not include the amount of CaO in the form of $CaCO_3$ and sulfates (Tonoli et al. 2019).

2.6 Physical properties of fiber-cement composites

Water absorption (WA), apparent porosity (AP) and apparent density (AD) tests were performed following the procedures specified by standard C 948-81 (ASTM 2016). Eight specimens with dimensions of 50 mm × 16 mm × 200 mm (width, thickness, and length, respectively) were utilized for each condition. Physical properties were obtained using Equations (4), (5), and (6).

$$\mathrm{WA}\ (\%) = \frac{(\mathrm{m}_f - \mathrm{m}_o)}{\mathrm{m}_o} . 100 \qquad (4)$$

$$\mathrm{AP}\ (\%) = \frac{(\mathrm{m}_f - \mathrm{m}_o)}{(\mathrm{m}_f - \mathrm{m}_i)} . 100 \qquad (5)$$

$$\mathrm{AD}\ (\mathrm{g/cm}^3) = \frac{\mathrm{m}_o}{(\mathrm{m}_f - \mathrm{m}_i)} . \rho \qquad (6)$$

where m_f is the specimen mass after 24 h of immersion in water with a dry surface, m_o is the dry mass of the specimen after 24 h at 105 °C, m_i is the specimen's mass immersed in water and ρ is the density of water (1.0 g/cm^3).

2.7. Mechanical properties of fiber-cement composites

To determine the mean values of modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE) and toughness, static bending tests were performed using a universal testing machine (EMIC DL, Brazil) equipped with a 20 kN load cell. A four-point bending test was set up with a span of 150 mm, and the crosshead speed of the machine was equal to 1.5 mm/min. Eight specimens were used for each composition studied, and the procedures to perform the mechanical tests were based on the procedures of Rilem (1984).

Mechanical properties were obtained using Equations (7), (8), (9) and (10), as described by Tonoli et al. (2009).

$$\mathrm{MOR\ (MPa)} = \frac{(P_{max}.\mathrm{L})}{(\mathrm{b.h^2})} \tag{7}$$

$$\mathrm{LOP\ (MPa)} = \frac{(P_{lop}.\mathrm{L})}{(\mathrm{b.h^2})} \tag{8}$$

$$\mathrm{MOE\ (MPa)} = \frac{(276\ .\ \mathrm{L^3})}{(1296\ .\ \mathrm{b}\ .\ \mathrm{h^3})}\ .\,\mathrm{m} \tag{9}$$

$$\mathrm{Toughness\ (N.mm/mm^3)} = \frac{(\mathrm{Energy})}{(\mathrm{b.h})} \tag{10}$$

where P_{max} is the maximum load supported by the specimen; P_{lop} is the load at the upper point of the linear portion of the load–deflection curve; L is the span equal to 150 mm; b is the width of the specimen equal to 50 mm; h is the height of the specimen equal to 16 mm; and m is the tangent of the slope angle of the straight line corresponding to the elastic region of the load-deflection curve. Toughness is the energy absorbed during the bending test divided by the specimen cross-sectional area.

2.8. The effect of rubberwood fibers on the hydration of cement paste

The method used to determine the inhibition caused by rubberwood fibers on cement hydration was modified from Hofstrand et al. (1984). Fifteen g of dry rubberwood fibers, 200 g of Portland cement CPV-ARI/Plus, and 90 mL of water were combined for this test. These materials were homogenized for 5 min and placed in Styrofoam boxes lined with aluminum foil to minimize temperature loss to the environment. Each Styrofoam box contained a temperature sensor connected to a Datalogger, which recorded data at one-second intervals over 24 h. For this test, a PicoLog data acquisition system, model TC-08, connected to a computer and type k thermocouples was used.

The box and data acquisition system were housed in an acclimatized room set to a temperature of 20 ± 1 °C and relative humidity of 60 ± 5% to eliminate external interferences. Three replicates using rubberwood fibers were performed, in which the fibers were retained between 2.4 - 4.9 mm sieves. The inhibition index was calculated using Equation (11) (Okino et al. 2004):

$$\mathrm{I} = \left[\frac{(\mathrm{TC} - \mathrm{TS})}{TC}\right]\left[\frac{(\mathrm{HS} - \mathrm{HC})}{HC}\right]\left[\frac{(\mathrm{SC} - \mathrm{SS})}{SC}\right] \times 100 \qquad (11)$$

where I is the cement curing hydration index (%), TC is the maximum temperature of the cement paste (°C); TS is the maximum temperature of the rubberwood fiber/cement paste (°C), HC is the time to reach the maximum cement hydration temperature in the cement paste (h), HS is the time to reach the maximum temperature of the cement hydration mixture in the rubberwood fiber-cement paste (h), SC is the maximum temperature increment of the curve in the cement paste (°C/h), and SS is the maximum temperature increment of the curve in the rubberwood fiber-cement paste (°C/h).

The effect on cement curing inhibition is classified according to Okino et al. (2004) into four groups: low inhibition when the hydration index is less than 10%, moderate inhibition when it is in the range 10 - 50%, high inhibition for the range 50 – 100% and extreme inhibition for the hydration index greater than 100%.

2.9. Microstructural analysis of rubberwood fibers and analysis of the rupture surface of the composites

The morphological aspects of rubberwood fibers and the rupture surface of the composites were investigated by scanning electron microscopy (SEM) with an LEO EVO 40 XPV instrument. The specimens were covered with a gold layer in an evaporator and analyzed in an SEM operating at 20 kV to obtain the electron micrographs.

2.10. Air permeability tests

Permeability parameters were determined using experimental data and fitting of Forchheimer's equation (Equation 12), a well-established empirical relationship that expresses the parabolic dependence of pressure drop (ΔP) with the resulting superficial or face velocity (v_s) of the fluid through the medium (Innocentini et al. 2017; Innocentini et al. 2019; Innocentini et al. 2005; Fioroni et al. 2020; Corradetti et al. 2016).

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s + \frac{\rho}{k_2} v_s^2 \qquad (12)$$

where L denotes the medium length or thickness along the macroscopic flow direction and μ and ρ denote the fluid's viscosity and density, respectively. The parameters k_1 and k_2 are referred to as Darcian and non-Darcian permeability coefficients, respectively. These coefficients are dependent only on the porous structure and are used in Equation (12) to balance the effects of viscous and inertial losses on the total pressure drop. For compressible flow, ΔP in Equation (12) must be determined using Equation (13).

$$\Delta P = \frac{{P_i}^2 - {P_o}^2}{2P} \qquad (13)$$

where P_i and P_o are the inlet and outlet absolute gas pressures, respectively. P denotes the absolute pressure at which v_s, μ and ρ are measured or calculated (in this work $P = P_o$).

The permeability coefficients k_1 and k_2 were determined experimentally using a laboratory-built device, with testing conducted in a steady-state regime with dry airflow at ambient temperature ($T = 23\text{-}29$ °C, $P_{atm} = 94.7$ kPa, $\mu = 1.88 \times 10^{-5}$ Pa.s; $\rho = 1.1$ kg/m^3) on three specimens of each batch. Further details of the method and the experimental setup are given elsewhere (Innocentini et al. 2017; Innocentini et al. 2019; Innocentini et al. 2005) and illustrated in Fig. 2.

