COLD-PRESSED HYBRID COMPOSITES REINFORCED WITH COIR FIBRES AND CEMENT PARTICLES: A STATISTICAL APPROACH

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https://doi.org/10.21452/bccm4.2018.02.19

Abstract: The mechanical properties of polyester hybrid composites reinforced with treated and untreated coir fibres and 15 wt.% of cement microparticles at different locations were investigated in this work. A Full Factorial Design ($2^1 3^1$) was conducted to identify the effect of the chemical treatment of coir fibres and location of cement inclusions on the tensile (and flexural) strength and modulus. In general, the alkaline treatment led to increased tensile properties and flexural strength compared to untreated fibre composites. The inclusion of cement particles, especially added throughout the whole sample, increased the tensile and flexural strength and tensile modulus of untreated coir fibre composites. A significant increase in flexural modulus was observed only in composites made with treated coir fibres along with the incorporation of cement particles in the whole sample.

Keywords: hybrid composites, coir fibre, polyester, cement, chemical treatment, mechanical properties.

1. INTRODUCTION

Natural fibres such as flax, hemp, jute, sisal, coir, and henequen have been used since ancient times in a wide variety of products, ranging from clothing to house roofing [1-3]. The investigation of natural fibres as reinforcements in hybrid composite materials has recently risen as sustainability and environmental issues are considered, owing in particular to their renewability and biodegradability. [4-6].

Coir is a low cost, versatile, renewable and biodegradable lignocelullosic fibre obtained from the coconut palm (*Cocos nucifera*), which is very abundant in coastal areas of tropical countries and plays an important role in the economy of these regions [2, 7-8]. Apart from its conventional applications (e.g., cordage, cushions, sacking, floor-furnishing [7], mats, carpets and seat covers [9]), coir has recently been used as a reinforcement in cement, concrete, particleboard and polymer composites due to their valuable properties such as renewability, resilience, rigidity, toughness, elongation at break and weather resistance [7]. These composite materials may potentially be used in the aeronautics, leisure, construction, sport, packaging and automotive industries [10-11].

An important issue, however, is fibre-matrix compatibility [12]. Natural fibres are in general of hydrophilic nature while the polymeric matrices are hydrophobic, resulting in low fibre-matrix adhesion, which plays an important role in the final mechanical performance [13]. Such compatibility may be substantially improved through the chemical modification of the fibre surface [14-16]. Alkaline treatments are currently considered the most economical and effective technique, removing impurities such as pectin, fats and lignin. This process increases the roughness of the fibre surface and consequently improves fibre-matrix mechanical interlocking [17].

In addition, the thermal stability and stiffness of fibre-reinforced composites improve upon the incorporation of a second rigid particulate phase, such as SiO_2 micro- or nanoparticles, glass, Al_2O_3, $Mg(OH)_2$, $CaCO_3$ and carbon nanotubes [18-20]. According to Silva *et al.* [21], when the composite material is under stress, the crack propagation is prevented across the locations where the particles are concentrated due to their high strength. Therefore, additional stress is required for the crack propagation through the fibre–particle or particle–matrix interfaces, which increases the mechanical strength of the composites. However, this increase strongly depends on particle size, particle content and interface adhesion [20].

This work investigates, via ANOVA and Design of Experiments (DoE), the effect of the treatment of coir fibres and the location of microparticles on the mechanical properties of hybrid short coir fibre composites with Portland cement particle inclusions. A preliminary experiment was conducted to identify the effect of particle type (silica or cement) and particle mass fraction (5, 10 or 15 wt%) on the mechanical properties of the matrix phase. The best experimental condition was used as a reinforced matrix phase for the fabrication of the hybrid composites.

