

EFFECTS OF TREATMENT OF COIR FIBER AND CEMENT/FIBER RATIO ON PROPERTIES OF CEMENT-BONDED COMPOSITES

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This study investigated the effects of different treatments of coir fibers (*Cocos nucifera* L.), and cement:coir ratio on physical and mechanical properties of cement-bonded composites. Three treatments: adding 4% of CaCl₂, immersion in hot water at 80°C for 90 minutes, and immersion in NaOH aqueous solution at 5% for 72 hours and two cement:coir ratios (3:1 and 4:1) were chosen for manufacturing 24 panels. After 28 days of setting, characterization was made through static bending (MOE, MOR), parallel compression (COMP), internal bonding (IB), thickness swelling (TS), and water absorption (WA) (2 and 24 hours of water immersion) tests. Treating coir fibers with hot water provided an improvement in the panel's properties. This treatment had better results in MOE and COMP. Panels produced with CaCl₂ addition were resistant as well; however coir fibers treated with NaOH produced cement/coir composites with unsatisfactory physical and mechanical properties.

Keywords: Cement-lignocellulosic composites, *Cocos nucifera*, Panels' properties

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INTRODUCTION

A large amount of agricultural waste is produced each year worldwide. This waste can be dealt with in different ways, but the most effective approach is to reuse it to produce new products (Khedari et al. 2001). An interesting alternative to waste disposal, using a strategy that supports environmental sustainability, involves addition of the waste to a mineral binder like cement (Abdel-Kader and Darweesh 2010).

The introduction of vegetable fibers in the manufacture of composite materials has received great attention from both researchers and the industry. Natural fibers have excellent mechanical properties, biodegradable, and are inexpensive compared to synthetic fibers. The natural vegetable fibers most often used in such applications include sisal, jute, kenaf, and coir, among others. The use of building materials based on fiber-reinforced cement has increased rapidly in recent years, especially in developing countries, which have invested heavily in the technology. The current global production of the material is estimated at approximately 30 million tons per year, mainly in Asian countries (Campello et al. 2007).

Lignocellulosics-cement composites offer some advantages over some conventional building materials. Because such products fall within the category of cement-based materials, they increase the sense of security by meeting safety and health requirements,

resisting attack by fungi and insects, and resisting fires. The thermal and acoustic insulation provided by this type of panel should also be considered (Ramirez-Coretti et al. 1998).

The physical and mechanical properties of cement-bonded panels, in addition to the facility of being sawn, glued, and nailed, are indicators of these composites' applications. These features allow them to be painted, roughcasted, and coated with other types of materials. The versatility of the composite allows manufacturers to add value to the product by delivering it in sanded, painted, or coated forms. With this versatility, the composites can be used directly with finishing material. Nevertheless, the properties of these kinds of composites are greatly affected by: the species of lignocellulosic material, particle geometry, the type of mineral binder, additives, the lignocellulosic:cement ratio, and other factors. Thus, according to Del Menezzi et al. (2007) there are obstacles to the utilization of lignocellulosic materials in mixtures with cement. The main problem is the inhibitory effect caused by lignocellulosic material on the setting of the cement. The compounds of the material, mainly extractives and polysaccharides, affect reactions between lignocellulosic materials and cement, resulting in composites of low quality.

Lignocellulosic cement-bonded composite ranks high among forest products, but its use can be restricted, as some limitations in the production process may apply. The high variation in the compatibility of lignocellulosic species mixed with cement, due to inhibitory substances, is one of those limitations that leads to the necessity of using additives to overcome this problem (Wei et al. 2000; Asasutjarit et al. 2007). It should be also highlighted that due to the alkaline characteristic of the cement, lignocellulosic materials can be chemically degraded, thus reducing the durability of the composite. According to Mansur et al. (2008) by controlling the cement alkalinity and reducing the initial pH of the system, the durability can be improved.