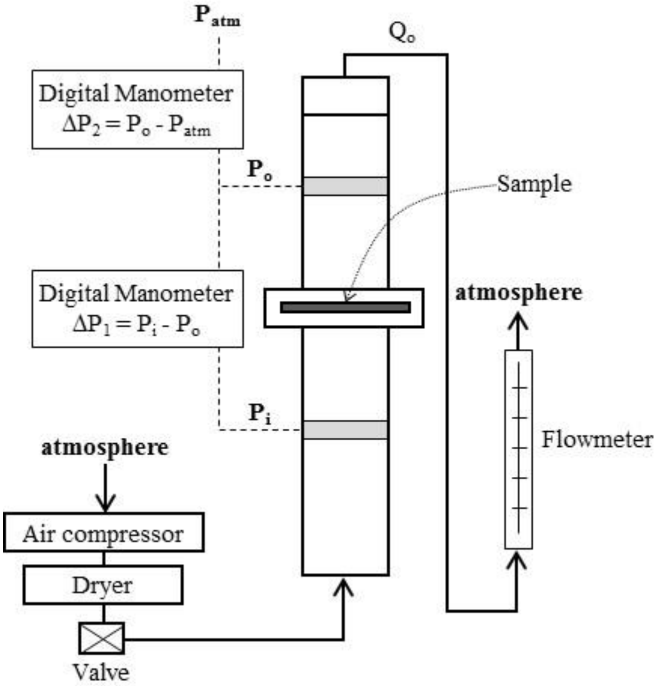


Fig. 2 Scheme of the airflow permeation apparatus

2.11. Statistical analysis

To evaluate the variations in the physical and mechanical properties of the fiber-cement produced in this work, they were submitted to analysis of variance and Tukey's test at 5% significance.

3. Results and discussion

3.1 Rubberwood fiber properties

The rubberwood fibers showed a value of 0.544 ± 0.01 g/cm³ for basic density, a result slightly lower than that observed by Faria et al. (2021), in which the authors obtained a basic density of 0.559 g/cm³. The density of rubberwood fibers influences the extrudability of the mixture during the extrusion process. Even for low fiber insertion contents, a large volume of the composition is obtained, which may cause loss of extrudability because the fiber intertwines. Moreover, the relation between low density and problems for extruding mixtures with a high volume of fibers may occur due to the difficulty in producing a homogeneous mixture, resulting in fiber agglomeration and clogging of the extruder die (Fonseca et al. 2019).

The chemical constituents present in vegetable fibers are related to a certain level of adhesion with the cement matrix, depending on their content, since they affect reactivity, interfering with chemical compatibility and the curing process; consequently, the mechanical properties of the composites produced are affected (Moslemi 1984; Alberto et al. 2000). Table 1 presents the chemical constituents present in rubberwood fibers.

Table 1. Characteristics of rubberwood fibers (%, dry basis).

Chemical constituents	Untreated	Alkali treated	Difference[1]
Total extractives (%)	9.97 ± 0.98*	3.25 ± 0.46*	6.72
Insoluble lignin (%)	25.84 ± 1.67*	17.19 ± 0.64*	11.65
Holocelluloses (%)	62.23 ± 0.89*	78.09 ± 2.78*	-15.86
Ash (%)	1.96 ± 0.07*	1.47 ± 0.06*	0.49

*Standard deviation. (1) Untreated - Alkali treated.

There was a considerable reduction in the levels of chemical components present in the fibers of rubberwood, except for the content of holocellulose. The increase in the holocellulose content after the alkaline treatment is due to the removal of other chemical components; thus, the fibers have a higher cellulose content in their composition. Alkaline treatment is a key step to remove lignin, hemicelluloses, and extractives from the surface of the fibers, increasing the interfacial bond between rubberwood fibers and the cement matrix (Ferraz et al. 2012). NaOH treatment is effective in increasing fiber surface stability, reactivity, and surface roughness (Santos et al. 2018).

Extractives are chemical components found occasionally in lignocellulosic materials; therefore, they are not part of cell wall formation. Extractives are considered one of the main components that cause the selectivity of the fiber to be used in cementitious composites (Castro 2021). Yasuda et al. (2002) emphasize that the inhibition of cement curing is more related to the levels of extractives present in plant fibers, while Hachmi and Moslemi (1989) state that the types of extractives have different inhibitory capacities.

The alkaline treatment, despite being satisfactory, reduced the extractive content by 67.40% and was not able to remove all the extractives present on the fiber surface. This fact can mainly be attributed to the removal method and, as observed in the present study, is similar to that reported by Sivasubramanian et al. (2021), with a total extractive content of 2% in *Acacia caesia* bark fibers after alkaline treatment with NaOH.

Lignin, together with cellulose and hemicelluloses (quantified by the holocellulose content), are fundamental components present in the cell wall of plant fibers. Lignin acts as a binding element, promoting stiffness; thus, higher levels of this constituent are desired, resulting in a composite with better mechanical properties. Despite the fact that holocellulose has no inhibitory effect on cement paste, the major difficulty arises from cellulose mineralization, a phenomenon caused by the presence of cement hydration products (ettringite and calcium hydroxide) in the lumen of plant fibers. While this is advantageous during composite formation, it can result in an increase in the adhesion between the organic and inorganic materials. On the other hand, mineralization weakens the fibers, impairing the composite's properties over time (Tonoli et al. 2009).

In terms of ash content, this unintentional component can influence the cement hydration process through elements such as calcium and potassium. As a result, despite being sensitive to the decrease after alkaline treatment, it tends to aid in the adhesion between the fibers and the cement matrix.

Based on Equation (1), an MC of 454.19% was calculated for rubberwood fibers. In this way, based on the physical and chemical characteristics, when inserting the fibers into the cementitious composite, they absorb the water, increasing the viscosity of the flow in the system, which may cause porosity in regions where the fibers do not disperse (Teixeira et al. 2019). In this way, the water sequestered from the system is necessary to facilitate the movement of the fibers.

3.2 Physical properties of composites

The surface treatment with alkaline solution as well as the curing under accelerated carbonation obtained statistically significant results for the physical properties of apparent density, water absorption and apparent porosity. Fig. 3 shows the apparent density results for the composites produced, in which those subjected to curing under carbonation had the highest average values, regardless of the fiber surface treatment condition. Greater densification of the carbonated cementitious composite is expected since calcium carbonate has a higher density in relation to calcium hydroxide (Almeida et al. 2010).

