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Assessment of the potential use of rubber tree endocarp as a reinforcement element in cementitious composites

Avaliação do potencial de uso do endocarpo da seringueira como elemento de reforço em compósitos cimentícios

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Abstract
The valorization of agroindustrial residues in cementitious composites is a promising strategy to reduce environmental impacts and develop bio-based materials with improved post-cracking behavior. This study evaluated the potential use of rubber tree (*Hevea brasiliensis*) endocarp as a reinforcement element and lightweight bioaggregate in cementitious composites. The endocarp was characterized by chemical analysis, physical tests, semi-adiabatic calorimetry, scanning electron microscopy, atomic force microscopy, and flexural testing. The material presented 5.72 ± 1.30% extractives, 69.17 ± 0.69% holocellulose, 23.74 ± 0.90% insoluble lignin, and 1.37 ± 0.08% ash. Its low density, 0.80 ± 0.01 g/cm³, confirmed its potential for lightweight composites, although its high water absorption, 74.51 ± 0.60%, indicated the need for surface treatment. Hot-water washing, $Ca(OH)_2$ treatment, and XSBR coating were investigated. The external surface showed higher roughness, 42.47 µm, than the internal face, 14.78 µm, favoring mechanical interlocking. Although no clear increase in peak load was observed in mortars, XSBR-coated endocarps promoted a more gradual post-cracking response. In concrete, XSBR-coated endocarp reached 0.78 ± 0.13 MPa in flexural strength, corresponding to approximately 74% of the value obtained with polypropylene fibers. The results indicate that rubber tree endocarp is a promising bio-based component for sustainable cementitious composites.
Keywords: Cementitious composites, natural fibers, post-cracking performance, XSBR, bioaggregate.

Resumo
A valorização de resíduos agroindustriais em compósitos cimentícios é uma estratégia promissora para reduzir impactos ambientais e desenvolver materiais de base biológica com melhor comportamento pós-fissuração. Este estudo avaliou o uso do endocarpo da seringueira (*Hevea brasiliensis*) como elemento de reforço e bioagregado leve em compósitos cimentícios. O material foi caracterizado por análise química, ensaios físicos, calorimetria semiadiabática, microscopia eletrônica de varredura, microscopia de força atômica e ensaios de flexão. O endocarpo apresentou 5,72 ± 1,30% de extrativos, 69,17 ± 0,69% de holocelulose, 23,74 ± 0,90% de lignina insolúvel e 1,37 ± 0,08% de cinzas. Sua baixa densidade, 0,80 ± 0,01 g/cm³, confirmou o potencial para compósitos leves, embora a elevada absorção de água, 74,51 ± 0,60%, tenha indicado a necessidade de tratamento superficial. Foram avaliadas lavagem em água quente, tratamento com $Ca(OH)_2$ e recobrimento com XSBR. A superfície externa apresentou maior rugosidade, 42,47 µm, que a interna, 14,78 µm, favorecendo o intertravamento mecânico. Embora não tenha havido aumento claro da carga máxima em argamassas, o XSBR promoveu resposta pós-fissuração mais gradual. Em concreto, o endocarpo recoberto com XSBR atingiu 0,78 ± 0,13 MPa de resistência à flexão, cerca de 74% do valor obtido com fibras de polipropileno.
Palavras-chave: Compósitos cimentícios, fibras naturais, desempenho pós-fissuração, XSBR, bioagregado.

1 INTRODUCTION

The construction industry is one of the largest consumers of mineral resources and is responsible for a significant share of global greenhouse gas emissions, mainly due to the production of Portland cement and the intensive use of conventional aggregates [1]. In this context, the development of cementitious composites incorporating renewable, low-density, and locally available materials has become an important strategy to reduce environmental impacts, promote circular economy principles, and increase the added value of agricultural and agroindustrial residues [2-4]. Among the different alternatives investigated, plant-based residues have attracted increasing attention because they can partially replace conventional raw materials, act as reinforcement phases, or be used as lightweight bioaggregates in cement-based systems [5-7].

Cementitious materials exhibit high compressive strength but limited tensile and flexural capacity, with a brittle response after cracking. This behavior restricts their performance in applications where crack control, toughness, and residual load-bearing capacity are required [8, 2]. The incorporation of fibers into cement matrices is a well-established approach to improve toughness, control crack propagation, and enhance post-cracking behavior [2, 9-11]. Steel, glass, and polypropylene fibers are commonly used for this purpose; however, their production is associated with high energy demand, cost, and environmental impacts [12-13]. Therefore, the use of plant fibers and lignocellulosic residues has been investigated as a more sustainable alternative, particularly in non-structural or semi-structural applications where reduced density, improved ductility, and lower environmental impact are desired [6,14-16].

Plant residues used in cementitious composites may include fibers, particles, shells, husks, stems, endocarps, and other lignocellulosic fractions generated by agricultural, forestry, or agroindustrial activities [7]. Materials such as sugarcane bagasse, coconut fiber, bamboo, eucalyptus residues, rice husk, coffee husk, banana pseudostem, wheat straw, and other vegetable residues have been evaluated in cement-based boards, mortars, concretes, and lightweight composites [7, 17-20]. These materials are attractive because of their low density, renewable origin, availability in large volumes, and ability to reduce the consumption of mineral aggregates or synthetic reinforcements. In addition, their cellular structure, surface roughness, and elongated or irregular geometry may contribute to mechanical interlocking with the cementitious matrix, favoring stress transfer after cracking [16, 21].