Several studies, basically including removal of chemical substances by means of treatments of the lignocellulosic fibers in cold (Sutigno 2000) or hot water (Moslemi et al. 1983; Asasutjarit et al. 2007) and immersion in an aqueous solution of NaOH (Prasad 1983; Alberto et al. 2000), confirm that the compatibility with cement is improved. Treatment with NaOH aqueous solution works by modifying cellulose crystallinity, cleaning the fiber surface, increasing surface roughness, removing or solubilizing amorphous polysaccharides, and degrading lignin, waxes, and oils (Carvalho et al. 2010; Thiruchitrambalam et al. 2010). These pre-treatments are applied before blending fibers with the mineral binder, thus removing inhibitory substances.

Chemical additives play another role by speeding cement hydration before the inhibitory effect of the compounds in the lignocellulosic material can take place. Accelerators such as calcium chloride (CaCl_2) (Almeida et al. 2002; Okino et al. 2004; Olorunnisola 2009) and magnesium chloride (MgCl_2) (Wei et al. 2000; Latorraca and Iwakiri 2000) have been widely used. However, the main drawback of using these accelerators is that chloride ion can accelerate the corrosion of steel.

In this context, the present study aimed to evaluate the technical feasibility of producing mineral cement panels reinforced with coir fiber under different treatments, and to assess the effect of the cement:coir fiber ratio variation on the physical and mechanical properties of the panel.

EXPERIMENTAL

Materials Processing and Characteristics

Coir fiber (*Cocos nucifera* L.) from 10-year old palm trees was obtained at a trading company located in the municipality of Camandatuba, Bahia, Brazil. The fibrous material came from processing of coconut outer shell harvested between the 7th and 9th months after inflorescence. Initially the fiber was processed into slender type particles in a hammermill with openings of 6.0 mm. Afterwards, the material was classified according to the following sieve openings: 3.0 mm, 1.5 mm, and 1.0 mm. The material collected on the sieves having 1.5 and 1.0 mm openings (-3.0+1.0 sieves) was used to manufacture panels; these fibers had the following characteristics: ≈ 30.52 mm (length), ≈ 0.194 mm (diameter), and ≈ 1.06 g/cm³ (density).

Ordinary Portland cement (CP-II-Z-32, Votorantim®) was used in this research. Table 1 presents some mechanical, chemical, and physical properties of the cement used. According to the producer this kind of cement meets Brazilian standard NBR 11578 (ABNT, 1991) regarding compression strength (>32 MPa) and all others requirements. No further material analysis was performed.

Table 1. Some Mechanical, Chemical and Physical Properties of the Cement Used

Class	Clinker + Gypsum	Limestone	Pozzolan	MgO	SO ₃	CO ₂	Specific area
	-----%-----						m ² /kg
32 MPa	76 – 94	0 – 10	6 – 14	≤ 6,5	≤ 4.0	≤ 5.0	≥ 260

Source: <http://www.votorantimcimentos.com.br/hotsites/cimento/base.htm> (accessed on July 20th 2011)

Production of the Coir-reinforced Cement Panel

The experimental design included three treatments: addition of 4% CaCl₂ in the mixture of cement and fibers (treatment #1), immersion of fibers in hot water at 80°C for 90 minutes before blending (treatment #2), and immersion of fibers in NaOH aqueous solution at 5% for 72 h before blending. Two cement:coir ratios (3:1 and 4:1) were also evaluated. CaCl₂ was not added in the mixture for treatments 2 and 3. The cement:water ratio was constant and equal to 2.5:1.

The combination of those factors generated a total of six treatments with four panels being produced for each treatment (Table 2), totaling 24 panels, each measuring 350 mm x 350 mm x 12.5 mm (length, width, and thickness) and having a nominal density of 1200 kg/m³. Previously ground coir fiber was added to a mechanical mixer and sprayed with water by aid of an air spraying system to moisten the fiber. Once the coir fiber was moistened, the cement was added to the mixture.

After mixture was prepared, the mat assembling began. Two smooth surface metal plates measuring 500 mm x 500 mm were used for each mat. The mats were assembled on the plates, with the help of a mat forming box (350 mm x 350 mm), which was placed on the metal plate with a plastic to prevent the mixture from sticking to the plate.