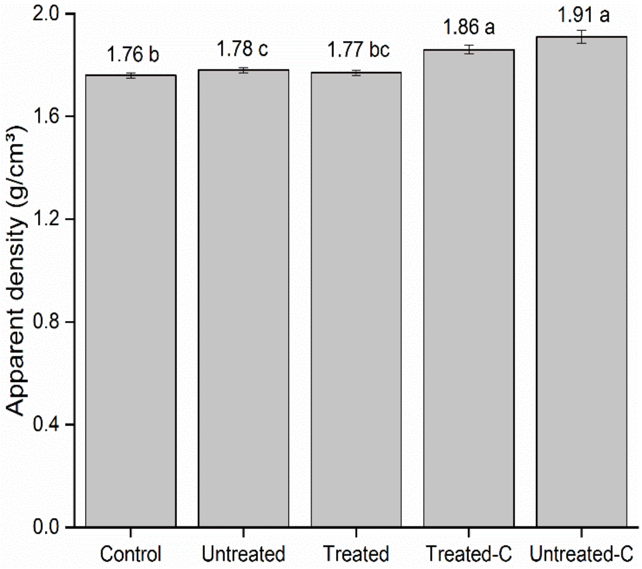


Fig. 3 Apparent density of control and fiber-cement produced with NaOH-treated and untreated rubberwood fibers subjected to accelerated carbonation (Treated-C) and not subjected to accelerated carbonation (Untreated-C). Averages followed by the same letter (a, b, and c) indicate no significant difference according to the Tukey test ($p > 0.05$)

Fig. 3 show that the composites produced with treated fibers presented values statistically equal to the control composition; that is, even though the plant fibers have a much lower density than the cement matrix, the final density of the composite did not change. For composites produced with fibers without surface treatment, the apparent density was greater than the control composition, a behavior explained by the presence of hemicelluloses in the cell wall of plant fibers, in which this constituent, being highly hydrophilic, sequestered and internally kept the available water from the cement paste.

The water absorption levels for the studied composites are shown in Fig. 4. Compared to the control, the specimens extruded with insertion of rubberwood fibers without surface treatment increased the levels of water absorption, while the composites produced with fibers subjected to alkaline treatment showed no significant differences.

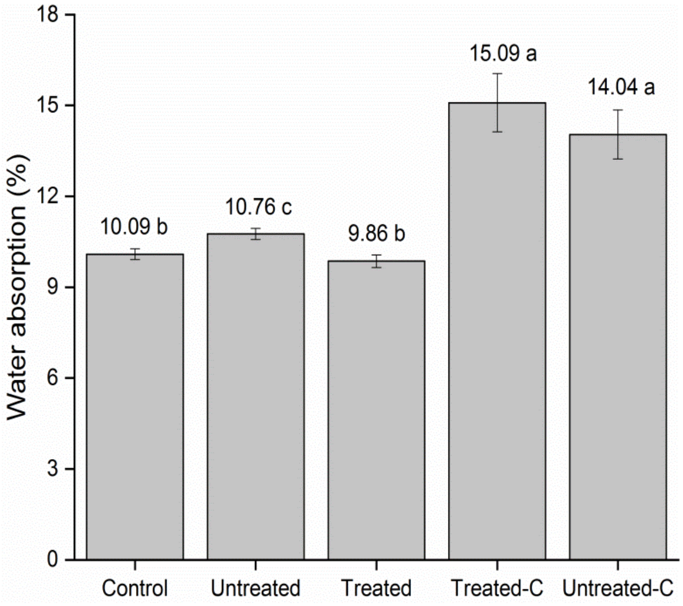


Fig. 4 Water absorption of control and fiber-cement produced with NaOH-treated and untreated rubberwood fibers subjected to accelerated carbonation (Treated-C) and not subjected to accelerated carbonation (Untreated-C). Averages followed by the same letter (a, b, and c) indicate no significant difference according to the Tukey test ($p > 0.05$)

Fibers that did not receive surface treatment had a higher proportion of hydrophilic structural carbohydrates (hemicelluloses), resulting in composites with higher levels of water absorption.

In relation to composites produced with curing under accelerated carbonation, a significant increase in water absorption levels was evidenced when compared to those without the effect of accelerated carbonation. The increase in water absorption is directly related to the increase in apparent porosity (Fig. 5), in which, due to greater apparent porosity, a greater amount of rubberwood fibers were more exposed. Accelerated carbonation significantly affected the apparent porosity levels for the composites under study.

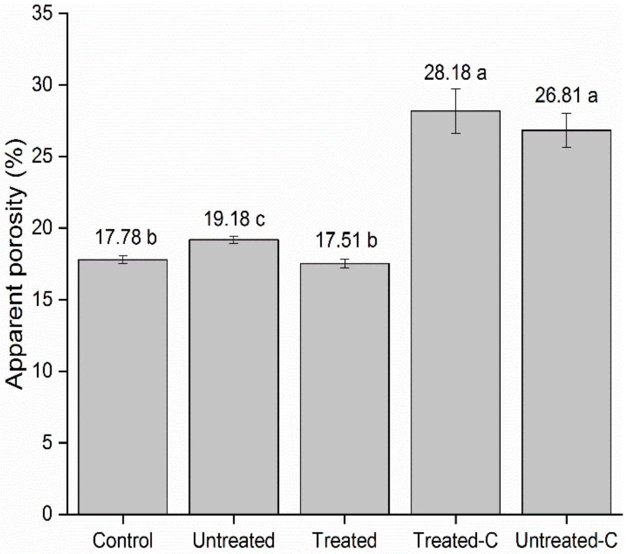


Fig. 5 Apparent porosity of control and fiber-cement produced with NaOH-treated and untreated rubberwood fibers subjected to accelerated carbonation (Treated-C) and not subjected to accelerated carbonation (Untreated-C). Averages followed by the same letter (a, b, and c) indicate no significant difference according to the Tukey test ($p > 0.05$)

The results for apparent porosity show that the composites produced with treated fibers presented values statistically equal to the cement paste without the addition of fibers; however, lower than the composites produced with the insertion of untreated fibers and those subjected to curing in a CO_2-rich environment. This result can be explained by the inhibition that occurred between the cement paste and the fibers without surface treatment (Fig. 6), in which, due to the inhibition, pores are observed around the fibers, thus resulting in the final porosity of the composite. Therefore, porosity is expected, since during the hydration of the cement matrix, there is the typical formation of pores, calcium silicate hydrate (C-S-H), regardless of the presence of fibers (Mehta and Monteiro 2006).

The increase in apparent porosity values for composites produced with curing subjected to accelerated carbonation is due to the dissolution reaction of CO_2 in water, in which it is transformed into carbonic acid (H_2CO_3) in the unsaturated pores of the cement matrix, resulting in dissociation of ions HCO_3^- and CO_3^- (Equations 14 and 15). At the same time, the dissociation of $Ca(OH)_2$ into ions Ca^{2+} and OH^- appears (Equation 16), which finally forms $CaCO_3$ (Equation 17). However, only part of the CO_2 dissolved in water forms $CaCO_3$; thus, as the CO_2 dissolved in water is not transformed, there is a water-rich environment in contact with the specimens. The porosity is higher because the rubberwood fibers absorb water from the cement microstructure, since the concentration of water surrounding it is higher than that inside the fiber. Subsequently, the fiber loses this water to the system, reducing its size and damaging the fiber-matrix interface, causing microcracks and the incidence of large pores (~10 mm) inside the hardened paste (Teixeira et al. 2019).