Despite these advantages, the incorporation of plant residues into cementitious matrices remains challenging. Lignocellulosic materials are chemically and physically complex, and their performance depends on parameters such as cellulose, hemicellulose, lignin, extractives, ash content, porosity, moisture content, and water absorption [7, 22]. Soluble sugars, tannins, organic acids, and other extractives may delay or inhibit cement hydration, reducing the formation of hydration products and impairing early-age mechanical development [23, 24]. In addition, the high alkalinity of the cement pore solution may degrade plant fibers over time, while the hydrophilic nature of lignocellulosic materials may lead to dimensional instability, increased water demand, poor interfacial adhesion, and durability concerns [3, 25, 26].

To overcome these limitations, different physical, chemical, and polymeric treatments have been proposed to improve the compatibility between plant residues and cementitious matrices. Hot-water washing can remove part of the soluble extractives and reduce the inhibitory effect of organic compounds on cement hydration [16, 27]. Alkaline treatments may modify the surface of lignocellulosic materials, remove amorphous constituents, increase surface roughness, and improve fiber-matrix interaction [16, 28]. Polymeric coatings, including carboxylated styrene-butadiene rubber latex (XSBR), have also been used to reduce water absorption, protect the lignocellulosic phase, and enhance interfacial bonding with the cementitious matrix [25]. Therefore, the efficiency of plant residues in cement-based composites depends not only on their intrinsic properties, but also on the adequacy of the treatment strategy used to control water absorption, hydration compatibility, and interface quality.

Within this broader field, bioaggregates and larger plant-derived particles represent an emerging class of materials for sustainable cementitious composites. Unlike conventional short fibers, these residues may combine low density, irregular geometry, internal porosity, and rough surfaces, allowing them to act simultaneously as lightweight aggregate, crack-bridging element, and mechanical anchorage component [25, 29, 30].

Previous studies have shown that endocarps and other hard lignocellulosic shells can be used as alternative aggregates or reinforcement elements in cement-based materials, depending on their morphology, chemical composition, and compatibility with cement hydration [27, 30-32]. However, the performance of each residue must be individually assessed, because plant species, anatomical structure, chemical composition, and surface characteristics strongly influence the interaction with cementitious matrices [7, 16].

The rubber tree (*Hevea brasiliensis*), native to the Amazon biome, is mainly known for latex production, which is widely used in the rubber industry [33, 34]. In addition to latex, rubber trees generate fruits composed of capsules containing seeds and a resistant endocarp [35]. While rubber tree seeds have been investigated for applications such as biofuels, food supplements, and animal feed [36, 37], the endocarp remains underexplored as an agroindustrial residue. Its rigid structure, low density, rough surface, and natural geometry suggest potential use as a bio-based component in cementitious composites. However, there is still limited information on its chemical composition, water absorption, compatibility with cement hydration, surface morphology, and contribution to mechanical behavior after cracking.

Thus, this study aims to evaluate the potential use of rubber tree endocarp as a bio-based reinforcement element and lightweight bioaggregate in cementitious composites. The work investigates the chemical and physical properties of the endocarp, its effect on cement hydration, its morphology and surface roughness, and the influence of different treatments, including hot water washing, calcium hydroxide immersion, and XSBR coating, on its performance in cement-based matrices. The mechanical response of mortars containing treated and untreated endocarps is evaluated with emphasis on endocarp orientation and post-cracking behavior, while the selected treatment was subsequently used in concretes compared with polypropylene- and steel-fiber-reinforced composites.

By combining material characterization, compatibility analysis, and mechanical testing, this study seeks to clarify whether rubber tree endocarp can be used as a sustainable residue-derived component for cementitious composites, contributing to waste valorization and the development of lower-impact construction materials.

2 MATERIALS AND METHODS

2.1 MATERIAL

The cementitious composites were produced using Portland cement Type V-ARI (CSN, Brazil), quartz sand with a fineness modulus of 2.30, granitic coarse aggregate with a maximum particle size of 9.52 mm, municipally treated water, and a commercial Quartzolit plasticizing air-entraining admixture. Carboxylated styrene-butadiene rubber latex, XSBR NTL-218 TB, supplied by Nitriflex, Brazil, was used as a polymeric coating for the rubber tree endocarps. The chemical composition and physical properties of the cement used in this study are presented in Table 1.

Table 1: Chemical composition and physical properties of the Portland cement Type V-ARI.