Table 2. Experimental Design

Treatments	Cement:coir fiber ratio	Number of replicates
CaCl ₂	3:1	4
	4:1	4
NaOH	3:1	4
	4:1	4
Hot water	3:1	4
	4:1	4

Manual pre-pressing was applied to reduce mat thickness, and then the mat was finally pressed in a hydraulic press at room temperature (23°C) and pressure of 3.55 MPa for 24 hours. After pressing, the panels were removed from the press and placed in an air-conditioned room for 27 days - at a controlled temperature (20 ± 3°C) and relative humidity (65 ± 1%) - to complete the curing process.

Physical and Mechanical Tests

After 28 days of setting, five specimens measuring 350 mm x 50 mm (length x width) were taken from each panel to determine all of the aforementioned physical and mechanical properties. Panels were evaluated for static bending (modulus of rupture - MOR and modulus of elasticity - MOE), parallel compression (COMP), internal bonding (IB), thickness swelling (TS), and water absorption (WA). All tests were conducted according to NBR 14810-3 (ABNT 2002).

Fiber Analysis by Scanning Electronic Microscope (SEM)

Uncoated fibers (natural and treated) were analyzed in order to identify any morphological change as a function of the applied treatments. A scanning electron microscope was employed in low vacuum mode (FEI, Model Quanta 200 3D - Dual Beam) and set as follow: large field detector (LFD), working distance from 8.7 mm to 11.8 mm, and 20 kV operating voltage. This analysis was done at the Ballistics Department of the Brazilian Federal Police.

Statistical Analysis

The analysis of the physical and mechanical properties of the panels under the effect of the three treatments (hot water immersion, immersion in NaOH solution, and CaCl₂ addition) and two cement:coir fiber ratios (3:1 and 4:1), along with the respective interactions, was performed from the full factorial analysis of variance (3 x 2). When significant statistical difference occurred among variances, the LSD (Least Significant Difference) mean test was applied at a 5% significance level.

RESULTS AND DISCUSSION

Preliminary tests revealed that during the blending processing some balling of the coir fibers was observed in the furnish as a result of the fibers moisture uptake and the

tumbling action of the rotating drum paddles, making it difficult to obtain an uniform mixture of the material (Fig. 1a). Balling causes the binder not to coat all the fibers, which leads to low fiber to fiber bonding and therefore diminishes panels' properties. Additionally, after cement setting the panels presented several white spots on the surface as result of the compression of the balled fiber during pressing (Fig. 2b). These spots are denser than other panel regions, which causes several weak points and reduces panel properties considerably. The formation of these fiber aggregates during processing is cited by Carvalho et al. (2010) as a drawback of lignocellulosic fibers.

Thus, the particle size had to be reduced to dimensions cited previously in order to improve the blending process. Figure 1c shows the overall appearance of the coir fiber-cement composite. The panels presented a suitable fiber-cement cohesion and consistency, and they could be properly handled and machined. It was also observed that coir fiber could be suitably coated by the cement during the blending process, and coir fiber balling was almost completely eliminated.