$$CO_2\ (g) + H_2O\ (l) \rightarrow H^+\ (aq) + HCO_3^-\ (aq) \tag{14}$$

$$HCO_3^-\ (aq) \rightarrow H^+\ (aq) + CO_3^{2-}\ (aq) \tag{15}$$

$$Ca(OH)_2\ (aq) \rightarrow Ca^{2+}\ (aq) + 2(OH)^-\ (aq) \tag{16}$$

$$Ca^{2+}\ (aq) + CO_2^{3-}\ (aq) \rightarrow CaCO_3\ (s) \tag{17}$$

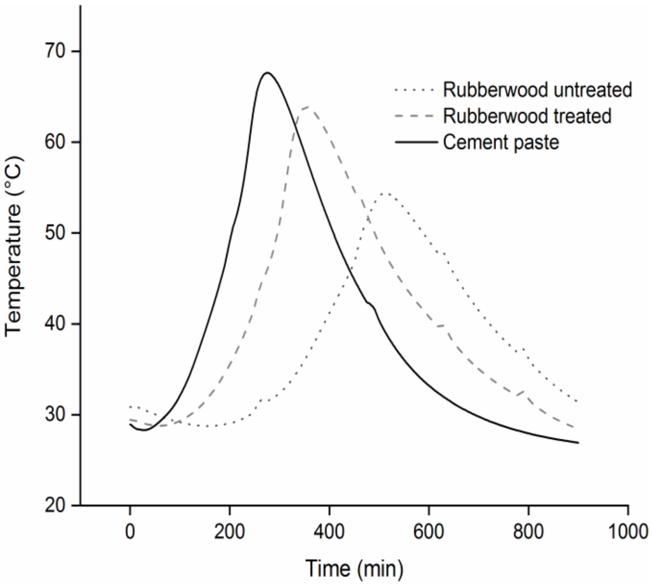


Fig. 6 Hydration curve of cement paste and untreated and treated rubberwood-cement paste

The cement paste containing untreated fibers of rubberwood calculated by Equation (10) presented a hydration index of 8.64%, while the cement paste containing treated fibers of rubberwood presented a hydration index of 0.04%. Based on Okino et al. (2004), both can be classified as low inhibition.

The extractives present on the surface of the fibers caused inhibition of cement hydration, in which there was a longer time to carry out the hydration of the cement. In addition, the maximum temperature reached was 54.38 °C, while the cement paste reached the maximum temperature of 67.63 °C. This difference is a result of the pectin present in untreated fibers, where it can be fixed to calcium, preventing the formation of hydrated calcium silicate structures (Pacheco-Torgal and Jalali 2011).

3.3. Mechanical properties of composites

The typical curve of normal bending stress and specific deformation (Fig. 7) presents the mechanical behavior for the composites produced.

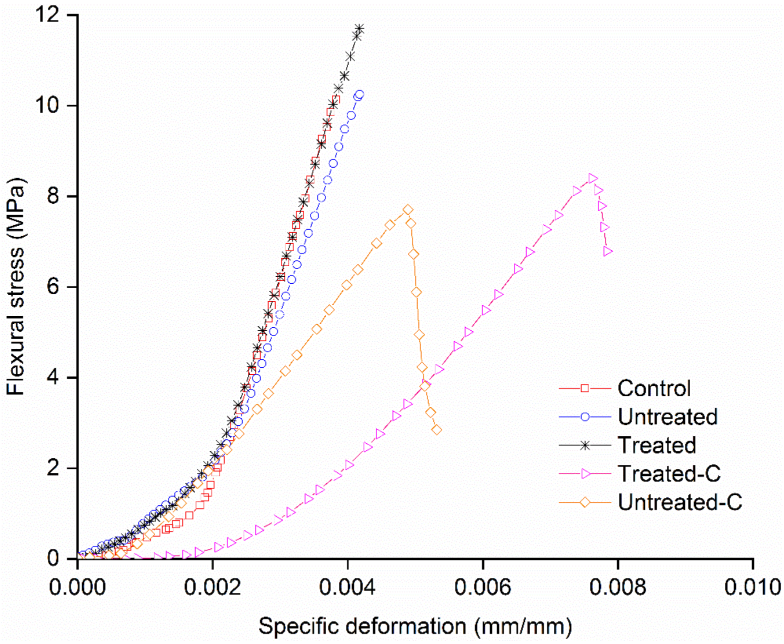
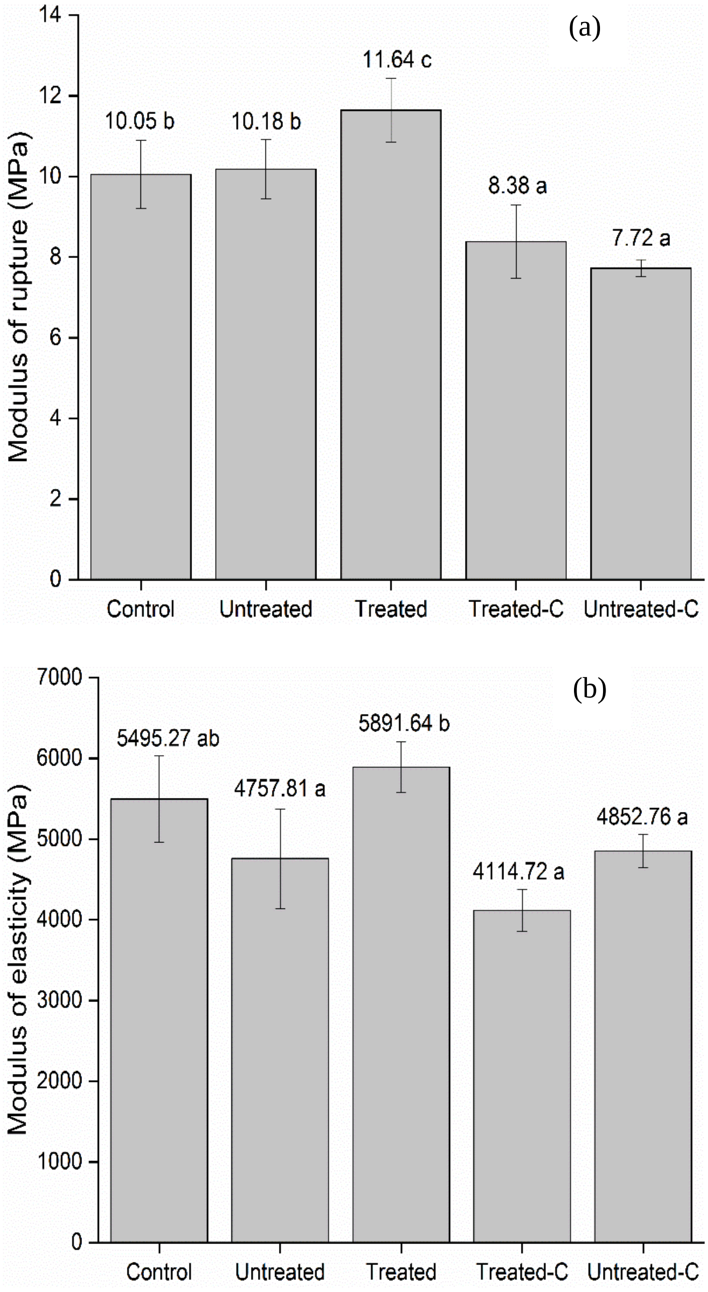


Fig. 7 Normal bending stress vs. specific deformation of fiber-cement composites

The composites produced with rubberwood fibers treated with an alkaline solution of NaOH presented the highest average values of maximum normal stress, significantly superior to the composites produced without the addition of fibers. For composites subjected to curing under accelerated carbonation, the values of specific deformation were significantly higher in relation to the other composites.

