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ARTICLE

Cementitious Composites Containing Multifunctional Sugarcane Fibres

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ABSTRACT

This work investigates the reuse of natural (SCB) and minopropyltriethoxysilane-modifed (MSCB) sugarcane bagasse fbres in cementitious composites. ugarcane bagasse fbres are pre-used in the treatment of motor oil contaminated effluents. A full factorial design is used to identify the effects of fbre type (SCB and MSCB), fbre length (0.6 and 1.2 mm), fbre amount (1 and 2 wt%) and fbre condition (before and after oil filtration) on apparent density, water absorption, apparent porosity, ultra-pulse velocity, dynamic modulus, flexural strength and modulus. SCB fibres lead to increased apparent density compared to MSCB fibre reinforced composites. MSCB fibres contribute to reduce composite porosity, leading to higher mechanical properties. The smaller area of MSCB fibres promotes a larger amount of cementitious phase per unit volume, thus increasing the strength of the sample. Longer sugarcane fbres (1.2 mm) have a larger surface area, leading to a higher fibre concentration per unit volume, which increases water absorption. The amount of fibre has no significant effect on mechanical and physical responses. Composites made with 2 wt% 0.6 mm long MSCB fibres achieve promising results for non-structural civil engineering applications.

1. Introduction

The construction industry requires significant amounts of raw materials and energy for its development, generating a significant amount of pollution, especially CO_2 emissions and waste. The search for sustainable solutions includes the correct use of industrial and agro-industrial materials. New approaches to efficient projects have been claimed to the development and use of natural building materials[1].

Some advantages of the use of natural fibres in composite materials are lower apparent specific gravity, higher porosity, moderate tensile and impact strength, greater fracture control and ductile failure, good thermal and acoustic insulation[2]. Further benefits include fibre biodegradability, reduced costs and damage to manufacturing machines compared to synthetic fibres and reduced CO_2 emissions. On average, the production of natural fibres

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requires 60% less energy than the production of glass fibres used as composite reinforcements[3]. On the other hand, cement-based matrices are fragile and may have small cracks even under small elastic loads or under deformation. Fibre embedding has been used to reduce the propagation of the matrix crack since the fibres can bind the sample parts between the cracks. As a result, fibre inclusions may increase sample toughness, tensile strength and impact resistance[1][3].

A variety of natural fibres, such as sisal, flax, coir, hemp, straw, linen, bamboo, cork, etc., has been used to replace synthetics in cementitious and polymeric composites for applications especially in the construction and automotive industries, respectively[2]. The performance of fibre-reinforced composites depends not only on composition parameters, such as fibre geometry, length, distribution and orientation, interfacial transition zone (ITZ) and matrix/fibre volume fraction, but also in the manufacturing process used[4][5].

Metha and Monteiro reported an inverse relationship between porosity and mechanical strength in solids. In a multiphase material, the porosity of each component is a limiting factor of the strength of the composite. Although the water/cement (w/c) ratio is the factor that most affects the porosity of the matrix phase and the ITZ, other parameters, such as densification, curing conditions (cement hydration degree), aggregate dimensions and mineralogy, admixtures and type of load, contribute to the mechanical properties of cementitious composites[6].

In addition to reinforcing composite materials, natural fibres can also be used as waste absorbing material. Water scarcity poses a challenge to the sustainable development of industrial and agricultural activities. Widespread production of industrial effluents and awareness of the impact of their effects on nature have forced industry to adopt new environmental policies[7]. One method widely applied in recent decades for the treatment of oil contaminated water is the use of absorbent materials that easily remove and recover oil. Many materials can be used at this stage, including natural organics from living organisms, known as biosorbents, with high availability and low cost. Some biosorbents, such as sawdust, rice husk, cellulose, cotton, peat, sugarcane bagasse, straw and corn cob are biodegradable and highly capable of floating, being suitable for treating oil contaminated water. These fibres are abundantly produced in Brazil, however, most of them are discarded due to lack of suitable applications[8][9].