Chemical components	**CP V – ARI (%)**
CaO	63.8
SiO_2	17.8
SO_3	3.82
Al_2O_3	4.98
K_2O	0.82
TiO_2	0.21
MnO	0.10
Others	8.47
Specific density (g/cm^3)	3.2
Surface area (m^2/kg)	420

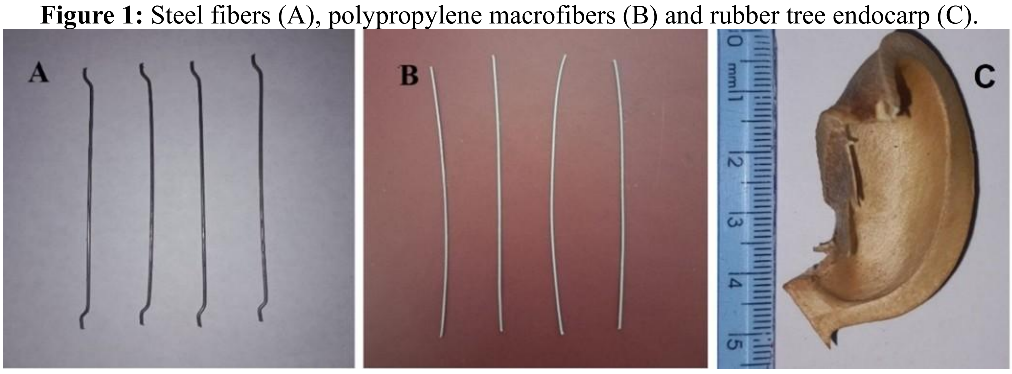
The rubber tree endocarp was the bio-based component evaluated in this study. It was collected on the campus of the Federal University of Lavras, Lavras, Minas Gerais, Brazil (21°14’45” S, 44°59’59” W), during February and March of 2022 and 2023. After collection, the endocarps were subjected only to a simple cleaning procedure, consisting of the removal of loose surface impurities by air blowing and sieving. The cleaned endocarps were then selected and submitted to the treatments described in the following sections.

Steel fibers, Dramix 3D 80/60, supplied by Belgo Arames, Brazil, and polypropylene macrofibers, supplied by ConCresolos, Brazil, were used as reference reinforcing materials. Their characteristics are presented in Table 2. These fibers were not selected as sustainable alternatives, but as mechanical benchmarks to evaluate the reinforcing efficiency of the endocarp in concrete. Since the endocarp presents a rigid geometry that may contribute to crack bridging and mechanical anchorage, its performance was compared with conventional fibers known to improve post-cracking behavior. Synthetic and metallic fibers were considered more suitable references than vegetable fibers in this first assessment, as low contents of plant fibers in concrete may not provide a clear mechanical response and may introduce additional variability related to degradation, water absorption, and fiber-matrix adhesion. The fibers and endocarp investigated are shown in Figure 1.

Table 2: Technical characteristics of the steel fibers and polypropylene macrofibers used in this study, according to the manufacturers' specifications.

Fiber	Length (L)	Diameter (d)	L/d	Tensile strength	Specific density
Steel fiber	60 mm	0.75 mm	80	1225 MPa	7.85 g/cm³
PP fiber	52 mm	0.35 mm	75	570 – 660 MPa	0.93 g/cm³

Figure 1: Steel fibers (A), polypropylene macrofibers (B) and rubber tree endocarp (C).



2.2 ENDOCARP CHARACTERIZATION

The chemical characterization of natural endocarps quantified total extractives, lignin, ash, and holocellulose, following NBR 14853 [38], NBR 7989 [39], and NBR 13999 [40]. Water absorption (24 h), moisture content, and basic density were determined according to ASTM D2395–17 [41].
As plant extractives may delay or inhibit cement hydration [23,24], the endocarps underwent three cycles of hot water washing, immersion in a $Ca(OH)_2$ solution [25,32], and coating with XSBR polymer [21,24,29,30], to mitigate this effect and enhance chemical compatibility and adhesion.

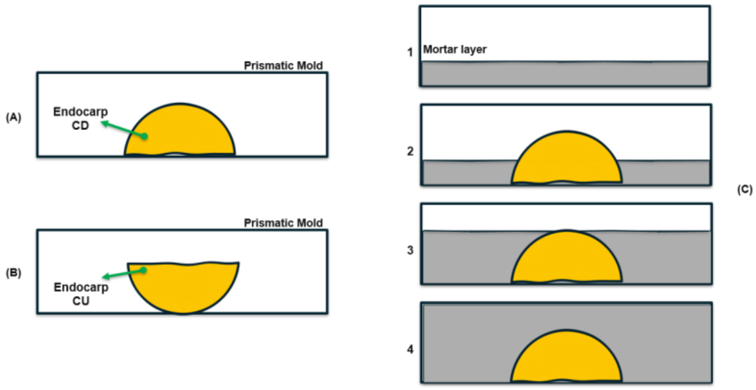
The hydration behavior of the cement pastes was evaluated through semi-adiabatic temperature monitoring, adapted from calorimetric procedures described in NBR 12006 [42]. Endocarps (2% by cement mass), in their natural, washed, $Ca(OH)_2$ treated, and XSBR coated forms, were tested. Cement pastes (500 g of cement, w/c ratio of 0.4) were placed in EPS boxes (418 cm³) with K 32 AWG thermocouples at the geometric center. Measurements were taken every 20 s for 48 h using a PicoLog TC-08 Data Logger (Pico Technology, UK) connected to a computer via PicoLog 6 software, in a controlled environment (20°C, 64% RH). The cumulative heat release was estimated from the temperature evolution curves by numerical integration of the temperature increase over time, normalized by cement mass, allowing a comparative assessment of the hydration behavior of the different formulations.
Endocarp morphology and surface structure were analyzed using SEM (LEO EVO 40 XVP, Carl Zeiss, Germany) [43]. Longitudinally sectioned samples were gold-coated and analyzed at 15 kV and 11 mm working distance. Surface rough-ness was evaluated using AFM (Nanosurf FlexAFM), with samples fixed on glass slides. The analysis was conducted in dynamic force mode (190 kHz, 2 s/line, 256 points/line, 6V vibration amplitude, Dyn190AI cantilever, <10 nm tip radius, 48 N/m spring constant) over 60×60 μm areas. Data processing was performed using Gwyddion 64bit software.