Based on the experimental procedures applied to the eight specimens under the static bending test at four points, the mean values and standard deviations for the modulus of rupture, modulus of elasticity, and limit of proportionality were calculated, and the results are shown in Fig. 8.



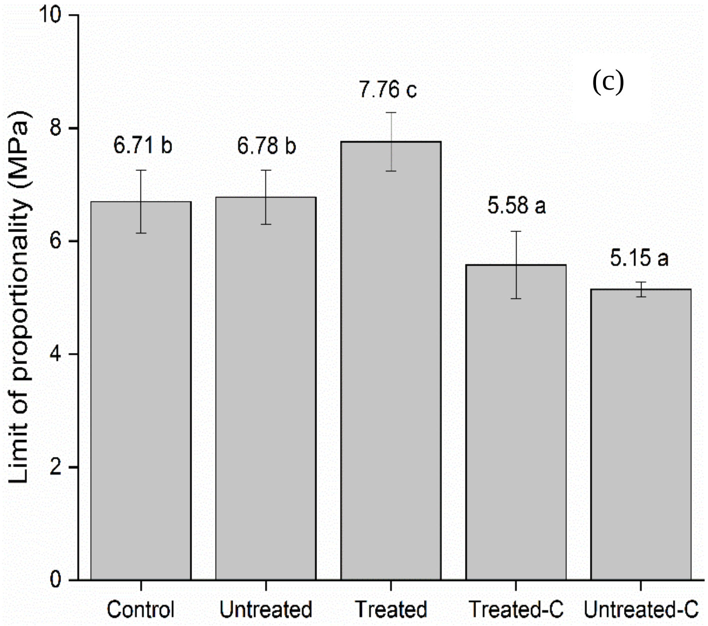


Fig. 8 Static bending testing of fiber-cement specimens produced with NaOH-treated and untreated rubberwood fibers subjected to accelerated carbonation (Treated-C and Untreated-C). a) Modulus of rupture; b) Modulus of elasticity; c) Limit of proportionality. C: curing under carbonation. Averages followed by the same letter (a, b, and c) indicate no significant difference according to the Tukey test ($p > 0.05$)

The composites produced with treated rubberwood fibers showed the highest average values for the modulus of rupture, which were significantly different from the other compositions studied (Fig. 8a). The same behavior was observed for limit of proportionality (Fig. 8c). For modulus of elasticity (Fig. 8b), it was observed that all composites produced presented values statistically equal to the control composition, that is, the stiffness of the composite was maintained. Therefore, a reduction in the average values obtained for the composites is observed, in which the modulus of elasticity decreased as a consequence of a greater number of voids in the composite, which can be seen compared to the results obtained for the apparent porosity of the mixture (Fig. 5). Additionally, fibers have a lower modulus of elasticity than cement paste.

Regarding toughness, curing under accelerated carbonation resulted in a significant increase in property for both composites produced with treated fibers and without surface treatment (Fig. 9). For materials that exhibit brittle rupture behavior, such as ceramic materials, toughness is a property of paramount importance given that the higher its value is, the greater the resistance to crack propagation (Correia et al. 2015).

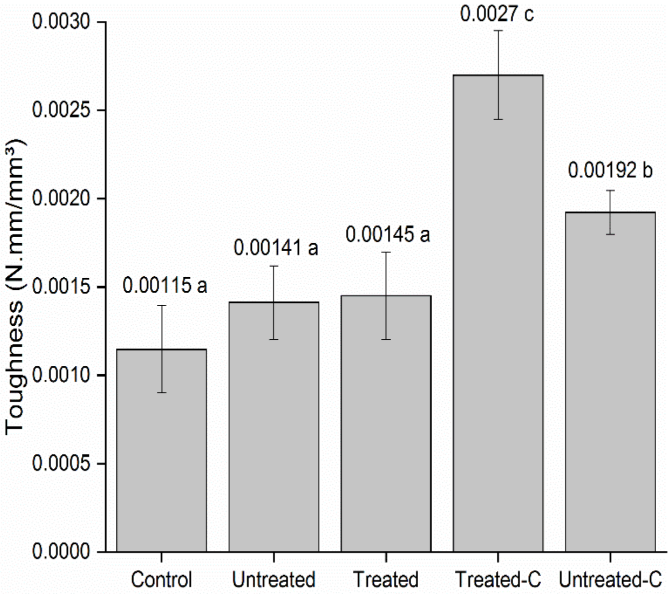


Fig. 9 Toughness of fiber-cement specimens produced with NaOH-treated and untreated rubberwood fibers subjected to accelerated carbonation (Treated-C and Untreated-C). C: curing under carbonation. Averages followed by the same letter (a, b, and c) indicate no significant difference according to the Tukey test ($p > 0.05$)

The results of this research suggest that the toughening mechanisms were better preserved in the fracture process of the carbonated composites, such as pull-out and bridging forces. It is essentially associated with two parameters: interfacial fiber-matrix bonding strength and degradation of the fibers in the cement matrix (Santos et al. 2015). This behavior is verified in the electron micrographs shown in Fig. 10, in which rubberwood fibers are preserved on the surface of composites subjected to accelerated carbonation curing.