While natural fibres may be an alternative for oil absorption, they have limitations because they also tend to absorb water. This side effect occurs due to the presence of hydroxyl groups in cellulose, hemicellulose and lignin. These groups, in particular, are responsible for the hygroscopic characteristic of lignocellulosic materials. However, the hygroscopic properties of such fibres can be reduced by surface modifications[10]. One possible approach is to replace the hydroxyl functional group with hydrophobic groups through chemical reactions [11].

This work investigates the use of multifunctional sugarcane bagasse fibres in pristine condition (SCB) and modified with aminopropyltriethoxysilane (MSCB) as reinforcement of cementitious composites (mortars). These fibres, which were extracted from sugarcane after ethanol production, were previously used as biosorbents in the treatment of motor oil contaminated effluents[11]. Sugarcane fibres without absorbed oil are also evaluated as a control group of reinforced samples. Furthermore, this work contributes to extending the useful life of multifunctional sugarcane bagasse, previously used as raw material for the production of ethanol and oil biosorbents, as well as providing a reinforced cementitious mortar for secondary structural in construction engineering.

2. Materials and Methods

2.1 Materials

Portland cement (ASTM III), quartz sand particles and sugarcane bagasse are supplied by Holcim (Brazil), Omega Mining Company (Brazil), and a local ethanol producer (Brazil), respectively. Aminopropyltriethoxysilane (APTS, 98%, $(H_2NCH_2CH_2CH_2)\text{-}Si\text{-}(OCH_2CH_3)_3$) is supplied by Dow Corning (Brazil) and used without further purification. The properties of a cement-reinforced mortar without sugarcane bagasse inclusions are presented in Table 1.

Table 1. Mean Values for the Cementitious Mortar Without Sugarcane Bagasse Inclusions

Property	Mean ± standard deviation
Flexural strength (MPa)	6.47 ± 0.41
Static modulus of elasticity (GPa)	0.42 ± 0.04
Pulse velocity (m/s)	370.76 ± 6.39
Dynamic modulus of elasticity (GPa)	0.28 ± 0.01
Water absorption (%)	7.39 ± 0.09
Apparent porosity (%)	14.73 ± 0.20
Apparent density (g/cm³)	2.35 ± 0.02

2.2 Methods

A Full Factorial Design (2^4) is conducted in this work with the following factors (levels): fibre type (pristine-SBC and modified-MSCB), fibre length (0.6 and 1.2 mm), fibre amount (1 and 2%wt as a replacement of aggregates), and

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fibre condition (before and after oil absorption), as shown in Table 2 according to the Design of Experiment (DoE) and Analysis of Variance (ANOVA) techniques. DoE and ANOVA are performed using Minitab v18 software to verify the significance of each factor on the investigated responses.

Table 2. Factors and Experimental Levels ($2^4 = 16$ Conditions)

Setup	Factors / Levels			
	Fibre type	Fibre length (mm)	Fibre amount (wt%)	Fibre condition
C1	SCB	0.6	1	Before oil absorption
C2	SCB	0.6	1	After oil absorption
C3	SCB	1.2	1	Before oil absorption
C4	SCB	1.2	1	After oil absorption
C5	SCB	0.6	2	Before oil absorption
C6	SCB	0.6	2	After oil absorption
C7	SCB	1.2	2	Before oil absorption
C8	SCB	1.2	2	After oil absorption
C9	MSCB	0.6	1	Before oil absorption
C10	MSCB	0.6	1	After oil absorption
C11	MSCB	1.2	1	Before oil absorption
C12	MSCB	1.2	1	After oil absorption
C13	MSCB	0.6	2	Before oil absorption
C14	MSCB	0.6	2	After oil absorption
C15	MSCB	1.2	2	Before oil absorption
C16	MSCB	1.2	2	After oil absorption