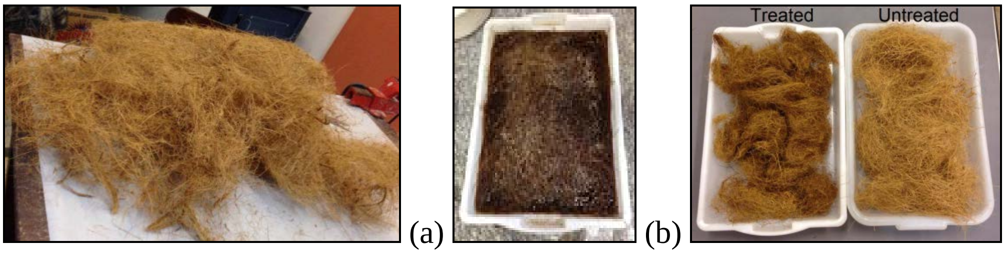
2. MATERIALS AND METHODS

2.1 Materials

Polylite polyester resin 10316-10 and Methyl Ethyl Ketone Peroxide hardener were supplied by Reichhold (Brazil). Coir fibres were supplied by *Deflor Bioengenharia* (Brazil), being treated with sodium hydroxide (NaOH, 99%, sourced from Sulfal - Brazil). Portland cement (ASTM III) and silica microparticles were supplied by Holcim (Brazil) and *Moinhos Gerais* (Brazil), respectively. Both particles were classified by sieving process in a particle size range of 44 - 26 μm.

2.2 Coir fibre treatment

Coir fibres in pristine conditions were manually cleaned for the complete removal of plant debris (Figure 1a). The fibres were subsequently immersed in a 10 wt.% sodium hydroxide solution at room temperature for 15 h (Figure 1b) and then washed in water. Finally, the treated coir fibres were oven-dried at 60 ^{o}C for 72 h (Figure 1c).



(a) (b) (c)

Figure 1. Coir fibres: (a) in pristine condition, (b) under sodium hydroxide treatment, (c) ready to be used as a reinforcing material.

2.3 Design of experiment

The factors and levels investigated in the experiment were set based on preliminary tests. A Full Factorial Design ($2^1 3^1$) was established to investigate the effect of the fibre chemical treatment (treated and untreated) and cement location (no particles; upper (under compression) beam side and whole sample) on the flexural and tensile strength and modulus of hybrid composites, resulting in 6 experimental conditions (E.C.), as shown in Table 1. Minitab v.17 software was used to perform the Design of Experiment (DoE) and Analysis of Variance (ANOVA) techniques.

Table 1. Full Factorial Design ($2^1 3^1$).

E.C.	Chemical Treatment	Cement Location	E.C.	Chemical Treatment	Cement Location
1	Untreated	No particles	4	Treated	No particles
2	Untreated	Upper side	5	Treated	Upper side
3	Untreated	Whole sample	6	Treated	Whole sample

The constant factors considered in this experiment were established based on a previous work [22]: type of matrix (polyester), fibre grammage (300 g/m^2), fibre volume fraction (30%), particle mass fraction (15 wt.%), number of fibre layers per composite plate (3 layers), cold-pressing time (22 h), compaction pressure (654 kPa) and curing time (28 days).

Ten specimens (five for tensile and five for flexural tests) were fabricated for each of the 6 experimental conditions. Two replicates were considered, running a total of 120 specimens.

2.4 Manufacturing process

The composite materials were fabricated using the hand lay-up technique followed by cold compaction. The treated/untreated short coir fibres (20 – 180 mm) were weighed according to the required grammage (300 g/m^2) and randomly distributed into a 300 x 300 mm metallic mould (Figure 2a). A preliminary uniaxial pressure of 654 kPa was applied for 2 min to obtain a randomly oriented coir fibre fabric (Figure 2b). An aluminium plate (Figure 2c), covered with a thin wax layer as a release agent, was placed into the mould to provide good surface finish. The polymer matrix was prepared by first mixing the resin and hardener according to the fabricant’s orientation (2 wt%). The microparticles were subsequently added and hand-mixed for 5 min at room temperature (~23 ^{o}C). Three fabric layers were assembled into the mould and the polymer matrix (modified and non-modified, depending on the experimental condition considered) was uniformly spread over the coir fibre layers (Figure 2d). A lid was then placed on the mould and a pressure of 654 kPa was applied for 22 h (Figure 2e). The material was subsequently demoulded (Figure 2f) and placed in a plastic container to prevent moisture absorption for curing (28 days). The composite plate was finally cut (Figure 2g) according to ASTM standards and tested.