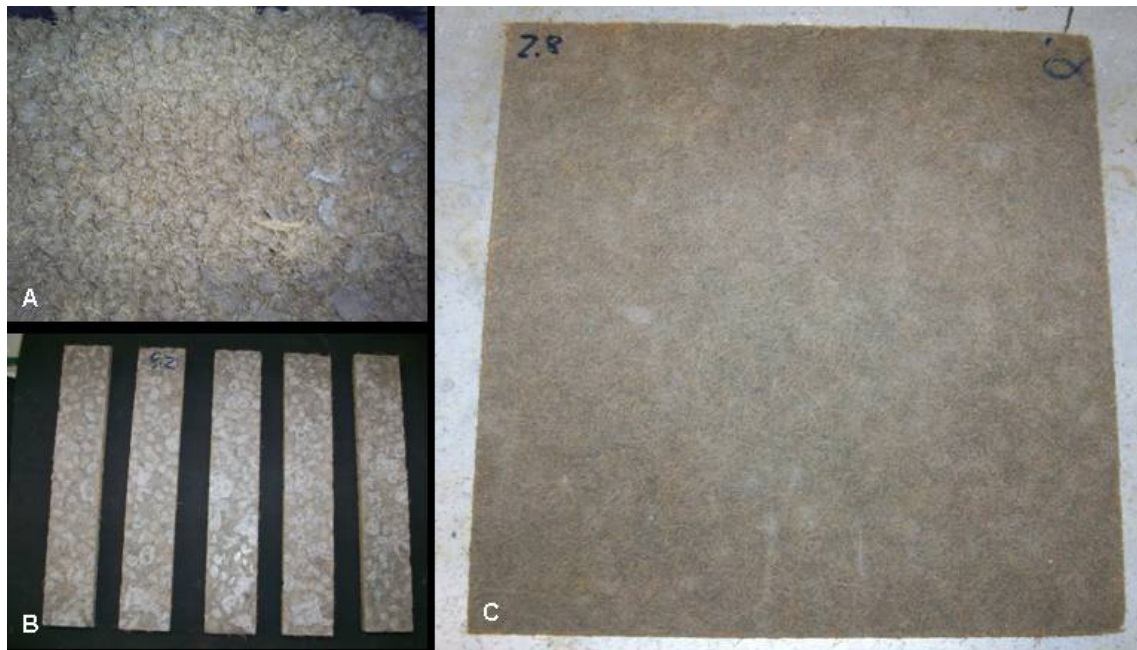


Fig. 1. Balling of the coir fibers (A and B) during blending before particle processing and overall appearance of the panel after adjustment of the particle dimensions (C)

The effects of treatments, cement: fiber ratios and their interactions on the physical and mechanical properties of the panels are shown in Table 3. Based on the results, one can note that hot water treatment resulted in means values of MOE, COMP and TS24 h considerably better than the values obtained for the others treatments.

Hot water treatment usually removes water-soluble extractives, sugars, starches, and other amorphous polymers, which play an important role on the inhibition of the cement hydration. Coir fiber presents a relatively high extractive content, as reviewed by Abdul-Khalil et al. (2006), while hemicelluloses content can reach up to 16%, as presented by Asasutjarit et al. (2007). According to Ferraz (2011) extractive content of the hot water treated fiber (3.68%) was lower in comparison with untreated fibers

(4.86%). In this context, it can be assumed that hot water treatment reduced the extractive content of the coir fibers, improving the quality of the cement hydration reaction, thus yielding a composite with better properties.

Nazerian et al. (2011) argued that impermeable hydrates are formed around unhydrated cement grains when extractives are present. Hot water treatment yielded a composite with higher MOE, while MOR was close to that reported by Asasutjarit et al. (2007), which ranged from 169 to 498 MPa for MOE and from 1.53 to 4.04 MPa for MOR.

Table 3. Physical and Mechanical Properties of Coir-fiber Cement bonded-Composites

Source of Variation	MOE	MOR	COMP	IB	TS		WA	
					2 h	24 h	2 h	24 h
A - Treatment	-----MPa-----				-----%-----			
1 - CaCl ₂	634 ^a	1.84 ^a	1.77 ^a	0.39 ^a	1.06 ^a	1.60 ^a	19.34 ^a	26.74 ^a
2 - NaOH	694 ^a	2.37 ^b	1.81 ^a	0.11 ^b	2.38 ^b	2.25 ^b	34.58 ^b	37.63 ^b
3 - Hot Water	1040 ^b	2.60 ^b	2.37 ^b	0.26 ^c	0.68 ^a	0.79 ^c	27.43 ^c	30.66 ^b
B – Cement: fiber ratio								
1 - 3:1	854 ^a	2.76 ^a	2.50 ^a	0.15 ^a	2.20 ^a	2.53 ^a	28.05 ^a	32.48 ^a
2 - 4:1	725 ^a	1.77 ^b	1.47 ^b	0.35 ^b	0.55 ^b	0.75 ^b	26.18 ^a	30.87 ^b
C - Interaction								
A1 x B1	410 ^a	2.09 ^b	NS	0.17 ^a	NS	NS	NS	NS
A1 x B2	885 ^a	1.21 ^a	NS	0.55 ^b	NS	NS	NS	NS
A2 x B1	565 ^a	2.49 ^b	NS	0.08 ^a	NS	NS	NS	NS
A2 x B2	641 ^a	1.83 ^a	NS	0.10 ^a	NS	NS	NS	NS
A3 x B1	1147 ^b	3.78 ^b	NS	0.19 ^a	NS	NS	NS	NS
A3 x B2	884 ^a	2.06 ^a	NS	0.35 ^b	NS	NS	NS	NS
Bison/ISO 8335	3000 ¹	9.00 ¹	-	0.40 ¹	1.2 – 1.8 ²		-	-