The absorbed oil fibres are prepared using contaminated effluents from a local engine rectifier company (Brazil). The mixing ratio between sugarcane and contaminated effluent is 10 g of sugarcane/litre of effluent under stirring for 24 h. This procedure is carried out considering one hundred grams of fibres per batch. Subsequently, the fibres are filtered and dried at room temperature as described in the previous work[11]. Samples are prepared according to ASTM C192[12]. A mortar matrix consisting of one-part cement, three parts standard sand and water/cement ratio of 0.55 is used. The particle size distribution of quartz-sand is: 20 wt% of 4-20 US-Tyler (coarse), 50 wt% of 20-50 US-Tyler (medium) and 30 wt% of 50-200 US-Tyler (fine). The mixture is inserted into 160 x 40 x 40 mm^3 prismatic moulds. An electromagnetic vibrator is applied for 8 minutes in the moulds to ensure a uniform distribution of sugarcane bagasse fibres and to improve the homogeneity of the present results. The samples are covered with a plastic film to prevent moisture loss, being demoulded after 7 days and kept at room temperature. Five (5) samples are produced per experimental condition and per replicate, resulting in 160 samples for each group of experiments (mechanical and physical tests).

Samples are tested after 28-days of curing under three-point bending load according to ASTM C348-14[13] at a crosshead speed of 0.5 mm/min to obtain flexural strength (σ_f). The bending test is performed on a 100 kN Shimadzu AGX test machine. The static modulus of elasticity (E_s) is then calculated based on ASTM C580-02[14]. Pulse velocity (ν) and dynamic modulus (E_d) are obtained using portable ultrasonic non-destructive equipment (PUNDIT) following ASTM C597-09[15] recommendations. Physical properties such as water absorption (W_{abs}), apparent porosity (P_a) and apparent density (ρ_a) are determined according to BS 20545-3[16].

3. Results

The Design of Experiment (DoE) verifies whether factors such as fibre type (SCB and MSCB), fibre amount (1 and 2% wt), fibre length (0.6 and 1.2 mm) and fibre condition (with and without absorbed oil) significantly affect the evaluated responses. Analysis of variance (ANOVA) is shown in Table 3. P-values below 0.05, underlined in Table 3, indicate significant factors and interactions within a 95% confidence level. Due to the small percentage contributions of interactions, only main effects will be interpreted in this paper. The main effect plots for all properties analysed are shown in Figures 1 to 7, which show the mean values for each level of significant factors according to the ANOVA. The R^2 values given in Table 3 show that the model adjustments are satisfactory for all variables. The assumption of data normality is an additional requirement to validate the use of ANOVA. The Anderson-Darling test is used to verify data normality, in this case, the P-value must be greater than 0.05. All data follow a normal distribution (P value > 0.05), validating the ANOVA model (Table 3).

Table 3. Analysis of Variance Results – P-values

	Factors	ρ_a	W_{abs}	P_a	ν	E_d	σ_f	E_s
Main	Fibre Type (T)	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	<u>0.022</u>	<u>0.003</u>	<u>0.035</u>	<u>0.008</u>
	Fibre Amount (A)	<u>0.009</u>	<u>0.000</u>	<u>0.000</u>	0.530	0.172	0.126	0.100
	Fibre Length (L)	0.279	<u>0.000</u>	<u>0.000</u>	0.101	<u>0.032</u>	<u>0.002</u>	0.267
	Fibre Condition (C)	0.571	<u>0.005</u>	<u>0.000</u>	0.083	0.173	0.586	0.262