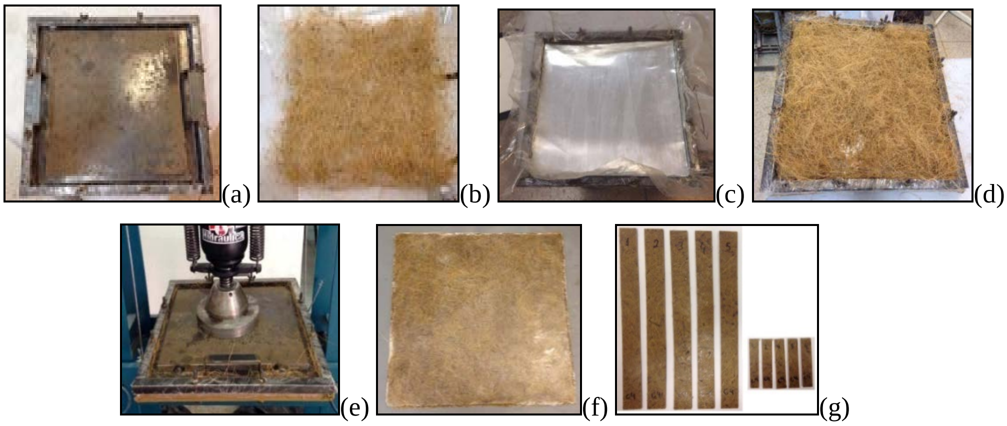


Figure 2. Fabrication process of the composites: (a) metallic mould, (b) fabric of random coir fibres, (c) aluminium plate, (d) fabric arranged inside the mould, (e) cold pressing, (f) composite plate and (g) specimens for testing.

2.5 Characterisation

The mechanical properties of the reinforced polymer matrix with 5, 10 and 15 wt.% Portland cement or silica particles was characterised via flexural, tensile and compressive tests, performed according to ASTM D790-15 [23], ASTM D638-14 [24] and ASTM D695-15 [25], respectively. Tensile and flexural tests were also conducted for the characterisation of the hybrid composites, following the recommendations of ASTM D3039-14 [26] and ASTM D790-15 [23], respectively. A Shimadzu AG-X Plus test machine equipped with a 100 kN and velocity of 2 mm/min load cell was used to perform the mechanical tests.

3. RESULTS AND DISCUSSION

3.1 Matrix phase characterisation

Table 2 presents the mean and standard deviation of the mechanical test results for the polyester polymer in pristine conditions and modified with silica and Portland cement particles.

The incorporation of microparticles decreased the tensile and flexural strength of the matrices, which can be attributed to the low particle-matrix interfacial adhesion and stress concentration at larger deformations. On the other hand, the compressive strength was increased due to the greater capacity of these microparticles to withstand stress under compressive loads. These results justify the choice of using microparticles in the upper beam side of the laminates, since the specimens will be under compressive stress when subjected to flexural loading, compensating for the low compressive strength of the fibres. In addition, the use of microparticles, especially at higher mass fractions, may contribute to the enhancement of the mechanical modulus of the composites, due to their inherent high stiffness.

In general, the inclusion of cement microparticles provided better results on the mechanical properties of modified matrices when compared to silica results. Similar findings have been reported by Torres *et al.* [27] and Martuscelli *et al.* [28], which evaluated the modified epoxy polymer matrix with silica and cement microparticles under compression. This mechanical enhancement has been attributed to the presence of hydrated cement products, which stiffen the polymer [28].