2.3 PRODUCTION OF CEMENTITIOUS COMPOSITES WITH ADDED ENDOCARPS

The mortar was prepared with a 1:3 cement:sand ratio and a w/c ratio of 0.60. Prismatic specimens with dimensions of 40 × 40 × 160 mm were produced, following the geometry commonly used for flexural testing of hydraulic-cement mortars according to ASTM C348 [44] and EN 196-1 [45]. Flexural behavior was evaluated by three-point bending using an AROTEC universal testing machine equipped with a 20 kN load cell. The load was applied at mid-span, with a support span of 100 mm, following the specimen geometry and loading configuration commonly adopted in EN 196-1[45].

The experimental program was designed to investigate both the influence of endocarp geometry and the effect of different surface treatments on the mechanical performance of the mortars. Endocarps were incorporated into the mortar specimens with their concavities facing either upward (CU) or downward (CD), as illustrated in Figure 2. This variation in positioning was adopted because the natural concave geometry of the endocarp could affect crack propagation, stress transfer mechanisms, crack opening control, and post-cracking load-bearing capacity. In addition, the comparison among untreated and treated endocarps allowed the identification of the treatment with the best mechanical performance, which was subsequently selected for the production of the concrete composites.

Figure 2: Molding of the mortar specimens for the flexural test, with the concavities of the endocarps facing downwards (CD) (A) and upwards (CU) (B) and the specimen molding process (C).



Five mixture conditions were evaluated: reference mortar without endocarps, natural endocarp, hot-water-washed endocarp, calcium hydroxide-treated endocarp, and XSBR-coated endocarp. For all mixtures containing endocarps, both orientations (CU and CD) were tested. A total of 27 specimens were produced, with three replicates per condition, and tested after 28 days of curing. The experimental matrix is summarized in Table 3.

Table 3: Summary of all mortar composites that were produced for the endocarp adhesion test (in relation to their position within the sample).

Reference, without added endocarps
Natural endocarp with concavity upwards (CU) and downwards (CD)
Endocarp washed in hot water with concavity upwards (CU) and downwards (CD)
Treated with calcium hydroxide ($(CaOH)_2$) with concavity upwards (CU) and downwards (CD)
Coated with XSBR polymer with concavity upwards (CU) and downwards (CD)

For the concrete specimens, three reinforced mixtures were designed containing XSBR-coated endocarp, polypropylene macrofibers, or steel fibers. In all mixtures, the reinforcing materials were incorporated at a volume corresponding to 1% of the coarse aggregate, with proportional adjustment of the gravel content. The mix proportions were defined based on previous studies [32,46] and are presented in Table 4. A commercial Quartzolit plasticizing air-entraining admixture was added at 0.9% by mass of cement to improve workability, according to the information presented in Table 5. The mixtures were prepared in a 400 L mixer at the UFLA Structural Laboratory, and the slump values ranged from 17 to 19 cm.

Prismatic concrete specimens measuring 500 mm × 150 mm × 150 mm were produced for the flexural tests. After 28 days of curing, the specimens were tested under three-point bending using an Instron testing machine equipped with a 300 kN load cell. The load was applied at mid-span, following a center-point loading configuration adapted from ASTM C293/C293M [47]. Based on the specimen depth of 150 mm, a support span of 450 mm is recommended, corresponding to three times the beam depth. This configuration was used to compare the flexural response of concretes reinforced with XSBR-coated endocarp, polypropylene macrofibers, and steel fibers.

Table 4: Mix design (by mass of cement) adopted for the production of concrete.

Studied Concretes	Cement	Sand	Gravel	Endocarp	PP	Steel	w/c	Additive
Reference	1	2.6	2.70	-	-	-	0.6	0.09
1% treated endoc.	1	2.6	2.67	0.008	-	-	0.6	0.09
1% PP fiber	1	2.6	2.67	-	0.01	-	0.6	0.09
1% Steel fiber	1	2.6	2.67	-	-	0.07	0.6	0.09

Table 5: Physical properties of the air-entraining additive with plasticizing action from the Quartzolit brand, provided by the manufacturer.