Interactions	T*A	0.000	0.000	0.762	0.000	0.000	0.188	0.000
	T*L	0.023	0.000	0.000	0.349	0.021	0.047	0.002
	T*C	0.000	0.000	0.000	0.846	0.834	0.009	0.884
	A*L	0.000	0.000	0.000	0.213	0.278	0.604	0.866
	A*C	0.000	0.000	0.000	0.866	0.787	0.868	0.000
	L*C	0.000	0.000	0.000	0.088	0.455	0.030	0.001
	T*A*L	0.000	0.000	0.000	0.526	0.142	0.016	0.335
	T*A*C	0.004	0.185	0.000	0.000	0.033	0.084	0.000
	T*L*C	0.000	0.001	0.000	0.161	0.113	0.209	0.000
	A*L*C	0.000	0.000	0.000	0.521	0.790	0.128	0.521
	T*A*L*C	0.000	0.000	0.000	0.001	0.020	0.072	0.695
R^2 (%)		99.03	99.89	99.89	82.48	81.17	79.52	93.12
Anderson Darling		0.879	0.643	0.881	0.890	0.755	0.091	0.721

3.1 Physical Properties

Apparent density data range from 2.25 to 2.37 g/cm³. Benmansour *et al.* investigated the use of palm fibres in different lengths (3 and 6 mm) and amounts (5, 10 and 15wt%) in mortars, obtaining values of less than 0.984 to 1.476 g/cm³[1]. Aggarwal used sugarcane bagasse fibres in mortar involving two amounts of fibre (12 and 16wt%), obtaining a density variation between 1.55 and 1.65 g/cm³[3].

Table 3 shows that the type of fibre (treated and untreated) and the amount of fibre (0.6 and 1.2 mm) individually affect apparent density. The main effect plots for mean apparent density are presented in Figure 1. The use of natural bagasse fibres (SCB) increases composite apparent density when compared to treated fibres (MSCB – see Figure 1a). This behaviour can be attributed to the substantial 208% difference between SCB (~0.71 g/cm³) and MSCB (~0.23 g/cm³) fibre densities. Increases in the amount of fibre by 1 to 2 wt% lead to reduced composite density (Figure 1b), since the fibre density is lower than that of aggregate.

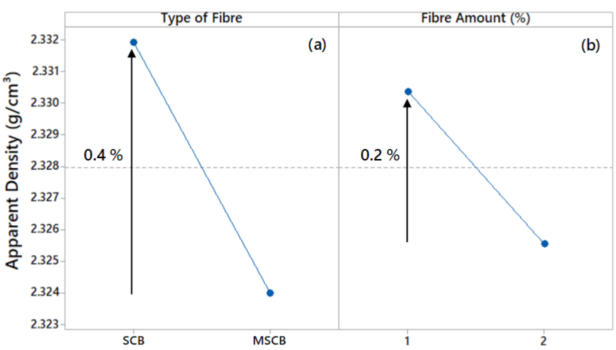

Figure 1. Main Effect Plots for Mean Apparent Density

Water absorption values range from 2.41 to 8.01%. Aggarwal studied two amounts of sugarcane bagasse particles (12 and 16wt%) as reinforcement in cementitious composites, exhibiting water absorption values between 12.5 and 14.5%[3]. Filho investigated sisal fibre reinforced composites in two lengths (25 and 50 mm) and three amount levels (2, 4, and 6wt%), reaching water absorption values from 7.73 to 11.47%[17]. Figure 2 shows the individual effects of factors on the mean water absorption of the composites. The main contributing factor is the fibre type with a percent variation between levels of 34%. These findings are discussed along with the porosity behaviour shown below.