It is also observed in Table 2 that the modified matrix containing 15 wt% of cement particles generally provided better results in comparison to the other amounts, which justifies the choice of this percentage as the reinforcement level used in the hybrid biocomposites.

Table 2. Mechanical properties of modified and non-modified matrices.

Type of Matrix (wt.%)		Tensile		Compressive		Flexural	
		Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)
Ref.	0	36.89 (±3.55)	2.43 (±0.01)	60.30 (±1.41)	1.93 (±0.03)	54.45 (±1.94)	2.22 (±0.11)
Silica	5	30.42 (±2.86)	2.42 (±0.02)	63.09 (±2.36)	2.11 (±0.07)	42.84 (±3.11)	2.44 (±0.04)
	10	25.04 (±2.58)	2.36 (±0.03)	65.62 (±1.96)	2.30 (±0.01)	48.09 (±0.89)	2.58 (±0.02)
	15	21.19 (±0.22)	2.51 (±0.18)	69.87 (±2.77)	2.45 (±0.04)	33.22 (±1.62)	2.69 (±0.09)
Cement	5	31.33 (±0.02)	2.39 (±0.01)	98.08 (±0.88)	2.81 (±0.01)	33.07 (±2.37)	2.50 (±0.08)
	10	30.67 (±0.02)	2.59 (±0.12)	106.79 (±3.95)	3.15 (±0.22)	37.76 (±1.66)	2.70 (±0.08)
	15	32.32 (±0.40)	2.66 (±0.15)	107.18 (±5.49)	3.34 (±0.16)	45.31 (±0.19)	2.81 (±0.06)

3.2 Statistical results

Table 3 presents the mean and standard deviation of the two replicates considered for the mechanical tests of hybrid coir fibre-reinforced composites with 15 wt.% of cement inclusions. Table 4 shows the Analysis of Variance (ANOVA) results obtained through Design of Experiment (DoE). If the P-value is less than or equal to 0.05, this implies that the effect (or interaction of effects) is statistically significant within a 95% confidence level (underlined values in Table 4). Values in bold were interpreted via effect plots. The values of R^2-(adjusted), ranging from 73.81% to 96.88% (Table 4), imply a satisfactory predictability for the underlying statistical model used. ANOVA was validated by the Anderson-Darling normality test, exhibiting P-values greater than 0.05 (0.503 – 0.977), implying that the data follow a normal distribution.

Table 3. Mechanical properties of the composite materials.

Experimental Condition		Tensile		Flexural	
		Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)
Replicate 1	1	9.25 (±0.32)	2.25 (±0.08)	18.27 (±0.77)	2.55 (±0.41)
	2	9.71 (±1.28)	3.48 (±0.23)	20.56 (±1.71)	2.55 (±0.15)
	3	12.11 (±1.84)	3.98 (±0.25)	24.15 (±2.25)	2.66 (±0.15)
	4	10.82 (±0.16)	2.61 (±0.14)	29.65 (±4.85)	2.63 (±0.32)

	5	14.14 (±0.15)	4.58 (±0.45)	25.67 (±1.12)	2.57 (±0.11)
	6	10.63 (±1.50)	4.32 (±0.51)	25.48 (±1.60)	2.89 (±0.11)
Replicate 2	1	9.30 (±0.96)	2.20 (±0.22)	15.50 (±1.33)	2.55 (±0.30)
	2	9.47 (±0.81)	3.54 (±0.48)	19.01 (±2.81)	2.56 (±0.18)
	3	11.59 (±1.59)	3.53 (±0.30)	24.50 (±2.03)	2.55 (±0.30)
	4	10.98 (±1.36)	2.60 (±0.01)	30.60 (±5.32)	2.39 (±0.24)
	5	13.18 (±1.81)	4.36 (±0.40)	26.30 (±1.60)	2.61 (±0.13)
	6	10.61 (±0.50)	4.18 (±0.59)	26.91 (±1.89)	3.04 (±0.40)

Table 4. Analysis of Variance (ANOVA).