NS = The F value calculated was non-significant at 5% significance level. Different letters within each source of variation in each column indicate significant difference using the LSD mean test at $\alpha = 0.05$. ^{1,2} Minimum and maximum requirements, respectively.

The CaCl₂ treatment showed IB, WA₂, and WA₂₄ values greater than those obtained for the other treatments. According to Ahn and Moslemi (1980) and Dow (2006), the addition of CaCl₂ to the mixture tends to reduce the setting time of cement, to improve cement bonding with the lignocellulosic material and to increase the water resistance of these panels. According to the results of Table 3 the water absorption did not imply dimensional instability of the composite, since CaCl₂ presented the lowest TS values.

CaCl₂ is the most employed and studied additive and acts by accelerating the cement hydration before the inhibitory compounds can cause any adverse effect. It promotes early cement hydration, increasing the temperature of the mixture and yielding a more dense paste structure with smaller pores (Nazerian et al. 2011). Almeida et al. (2002), studying cement bonded composites made with *Orbignya* coconut fiber, found

that addition of CaCl_2 significantly improved all mechanical properties and also resulted in a more dimensionally stable composite. It can be observed that, in general, CaCl_2 treatment yielded composites with properties statistically close to those for NaOH composites. Nevertheless, NaOH treatment yielded a composite with the poorest fiber-cement adhesion, as it can be seen from the IB values.

As can be observed, composites produced with NaOH-treated coir fibers had mechanical properties between those resulting from CaCl_2 and hot water treatments, except for IB. However, these composites presented low dimensional stability, since TS and WA values were the highest. It is well known that alkaline treatments modify fiber surface, increasing roughness. These changes augment fiber surface area and cause some surface erosion. This additional surface area might be used as new pathways for water absorption, which can explain the highest WA values.

As a direct consequence, the TS was significantly improved and NaOH composite did not meet Bison (1978) boards type HZ nor ISO 8335 (1987) requirements (1.2-1.8%). Additionally, as mentioned above, these composites presented poor IB, which means that they did not have enough strength to resist the stress generated by the hygroscopic swelling of the coir fiber. The relationship between TS and IB properties was evaluated by Del Menezzi et al. (2007).

The MOE and MOR values found for all treatments in this study were similar to those reported by Olorunnisola (2009) for cement/coir panels manufactured with addition of CaCl_2 ; MOE values ranged from 479 to 1013 MPa and MOR from 1.2 to 2.2 MPa. However, both MOE and MOR results of this study were less than 3000 MPa and 9 MPa, respectively, which are considered suitable values according to Bison (1978). This kind of board is resistant to fungi, termites, fire, and weathering, and it also has good mechanical properties. According to Okino et al. (2004) the methodology adopted by BISON is a pioneer in producing mineral composites; for this reason it is used in comparisons in the literature. The standard ISO 8335 (1987) requires similar values for flexural properties.

It can be observed that the 3:1 ratio of cement: fiber was better for MOR and COMP. Although a MOE value of the 3:1 ratio was higher than that found for the 4:1 ratio, this difference was not statistically significant. Moslemi and Pfister (1987) argued that when lignocellulosic fibers take up more volume in a panel (i.e. low cement: fiber ratio), the regions of stress concentration around the adjacent particles are diffused, resulting in an increase in the applied stress. The values found for MOR are consistent with the literature. Latorraca and Iwakiri (2000) and Zhou and Kamdem (2002) observed a reduction in MOR with the increase in the cement: wood ratio.