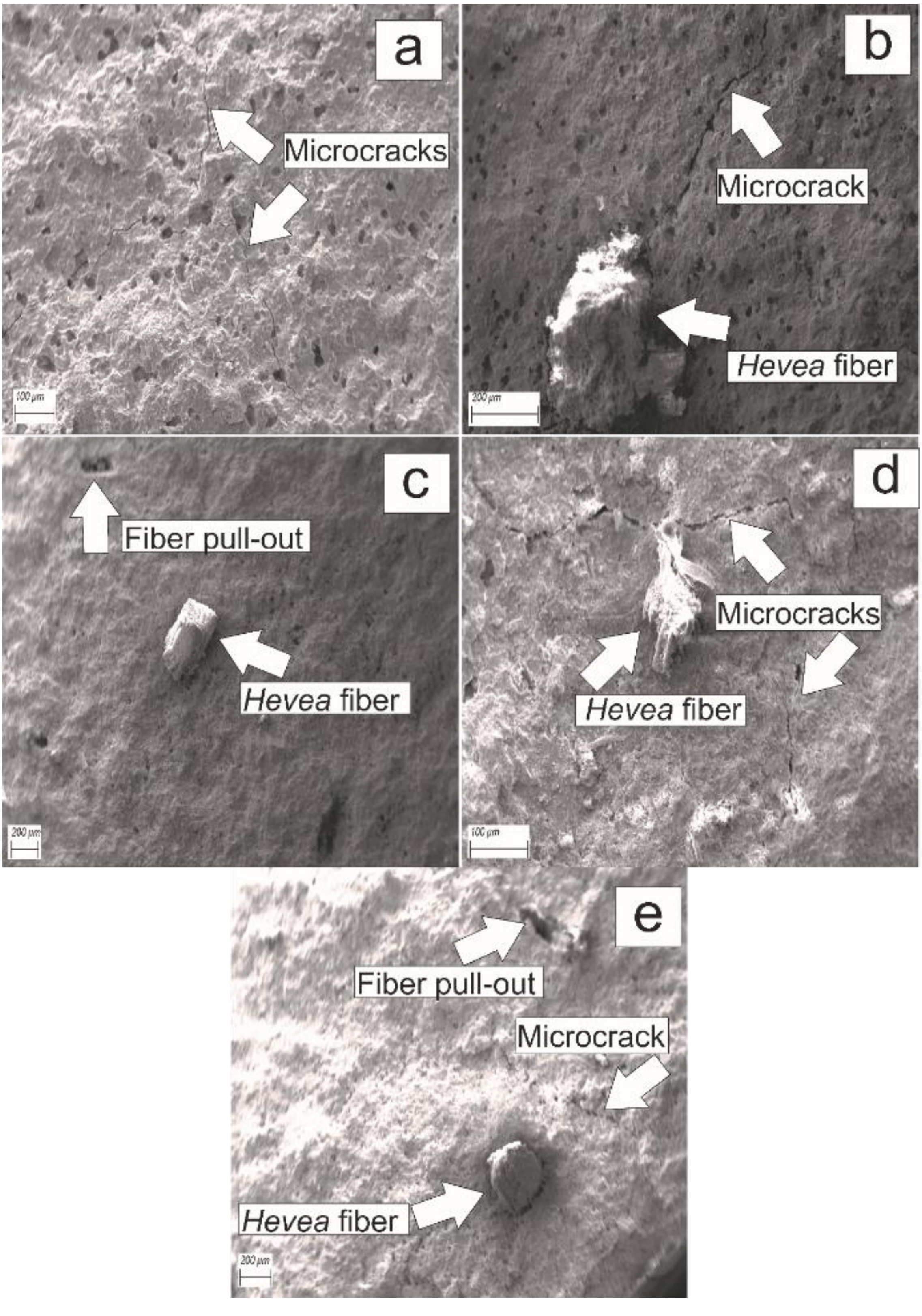


Fig. 10 Electron micrographs obtained from the surface of cement mixture extruded without and with rubberwood fibers, a) control composition; b) fiber-cement untreated; c) fiber-

cement treated; d) fiber-cement treated subjected to accelerated carbonation; e) fiber-cement untreated subjected to accelerated carbonation

Based on Fig. 10, microcracks are observed at various locations in the fiber composites and in the control composition. The fibers present in the cement matrix act as a barrier to the propagation of the crack, in which it needs to bypass the fiber to propagate, thus consuming more energy (Carter and Norton 2013). In addition, the presence of porosity around the fibers that did not undergo alkaline treatment was observed (Fig. 10b and Fig. 10e). As a result of the normal bending stress, several fibers were pulled (fiber pull-out) from the cementitious matrix; in these cases, the strength between the fibers and matrix was not enough to guarantee the connection between the reinforcement and matrix (Fig. 10c and Fig. 10e).

3.4. Effect of accelerated carbonation on the chemical properties of fiber-cement

A 1% phenolphthalein solution was used as an indicator of the pH of the fiber-cement extruded. Fig. 11 shows images of fiber-cement after curing under accelerated carbonation and the presence of carbonation evidenced by the phenolphthalein solution.

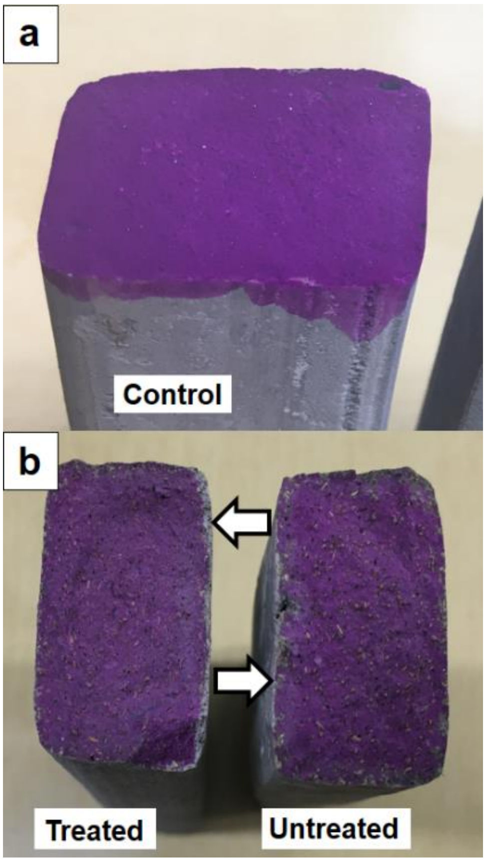


Fig. 11 Occurrence of carbonation in extruded fiber-cement specimens. a) Control; b) treated and untreated specimens with curing under carbonation. Arrows indicate the side surface of specimens with a thin layer unreacted with phenolphthalein

The control specimen, in which it was not subjected to accelerated carbonation curing, showed a violet color throughout the cross-sectional area, while the specimens subjected to accelerated carbonation showed a narrow thin on the side surface with no such coloration, thus indicating a low depth reached by the accelerated carbonation. When there is contact with basic elements ($pH>7$), such as the alkaline products of cement hydration, the phenolphthalein solution becomes violet and colorless when the pH is acidic ($pH <7$).

The pH value of the cementitious matrix is very alkaline, usually greater than 13. This high alkalinity occurs due to the formation of calcium hydroxide $Ca(OH)_2$ during the cement hydration process. However, this calcium hydroxide can combine with carbon dioxide (CO_2)

to form calcium carbonate ($CaCO_3$), a phenomenon known as carbonation (Dos Santos et al. 2019). In this way, the matrix becomes less alkaline with a reduction in the pH value, decreasing to values below 9, making the environment less aggressive for rubberwood fibers.

Fengel and Wegener (1984) reported that the more ions from the cementitious phases are reprecipitated on the fiber walls and inside their internal cavities (lumens), the greater the process of degradation of the lignocellulosic material structure by breaking intermolecular bonds. This process is called fiber mineralization and is therefore detrimental to the matrix properties (Tonoli et al. 2019).

Thus, accelerated carbonation is shown to be an effective alternative to make this environment less aggressive to plant fiber by reducing the alkalinity of the cement matrix. Some studies have even suggested that carbonation curing could replace steam curing and autoclaving under applicable environmental conditions (Wang et al. 2019).

The analysis with phenolphthalein is qualitative (visual), and to prove that the carbonation process occurred, thermogravimetric analysis was performed. Fig. 12 shows the mass loss (TGA) - solid line - and the differential weight loss (DTG) - dotted line - of the composites produced for the different fiber conditions. The marked loss of mass between 105 and 150 °C evident in the curve indicates the dehydration of calcium silicate hydrate (C-S-H) and calcium sulfoaluminates promoted by accelerated carbonation with the adsorption of CO_2 by the cement matrix under different curing conditions (Almeida et al. 2013).