ANOVA		P-value ≤ 0.05			
Experimental Factors		Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)
Main Factors	Chemical Treatment	0.000	**0.000**	0.000	0.060
	Cement Location	0.001	**0.000**	0.046	0.014
Interaction.	Chemical Treatment x Cement Location	**0.000**	0.076	**0.001**	**0.040**
	R^2 - adj	95.57%	96.88%	94.74%	73.81%
	P-value (Anderson Darling) ≥ 0.05	0.503	0.641	0.977	0.764

3.2.1 Tensile Test

Figure 3a presents the second-order interaction effect plot for the mean tensile strength. Letters stand for Tukey's comparison test, in which similar letters belong to the same group, i.e., equivalent means.

The chemical treatment provided an increase in tensile strength, when compared to composites manufactured with untreated fibres. This behaviour may be attributed to enhanced fibre bridging effects, since the alkaline treatment increases fibre roughness. In fact, Mulinari *et al.* [29], reports that the alkaline treatment enhances fibre roughness and fibre-matrix adhesion, owing to the removal of the superficial layer of coir fibres, increasing the exposure area of fibrils and surface pits (tyloses). Particles may also increase the composite strength, acting as barriers against crack propagation and enhancing fibre-matrix interlocking. It is worth noting a relevant increase (42.44%) observed for treated coir fibres with cement inclusions in the upper half of the laminate. A significant drop in strength (22.5%), however, was observed for treated fibre composites with microparticle inclusions in the whole laminate. Such behaviour demands further investigations.

Figure 3b presents the main effect plots for mean tensile modulus. Figure 3b, item a, reveals that the tensile modulus of treated coir fibre composites are 19.34% higher relative to untreated ones, which is attributed to enhanced interlocking as well as an increase in fibre stiffness after the alkaline treatment, as reported by Oliveira *et al.* [22]. Figure 3b, item b, shows a substantial increase in stiffness (65.22%) when cement microparticles were incorporated into the upper beam side and in the whole composite, with no difference between means as shown by the same group A. These results are consistent with the matrix characterisation results (Table 2), which presents an increase in modulus after the incorporation of 15 wt.% of cement particles, attributed to the high stiffness of the microparticles. In addition, cement particles also enhances fibre-matrix interaction through the interlocking effect.

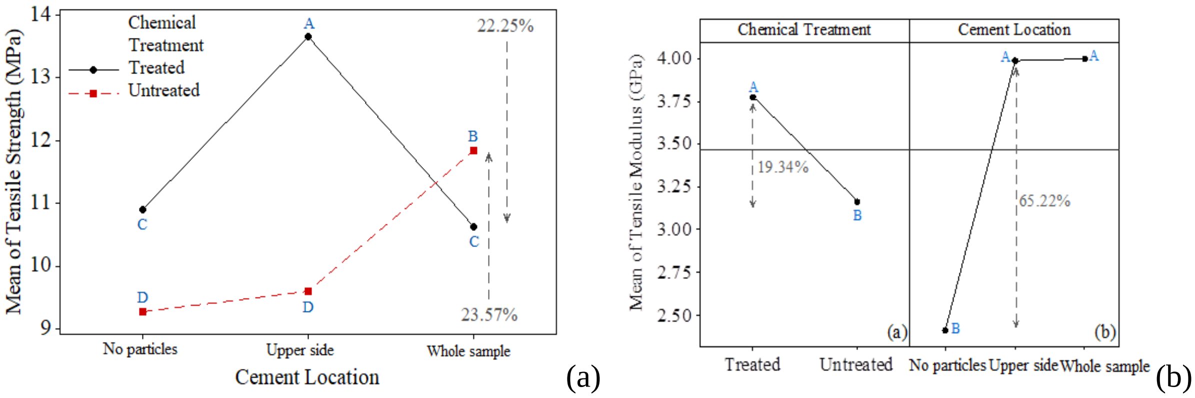


Figure 3. Effect plot for the mean: (a) tensile strength and (b) tensile modulus of the composites.