Physical properties	Value
Appearance	Light brown liquid
Formulation basis	Surfactants
Solids content	3.0 – 4.0 %
pH	4.5 – 7.5
Specific mass	1.002 – 1.004 Kg/dm³

3 RESULTS

3.1 ENDOCARP CHEMICAL AND PHYSICAL CHARACTERIZATION

The chemical composition of the rubber tree endocarp is shown in Tables 6. The rubber tree endocarp exhibited 5.72% extractives. This value is moderately higher than that found for macauba endocarp (3.72%) [27] and lower in comparison to bean residues (8.16%) [18]. Extractives are related to cement hydration inhibition and may cause chemical incompatibility between lignocellulosic residues and the cement matrix [48]. The low concentration of extractives in the rubber tree endocarp is advantageous as it minimizes interference in cement hydration, making it suitable for use in cement composites.

Table 6: Chemical composition of the endocarp of the rubber tree fruit in %.

	Total extractives	Insoluble lignin	Holocellulose	Ash
Endocarp	5.72 ± 1.30	23.74 ± 0.90	69.17 ± 0.69	1.37. ± 0.08

The holocellulose content was similar to residues from pine (66.43%) and miriti (69.91%) [49], and higher than jupati (45.68%) [50]. Holocellulose contains hydroxyl groups (-OH) that impart hydrophilic nature, increasing water absorption, which may affect the strength and durability of composites [50,51]. The ash content was lower than cocoa (3.70%) and wheat residues (10.74%) [52,53]. The rubber tree endocarp showed suitable chemical characteristics for cement composites.

Regarding the physical properties of the rubber tree endocarp, as summarized in Table 7, its basic density of the endocarp was lower than macauba (1.23 g/cm³) and higher than mandacaru (0.34 g/cm³) [27, 31]. The relatively low density may contribute to the production of lightweight cementitious composites.

The moisture content was similar to other lignocellulosic residues, such as corn cob (8.3%) and sunflower stalk (9.8%) [54]. The water absorption was higher than macauba (9.05%) and lower in comparison to bamboo (109.14%) [27, 55], which may compromise adhesion to the cement matrix [3]. The elevated absorption may also be related to the surface characteristics of the endocarp, as discussed in the morphological analysis presented in Section 3.2. Hydrophobic coatings can reduce water absorption and improve the interface.

Table 7: Physical properties of the endocarp.

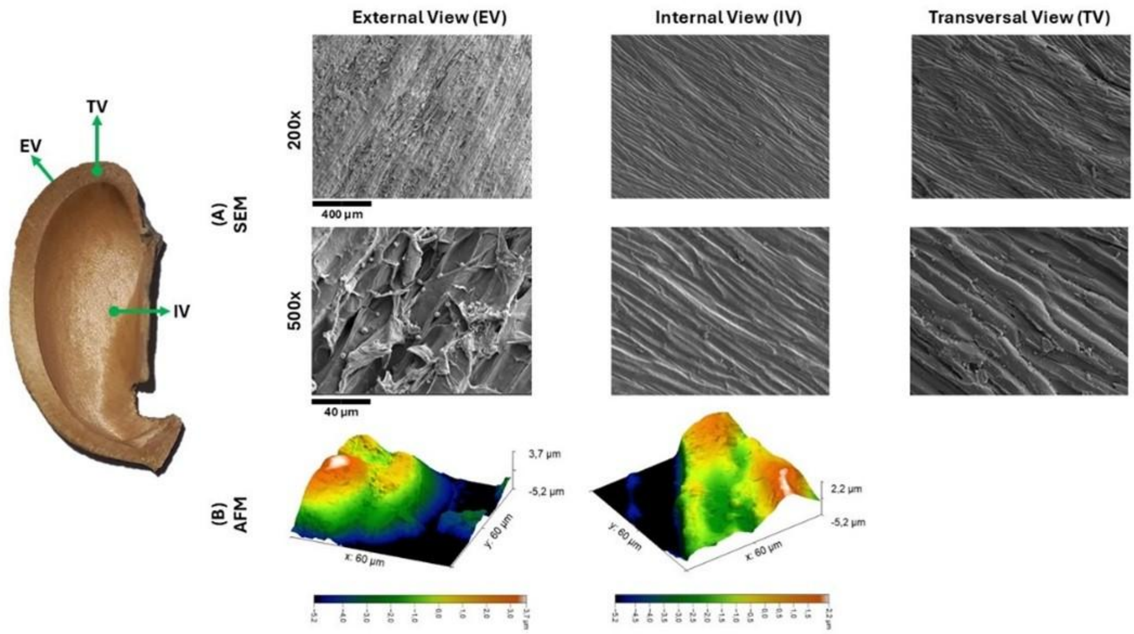
	Basic density (g/cm³)	Moisture content (%)	Water absorption (%)
Endocarp	0.80 ± 0.01	5.24 ± 0.18	74.51 ± 0.60

3.2 ENDOCARP MORPHOLOGY

The morphology of the rubber tree endocarp is presented in Figure 3. SEM images revealed a heterogeneous structure, with clear differences between the external, internal, and transversal surfaces. The external face showed a more irregular texture, with grooves, discontinuities, and exposed fibrous features, while the internal face presented a more organized and smoother fibrous arrangement. The transversal view confirmed the compact and fibrous nature of the endocarp, indicating that the material combines rigidity with a naturally textured surface.