It is well known that cement is a stiff material; thus the 4:1 ratio panels would be expected to be stiffer than the 3:1 panels. Nevertheless, the results indicated that increasing the amount of cement did not significantly improve the panel stiffness. Likewise, cement has very good compression strength, but COMP values were improved when the amount of cement in the mixture was reduced, similarly to what happened with MOR. During the bending testing the highest compression and tension stresses are located at the upper and at the lower fibers of the beam, respectively. Usually, the ultimate stress capacity of a beam is determined by its tension strength. Nevertheless, in this present study, the ultimate stress in bending might have been governed by the

compression strength of the panel: in Table 3 MOR and COMP values are quite close, except for the NaOH treatment.

The IB was positively influenced by the increase in the cement:coir fiber ratio. According to Del Menezzi et al. (2007) the IB evaluates tension strength perpendicular to the panel surface. In other words, IB measures the bonding quality of the matrix formed by the lignocellulosic and the cement. As the cement amount increased, the coating of the coir fibers increased, which improved bonding. In fact, Zhou and Kamdem (2002) observed that an increase in the cement:wood ratio improved IB.

As for the physical properties, it was observed that the higher the ratio (4:1), the better the results. It was found that TS2 and 24h were less pronounced in those panels with a 4:1 cement:coir fiber ratio. Since 4:1 panels presented higher IB values than 3:1 panels, this means they were stronger in their resistance to fiber swelling stresses, which cause thickness swelling. WA2h and 24h values were not affected by the cement:coir ratio and, in this case, there were no statistically significant differences between the ratios. A similar analysis was performed by Moslemi and Pfister (1987) when they studied the influence of the cement:wood ratio and the type of cement in the properties of wood-cement panels. The authors observed that the WA property was not affected by the variation in the cement:wood ratio from 2:1 to 3:1.

The treatment x ratio interaction was significant for the MOE, MOR, and IB properties. It should be mentioned that increasing the cement:coir ratio from 3:1 to 4:1 (except for the CaCl₂ treatment, which benefited from the ratio increase) resulted in better results only in the IB, which was heavily influenced by this factor. A similar analysis was performed by Latorraca and Iwakiri (2000), who observed a reduction in the mean values of the MOE, MOR, and COMP properties and an increase in the panel IB when the cement:wood ratio was increased from 2.5:1 to 3:1. It was found that for the treatment with NaOH, the properties tended not to be affected by ratio variation (except for MOR, which was higher for the 3:1 ratio). The hot water treatment, in turn, benefited from the ratio reduction as the properties tend to be improved.

The effect of treatments on the fiber surface was analyzed using the scanning electron microscope (SEM). Figure 2 compares photomicrographs of natural and treated coir fibers. The natural fiber (2a) presents a glossy surface that can be considered as a result of the presence of compounds such as oil, wax and extractives, as also mentioned by Carvalho et al. (2010).

The surface of the hot water treated fiber (2b) presented some roughness, probably by the partial removal of those compounds. The surface of NaOH treated fiber is shown in Fig. 2c. In contrast to natural fiber, the surface of NaOH-treated fiber was opaque and eroded as result of the action of an alkaline environment, which can clean the surface, degrade the lignin, and remove starches, sugars, and hemicelluloses.

The presence of those inhibitory compounds on the natural fiber surface confirms the result obtained in our previous work (Ferraz 2011), in which the natural fiber was rated as "extreme inhibition" in the cement hydration assay. When hot water treatment was applied, the compatibility between coir fiber and cement was rated as "mean inhibition". On the other hand, NaOH treatment reduced considerably the inhibitory effect of the coir fiber on the cement hydration, thus changing the rate to "low inhibition".