3.2.2 Flexural Test

Figure 4a presents the second-order interaction analysis for the mean flexural strength. A substantial increase in strength was observed when treated coir fibres were used, especially for those non-particulate composites (78.41%), attributed to enhanced fibre-matrix compatibility and bridging effects after the alkaline treatment. However, the inclusion of cement microparticles in the upper beam side and in the entire sample of treated composites led to a decrease in strength of 15.93%. An opposite behaviour was observed for untreated fibre composites, with an increase in flexural strength upon the inclusion of cement microparticles, which may act as barriers for crack propagation.

Figure 4b shows the plot of second-order interaction effects for the mean flexural modulus. Untreated and treated coir fibre composites present a similar behaviour at all levels (group B), except when the particles were added to the whole sample (group A). This increase of 13.82% is attributed to an increase in stiffness of post-treated fibres [22] as well as of the reinforced matrix phase. This also implies that the particles may play an important role even when added to the lower beam side (under tensile stress), which can be attributed to enhanced interlocking effects.

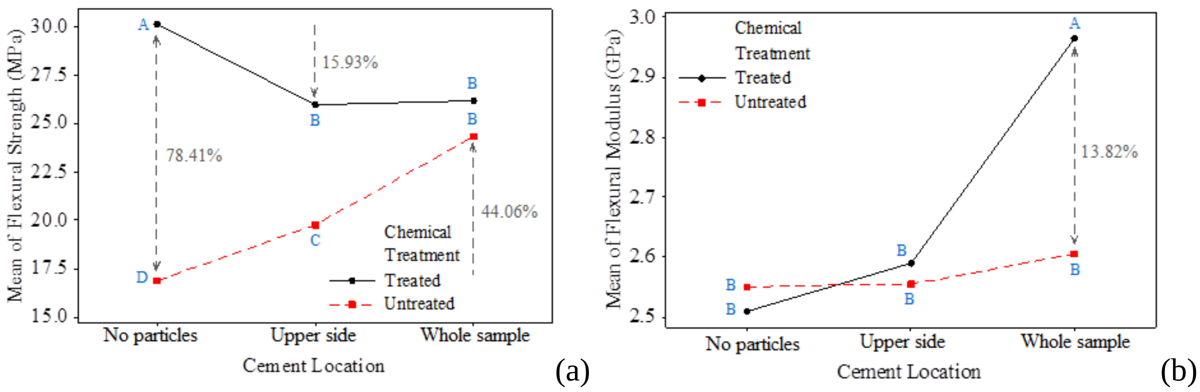


Figure 4. Effect plot for the mean responses: (a) flexural strength and (b) flexural modulus of the composites.

4. CONCLUSIONS

A Full Factorial Design was used to investigate the mechanical properties of hybrid composites reinforced with treated and untreated coir fibres and cement microparticles in different locations. The matrix phase was also characterized. The conclusions are described as follows:

- The incorporation of cement in the polyester matrix led to the increase of mechanical properties in relation to the silica particles, especially when it was considered 15 wt.%.
- The inclusion of particles reduced the tensile and flexural strength of the matrices, except for the compressive strength. In contrast, an increase in mechanical moduli was observed for all reinforced matrices.
- In general, the chemical treatment enhanced the tensile properties and flexural strength of the composites. However, a slight decrease in strength was obtained when the particles were incorporated throughout the treated fibre laminates.
- The cement particles promoted a substantial increase in tensile modulus for all laminates because of their high stiffness. A positive effect was also evidenced for the tensile and flexural strength of untreated coir fibre composites.
- A slight increase in flexural modulus was observed only in composites with treated coir fibres and cement added throughout the sample.

ACKNOWLEDGMENTS

The authors would like to thank CAPES (MSc scholarship), CNPq (PP-306767/2016-3) and FAPEMIG (PPM-00075-17) for the financial support provided.

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