AFM analysis supported these observations, showing higher roughness on the external face, 42.47 µm, than on the internal face, 14.78 µm. The irregular and porous morphology observed on the external surface may also contribute to the high water absorption previously discussed, since surface discontinuities and exposed fibrous structures favor water retention and interaction with the cement pore solution. At the same time, this rougher surface can favor physical anchorage and mechanical interlocking with the cementitious matrix. Therefore, the external morphology of the endocarp may contribute to stress transfer after matrix cracking, while its concave geometry may influence crack propagation depending on its orientation within the specimen. These results support the evaluation of the endocarp not only as a lightweight bioaggregate, but also as a geometry-dependent reinforcing element.

Figure 3: SEM micrographs of the external, internal and lateral faces of the endocarp of the rubber tree fruit and AFM image of the external and internal face of the endocarp.



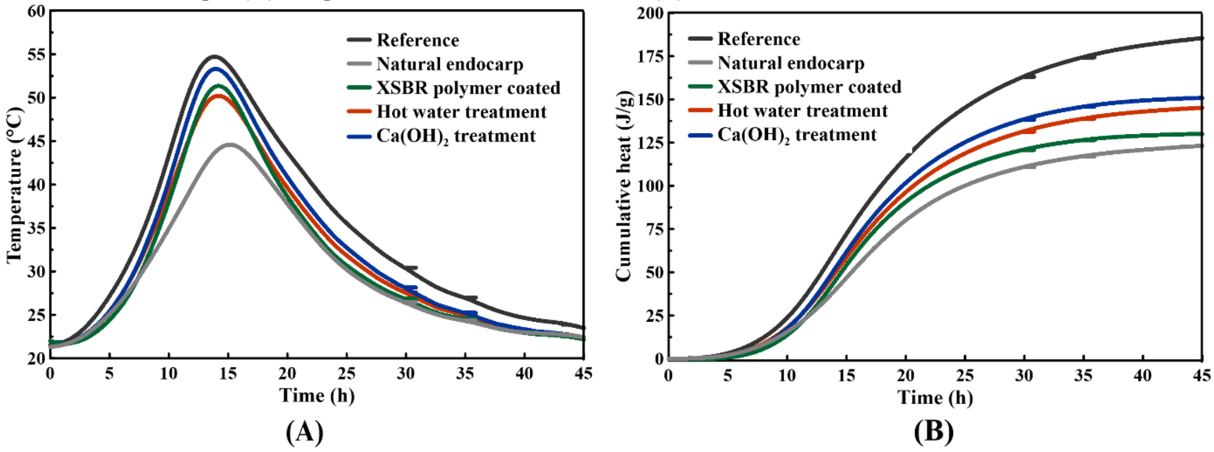
3.3 CEMENT HYDRATION

Semi-adiabatic calorimetry showed that the cement paste with natural endocarp experienced a delay in hydration, evidenced by a shift in the curve and a reduction in the peak compared with the reference paste (See Figure 4a). The reference mixture reached the highest peak temperature (approximately 54-55 °C), while the paste with untreated endocarp reached only 44-45 °C, indicating lower hydration intensity and reduced formation of hydration products (C-S-H and $Ca(OH)_2$). Similar behavior has been widely reported for lignocellulosic materials incorporated into cementitious matrices and is commonly associated with incompatibility between cement hydration reactions and soluble compounds released from plant-based particles [56-59].

The lower temperature peak observed for the paste containing natural endocarp can be related to the presence of soluble organic compounds in the lignocellulosic material. Saccharides and extractivederived compounds may retard cement hydration by complexing Ca^{2+} ions, adsorbing onto cement grain surfaces, and hindering the nucleation and growth of hydration products such as C-S-H [24, 60].

In addition, some sugars may interact differently with clinker phases, affecting C_3S hydration and early ettringite formation [61, 62]. Under the highly alkaline conditions of cement paste, saccharides may also degrade into sugar acid anions or calcium-sugar complexes, which can be more effective hydration retarders than the original compounds [63]. This explains why even moderate extractive contents may influence the hydration kinetics of cementitious matrices containing plant-based residues. Furthermore, the high water absorption capacity of vegetal aggregates may temporarily reduce the availability of free water during early hydration stages, further contributing to the lower temperature peak.

Figure 4: Curves obtained in the semi-adiabatic calorimetry test of cement pastes with natural and treated endocarps: (A) temperature as a function of time; (B) cumulative heat as a function of time.



Differences among the treatments demonstrated that surface modification of the endocarp partially mitigated these inhibitory effects. The hot-water washed endocarp showed higher peak temperatures (approximately 50-51 °C) than the untreated condition, probably because washing removed part of the soluble extractives and sugars responsible for hydration retardation. Similar improvements after treatments have been reported in wood-cement compatibility studies [28, 56]. The $Ca(OH)_2$ -treated endocarp presented the highest peak temperature among the treated conditions (approximately 53-54 °C), indicating improved hydration behavior, possibly due to previous saturation of reactive sites and enhanced chemical compatibility with the alkaline cementitious environment. Meanwhile, the XSBR-coated particles reached approximately 52-53 °C, suggesting that the polymer coating acted as a physical barrier that limited the leaching of inhibitory compounds and reduced water exchange between the endocarp and the cement paste.