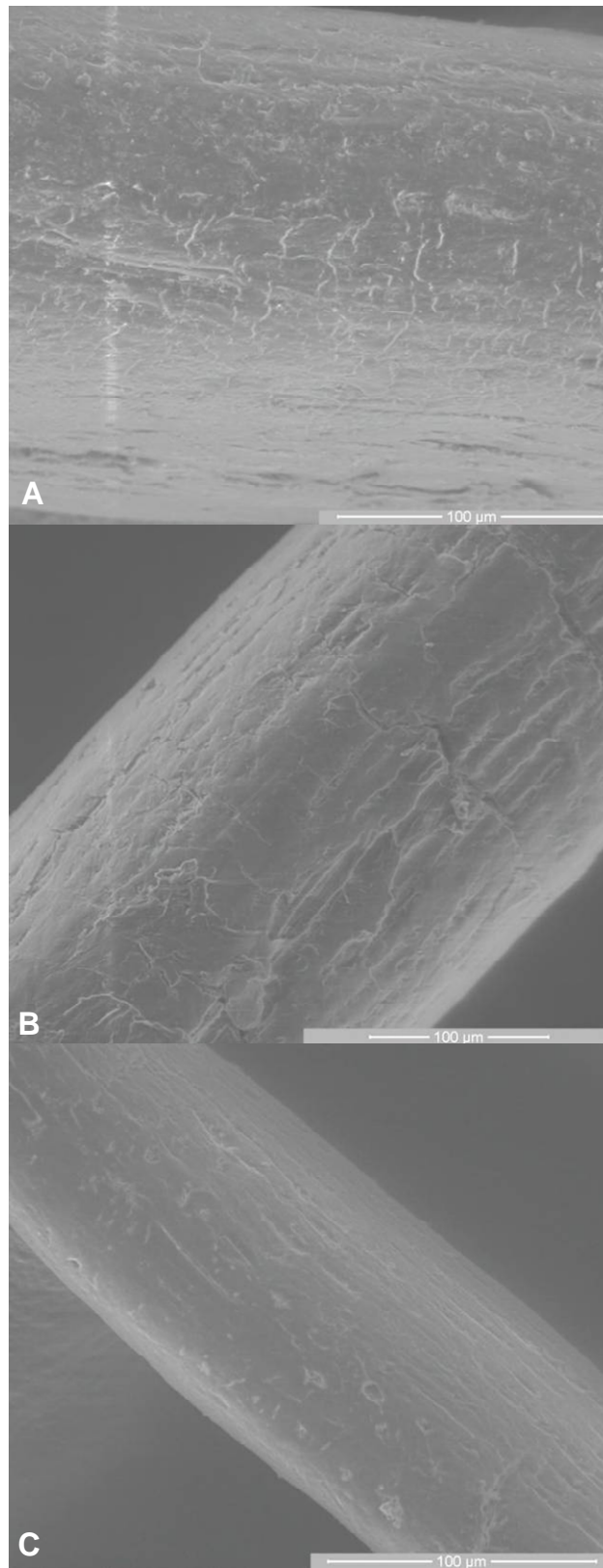


Fig. 2. SEM photomicrographs of natural coconut coir (A), after treatment with hot water (B) and with NaOH (C)

Taking in account these ratings, it would be expected that NaOH treatment would yield composites with better properties. However, contrary to expectations, there was a reduction in the mean values of MOE and IB. This treatment also negatively affected the physical properties, resulting in higher mean values for both TS2 and 24h and WA2 and 24h. These results might be explained by erosions on the fiber surface. Therefore, the production of cement-coir composites with this type of treatment is not recommended.

CONCLUSIONS

1. There was an isolated effect of all treatments on the physical and mechanical properties, where the fibers treated with hot water showed better MOE, COMP, TS2h, and TS24h results; the addition of CaCl₂ showed better results in IB, and NaOH treatment resulted in lower values of mechanical properties when compared to other treatments, negatively affecting the physical properties.
2. Increasing the cement:coir ratio had a negative effect on the panels' mechanical properties. The ratio variation showed an effect on the panel properties: the 3:1 ratio showed better results for MOR and COMP; the 4:1 ratio, in turn, showed better results for IB and TS 2 and 24 hours.
3. Finally, it could be concluded that in general the effects of hot water treatment were more evident when 3:1 cement:coir fiber ratio was used; on the other hand, the addition of CaCl₂ was more effective in composites made from 4:1 cement:coir fiber ratio; the alkaline treatment (NaOH) was not affected by any tested ratio.

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