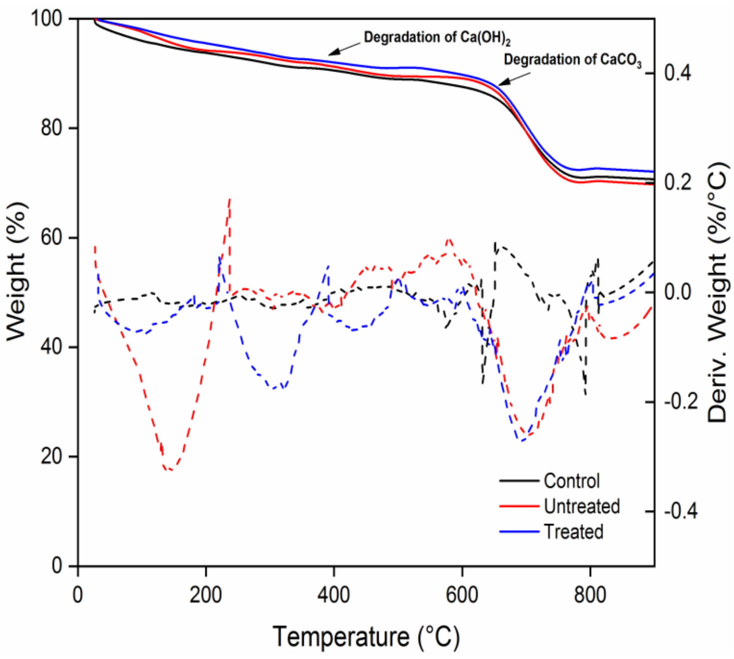


Fig. 12 Thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) curves for composites produced with rubberwood fiber with NaOH treatment and without NaOH treatment and control composition. Continuous line: TGA; dashed line: DTG

The reduction in the mass of the samples for the temperature range between 200 and 500 °C is related to the degradation of rubberwood fibers (Tahir et al. 2019), and greater mass loss was observed for fibers without alkaline treatment, due to lower temperature of thermal degradation of extractives (Floch et al. 2015). This fact is justified since, in this case, the fibers were present in a cementitious matrix, and as the treated fibers showed better adhesion with the matrix, their degradation was hampered.

At approximately 400 to 500 °C, the degradation of $Ca(OH)_2$ occurs, and it is visible that the mass decreases more quickly for the compositions that underwent the accelerated carbonation process compared to the control sample, which was not subjected to the carbonation process, as verified by Santos et al. (2015), Tonoli et al. (2019) and Fioroni et al. (2020). Thus, although carbonation occurs naturally in cement-based materials, due to the reaction between atmospheric CO_2 (0.04% concentration) and the cement matrix, the natural reaction is very slow (Tonoli et al. 2019); therefore, it is possible to prematurely decrease the alkalinity of this matrix (which causes degradation of the lignocellulosic material) when accelerated carbonation is applied to fiber-cement.

Accelerated carbonation increased the mass loss between 600 and 750 °C, related to the decomposition of poorly crystallized $CaCO_3$, and in the range between 750 and 850 °C, related to the decomposition of well-crystallized $CaCO_3$, as also reported by Santos et al. (2019) and Filomeno et al. (2020).

Fig. 13 shows the reaction of carbon dioxide with the products of the hydration reaction, which causes the conversion of unstable $Ca(OH)_2$ into stable $CaCO_3$, as well as the degree of carbonation (CD%), which expresses the theoretical maximum (47.59%) of possible carbonation of the hydrated products with the CPV-ARI cement used in the fiber-cement extrusion process.

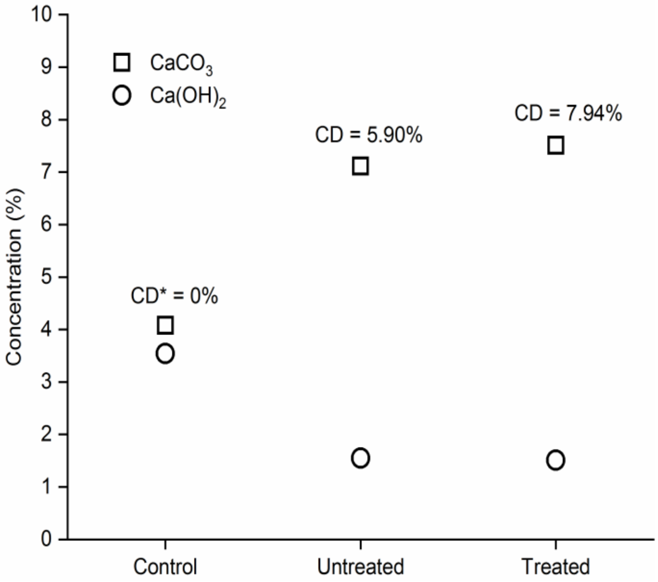


Fig. 13 Conversion of unstable $Ca(OH)_2$ to stable $CaCO_3$ and degree of carbonation (CD%) for composites produced with rubberwood fiber with NaOH treatment and without NaOH treatment and control mixture. (*) no accelerated carbonation

When the mixture is subjected to accelerated carbonation, part of the calcium hydroxide present is consumed in reaction to the hydration products. These results corroborate that there was an accelerated carbonation process, and the C-S-H gel was progressively decalcified and converted into $CaCO_3$ and S-H (silicate hydrates) (Bertos et al. 2004).

A higher efficiency of accelerated carbonation is related to lower moisture content and a reasonable pore volume to optimize the carbonation process (ISO 5267-1 1999). In this way, the apparent porosity of the composites produced before the accelerated carbonation process together with the insertion of rubberwood fibers caused undesirable effects in the process. Another option for this behavior is the method for performing accelerated carbonation. In this work, our own methodology was used, in which there was only control of the flow of CO_2 to the interior, without controlling for temperature and relative humidity. The choice of temperature is essential for greater process efficiency. It is reported that temperatures close to 60 °C increase the leaching of Ca^{2+} ions from the cement and consequently increase the absorption of CO_2 (Zhang and Scherer 2011).

3.5 Air permeability of composites

The samples for air permeability tests were named CS (control), HSC (composites with treated fibers), HST (composites with untreated fibers), H5C (composites with treated fibers subjected to carbonation), and H5ST (composites with untreated fibers subjected to carbonation). The permeability coefficients k_1 and k_2 were obtained from the fitted data and are given in Fig. 14.