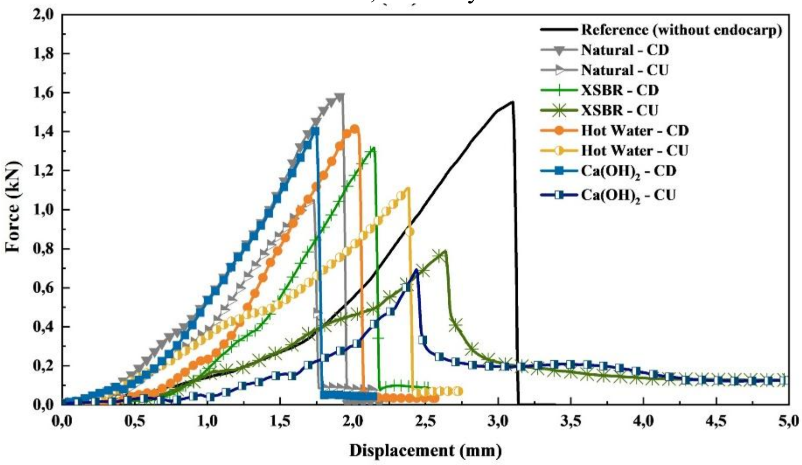
In addition to peak temperature analysis, the estimated cumulative heat release curves (Figure 4b) reinforced the differences among treatments throughout the hydration period. The reference paste presented the highest cumulative heat release, reaching approximately 185-190 J/g after 45 h, while the untreated endocarp condition showed the lowest values, around 120-125 J/g, confirming that vegetal aggregates reduced the overall hydration progression. The treated endocarps consistently exhibited intermediate behavior, with cumulative heat release values of approximately 150 J/g for the $Ca(OH)_2$-treated endocarp, 145 J/g for the hot-water washed condition, and 130 J/g for the XSBR-coated particles, indicating that the applied treatments partially restored cement hydration activity. Although the cumulative heat release values were estimated from temperature evolution rather than obtained from direct calorimetric measurements, the results consistently supported the same hydration tendency observed in the temperature-time curves.

Moreover, none of the treatments fully restored the hydration behavior observed for the reference paste, however, both the increase in peak temperature and the higher cumulative heat release compared with the natural endocarp condition demonstrate improved cement compatibility after the applied treatments. Therefore, the semi-adiabatic hydration monitoring results support the use of surface treatments before incorporating rubber tree endocarp into cementitious composites.

3.4 MECHANICAL PROPERTIES OF STUDIED COMPOSITES

The mechanical performance of the cementitious composites was evaluated by flexural testing. The results are discussed separately for the mortar specimens (Figure 5), used to assess the influence of treatment and endocarp orientation, and for the concrete specimens (Figure 6), used to compare the selected endocarp treatment with conventional reinforcing fibers.

Figure 5: Typical load-displacement curves of treated and untreated endocarps, with concavities facing up and down, at 28 days.



For the mortar specimens (Figure 5), all specimens presented a progressive increase in load-displacement curves until the peak load was reached, followed by either abrupt failure or a residual post-peak response, depending on the treatment and endocarp orientation. The reference mortar (without endocarp) reached the highest peak load, approximately 1.55 kN, but failed abruptly after cracking, indicating a typical brittle response of an unreinforced cementitious matrix.

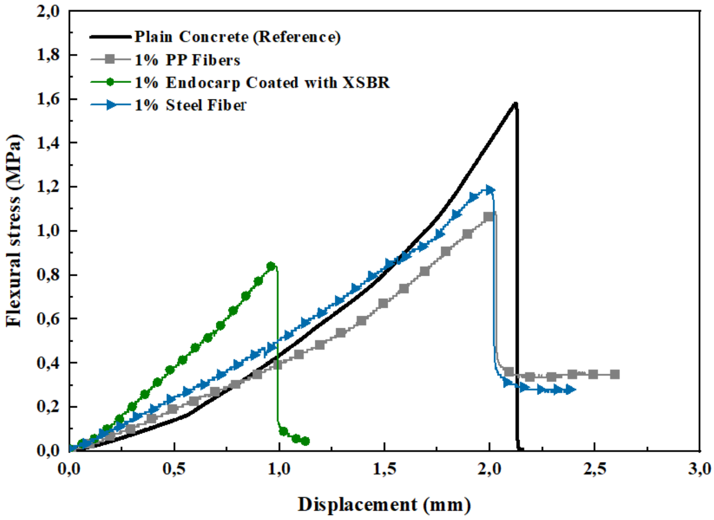
The incorporation of endocarps modified the flexural response of the mortars. In general, specimens with the concavity facing downward (CD) reached higher peak loads and showed higher initial stiffness than those with the concavity facing upward (CU). Natural-CD, Hot Water-CD, $Ca(OH)_2$-CD and XSBR-CD reached peak loads in the approximate range of 1.3-1.6 kN, indicating that the presence of the endocarp did not necessarily impair the maximum load-bearing capacity when its orientation favored a stiffer load-transfer configuration. However, most CD specimens showed a sharp load drop after the peak, suggesting limited residual load-bearing capacity after crack localization.

In contrast, the CU orientation generally led to lower peak loads but improved post-peak behavior. This was particularly evident for XSBR-CU and $Ca(OH)_2$-CU specimens, which maintained residual loads over a larger displacement range, extending up to approximately 4.5-5.0 mm. The XSBR-CU curve, for example, showed a gradual load decay after the peak instead of immediate collapse. This indicates that the upward orientation of the concavity may have favored crack-bridging and mechanical interlocking after matrix cracking. Similar behavior has been reported for bioaggregates embedded in cementitious matrices, in which the aggregate shape and surface irregularities promoted smoother post-peak responses due to mechanical anchorage and frictional effects [30]. The $Ca(OH)_2$-CU specimen showed a similar residual response, although with a lower peak load.

These results suggest that the effect of the endocarp is strongly dependent on its orientation within the mortar. The CD orientation favored peak load and stiffness, probably because the endocarp was positioned in a way that provided greater resistance before crack propagation. Conversely, the CU orientation appeared to favor residual load transfer after cracking, possibly by allowing the concave geometry to interact more effectively with the propagating crack. Therefore, the endocarp should not be evaluated only in terms of maximum load capacity, but also in terms of its ability to modify the post-cracking response of the cementitious matrix.

Considering the overall mechanical behavior observed in the mortar specimens, particularly the improved post-cracking response obtained for the XSBR-treated condition, this treatment was selected for the subsequent concrete production stage.

Figure 6: Flexural stress-displacement curves of reference and reinforced concrete specimens after 28 days of curing.



The flexural behavior of the reinforced concrete specimens is shown in Figure 6, while the average flexural strength values are summarized in Table 8. According to the average results, the steel fiber-reinforced concrete presented the highest flexural strength (1.45 ± 0.23 MPa), followed by the polypropylene fiber-reinforced concrete (1.05 ± 0.08 MPa), and the concrete containing XSBR-coated endocarp (0.78 ± 0.13 MPa). The results allowed a comparative evaluation of the mechanical response promoted by XSBR-coated endocarp, polypropylene fibers, and steel fibers under flexural loading.

Table 8: Average flexural strength and standard deviation of the concrete specimens reinforced with XSBR-coated endocarp, polypropylene fibers, and steel fibers after 28 days of curing.

Fiber Type	Flexural Strength (MPa)
XSBR-coated endocarp	0.78 ± 0.13
Polypropylene (PP)	1.05 ± 0.08
Steel	1.45 ± 0.23

The concretes reinforced with polypropylene and steel fibers presented distinct mechanical responses under flexural loading. The curves showed that both fiber-reinforced concretes maintained load-bearing capacity after cracking, confirming the crack-bridging effect provided by the fibers. The polypropylene fiber-reinforced concrete exhibited a more stable post-peak plateau, with residual flexural strength of approximately 0.35 MPa up to larger displacements. The steel fiber-reinforced concrete also showed post-cracking resistance, although with a slightly lower residual level, around 0.28-0.30 MPa. This behavior is consistent with the reinforcing mechanisms commonly reported for synthetic and metallic fibers in cementitious composites.

The concrete containing the selected endocarp treatment (XSBR) presented lower average flexural strength than the concretes reinforced with polypropylene and steel fibers. However, the flexural curve indicated that the endocarp contributed to stress transfer mechanisms within the cementitious matrix before and immediately after crack propagation. This behavior may be associated with the combined effect of the XSBR coating, surface roughness (42.47 µm and 14.78 µm), and irregular concave geometry of the endocarp, which may have promoted mechanical interlocking and partial crack-bridging effects. Previous studies have shown that XSBR coatings can improve interfacial bonding and stress transfer between lignocellulosic reinforcements and cementitious matrices [64,65].

Nevertheless, the post-cracking performance of the endocarp-reinforced concrete remained lower than that observed for polypropylene and steel fibers, with a more pronounced reduction in load after the peak. Therefore, although the selected endocarp treatment did not achieve the same post-cracking performance as conventional fibers, the results demonstrated that the endocarp was capable of contributing to stress transfer and residual load-bearing mechanisms within the cementitious matrix. Additional optimization of treatment strategy, content, and distribution within the matrix is still necessary to enhance residual load-bearing capacity after cracking.

Considering its bio-based origin, low density (0.80 ± 0.01), and waste valorization potential, the endocarp still represents a promising alternative material for sustainable cementitious composites.

4 CONCLUSION

The following conclusions can be drawn from the present study:

- Rubber tree endocarp presented low density and high surface roughness, indicating potential as a lightweight bioaggregate and reinforcement element in cementitious composites;
- The natural endocarp delayed cement hydration due to the release of soluble lignocellulosic compounds and its high-water absorption capacity. The applied treatments improved the compatibility between the endocarp and the cementitious matrix;
- XSBR coating promoted better interfacial interaction and a more gradual post-cracking response;
- Endocarp orientation influenced the flexural behavior of mortars, with concavity-downward specimens showing higher peak load and concavity-upward specimens showing improved residual behavior after cracking;
- XSBR-coated endocarp concrete exhibited lower flexural strength than polypropylene and steel fiber-reinforced concretes, but still demonstrated contribution to post-cracking stress transfer mechanisms;
- Rubber tree endocarp showed potential as a sustainable bio-based component for cementitious composites, although further studies on durability and optimization are still required.

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