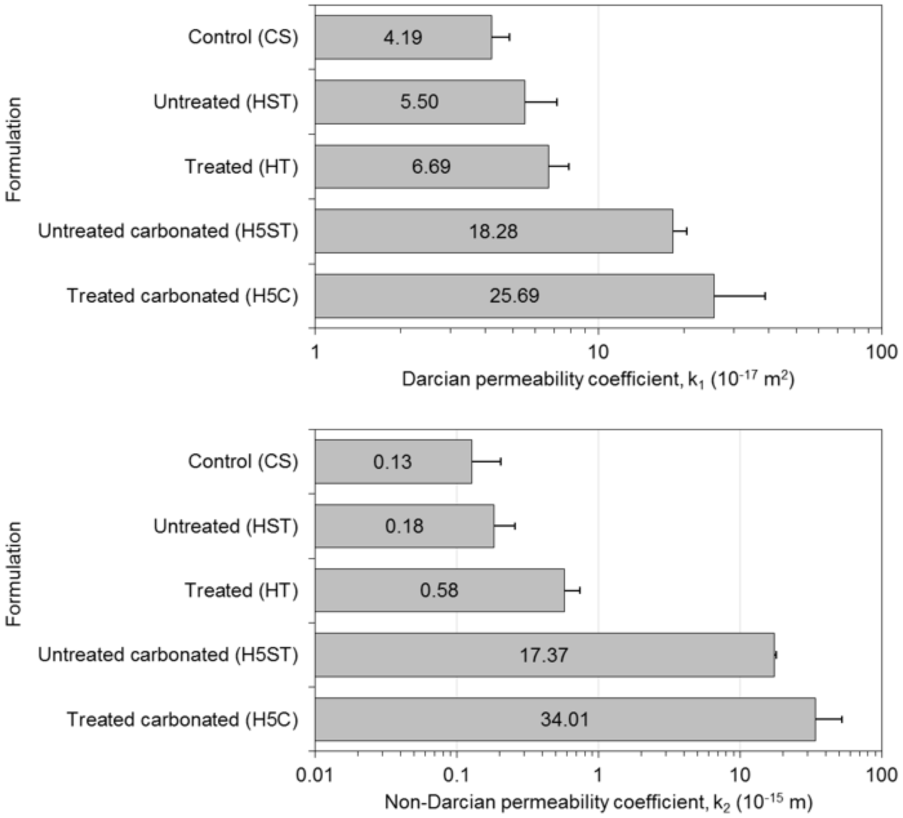


Fig. 14 Permeability coefficients of tested samples (average and deviation)

Increased apparent porosity had a significant effect on the mechanical properties of the composites, decreasing MOR, MOE, and LOP values for composites subjected to accelerated carbonation. As illustrated in Fig. 14, the accelerated carbonation increased the permeability coefficients k_1 and k_2. This behavior is explicable because the primary permeation pathway is located at the fiber-matrix interface (Fig. 10), not within the matrix (Fioroni et al. 2020). As a result of the accelerated carbonation, the interconnections at the fiber-matrix interfaces become larger, increasing permeability, as observed for apparent porosity (Fig. 5).

The produced composites can have their permeability level compared to other cement-based and porous ceramic materials described in the literature. The map shown in Fig. 15, proposed by Innocentini et al. (2005), gathers hundreds of experimental k_1 and k_2 values from the literature and classifies the permeability level of a wide range of porous materials in different applications (Innocentini et al. 2017; Innocentini et al. 2019; Innocentini et al. 2005; Fioroni et al. 2020; Corradetti et al. 2016).

The map also reveals an unequivocal relationship between k_1 and k_2, regardless of the type of material.

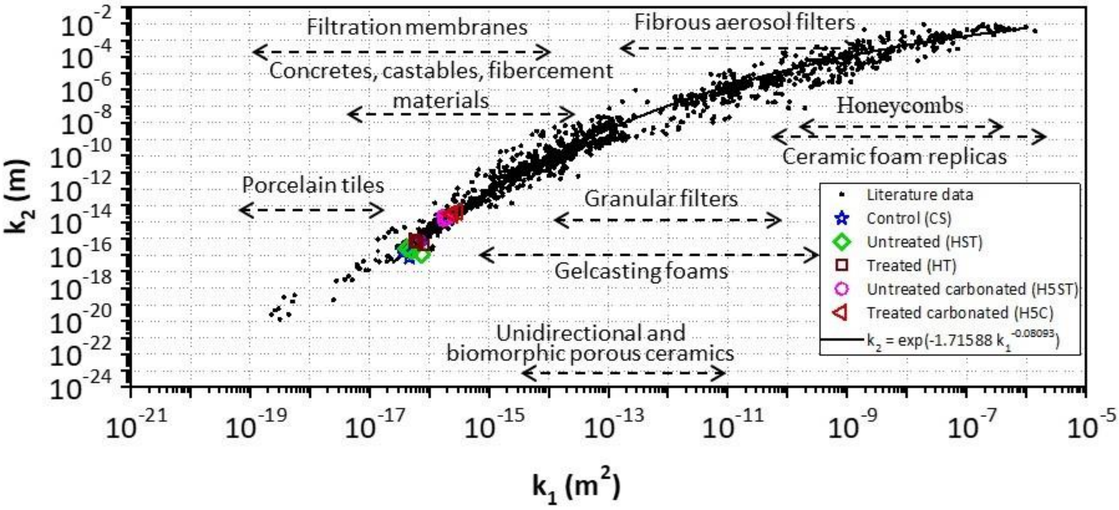


Fig. 15 Permeability map with the classification of porous materials and location of extruded cementitious composites tested in this work. Adapted from Innocentini et al. (2005)

The results of treated and untreated composites (2.9×10^{-16} $m^2 \geq k_1 \geq 3.7\times10^{-17}$ m^2) fall within the range of other cementitious materials, such as concretes, mortars, bricks, and fibercement boards, as depicted in Fig. 15. These materials are composed of fine particles bonded with hydraulic cement, resulting in a matrix with low porosity, small voids, and low permeability. Recently, similar permeability ranges for commercial corrugated sheets reinforced with plant fibers (Innocentini et al. 2019; Innocentini et al. 2005; Fioroni et al. 2020) were reported. The differences in permeability levels caused by carbonation and fiber treatment, as studied in this work, are also more apparent on the map.

4. Conclusions

The use of rubberwood fibers as reinforcement in cementitious composites showed great potential for the extrusion process. The chemical constituents present in the fibers influenced the hydration of the cement. Concerning the effect of the surface treatment of the reinforcing fibers and based on the statistical analysis, in the composites whose fibers were treated, it was found that the increases in the mechanical properties MOE, MOR and LOP were significant, while the increase in toughness was not significant. The better mechanical performance of the composite with fibers treated as also verified in SEM electron micrographs by the best adhesion between fiber and cementitious matrix, in which MOR values increased by 10.05 MPa for fiber-cement without the addition of fibers (control) to 11.64 MPa for those produced with treated fibers, a significant increase of 15.82%.

The physical and mechanical properties of the fiber-cement were affected significantly by curing under accelerated carbonation. There was an increase in the AD, WA and AP properties. For the MOR, MOE and LOP, a decrease in these properties has been demonstrated. The exception was toughness, a mechanical property that evaluates the resistance of crack propagation in the material, and accelerated carbonation, despite having reduced all other mechanical properties evaluated, which led to a better capacity of the composite in the absorption of total (elastic and plastic) energy, and this material can be used in fiber-cement tiles and precast materials.

The methodology for performing accelerated carbonation proved to be promising, low cost and capable of promoting accelerated carbonation in extruded fiber-cement.

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Statements & Declarations

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Availability of data and material

The datasets supporting the conclusions are included in the manuscript. Furthermore, the datasets analyzed in this study are available from the corresponding author upon request.

Code availability

Not applicable.

Authors' contributions

D.L. Faria and T.G.T. Pereira: Conceptualization, Investigation, Data Curation and Writing – Original Draft. M.D.M. Innocentini, J. Soriano and T.P. Protásio: Methodology, Investigation and Writing – Original Draft. L.M. Mendes and J.B. Guimarães Junior: Funding acquisition, Supervision, Resources, Project administration

Conflicts of interest

The authors declare they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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