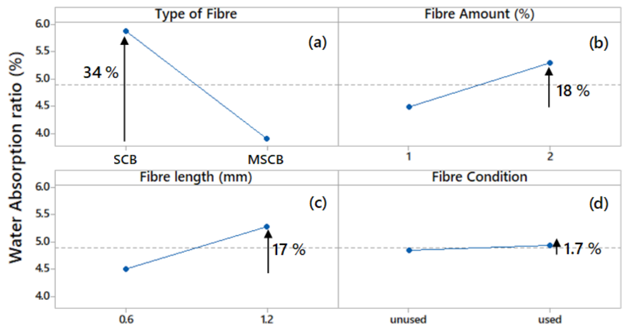

Figure 2. Main Effect Plots for Mean Water Absorption Ratio

Apparent porosity data range from 5.21 to 15.95%. Findings reported by Filho ranged from 14.87 and 21.48%[17]. Teixeira investigated *Curauá* fibre reinforced composites of different lengths (6 and 10 mm) and amounts (1 and 2%), with resulting porosity between 28.16 and 31.90%[18]. The results found in this study are significantly lower than those reported in the open literature. The reference condition (without fibres) also presented a higher level of apparent porosity (14.73%, see Table 1) than most of the tested conditions, similar to the water absorption results. Table 3 shows that all individual factors significantly affected apparent porosity (Figure 3). The factor that most contributes to porosity is the type of fibre that shows a percentage variation of 29% (Figure 3a).

In general, there are two effects associated with pore formation in the system. The first is related to the amount of water/cement (w/c) ratio, which affects the microstructure of the cementitious phase. The higher the w/c ratio, the greater the porosity. The amount of water in the system can be reduced due to the presence of natural fibres capable of absorbing it. The second effect is due to the presence of pores in the interfacial transition zone (ITZ) of the aggregates and fibres. Treated fibres (MSBC) tend to absorb more water from the system, leading to a dense cementitious phase, thereby decreasing water absorption and porosity of the composites (Figure 2a and 3a). A larger amount of fibres also provides an increase in the amount of ITZ, which is responsible for increasing water absorption and porosity levels, as shown in Figure 2b and 3b, respectively. In addition, longer fibres (1.2 mm) ex-

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hibit a surface area greater than 0.6 mm, which increases fibre concentration per unit volume and ITZ, increasing water absorption ratio and porosity by 17% and 13%, respectively (Figures 2c and 3c). A small contribution of the fibre condition factor (unused and used) is evidenced in Figures 2d and 3d. This behaviour also emphasises that the composite microstructure is mainly affected by the matrix phase characteristics.

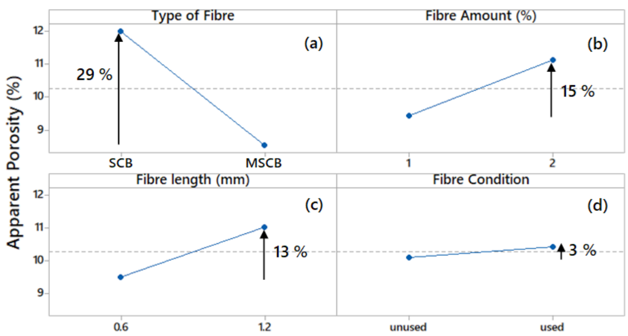

Figure 3. Main Effect Plots for Mean Apparent Porosity

3.2 Ultra-pulse Velocity and Dynamic Modulus

Pulse velocity (UPV) data for composite samples ranged from 352.97 to 393.17 m/sec. Valenciano *et al.* investigated cementitious composites with 2 wt% treated sugarcane bagasse fibres at different lengths, reaching UPV values from 1290 to 1880 m/s[19]. These results are substantially higher than those reported in the present work, being mainly attributed to the effects of w/c ratio on the cement microstructure.

The UPV values for the reference condition - without fibres (370.76 m/s) are less than 2 wt% SCB composites, confirming the effect of fibre water absorption on cement hydration, as previously discussed in porosity section. Higher UPV values indicate a denser solid.

The only main factor affecting the UPV response is fibre type (see Table 3). The ultra-pulse velocity of the composites increases when treated fibres (MSCB) are incorporated (Figure 4). Although a slight variation (1.5%) is obtained between the levels of SBC and MCBC; shows that the UPV technique is able to detect the effect of fibre composition on the composite microstructure. MSCB composites exhibit lower water absorption (Figure 2a) and porosity (Figure 3a), being attributed to the reduction in fibre-absorbed w/c ratio, which leads to denser material and higher ultra-pulse velocities (Figure 4).

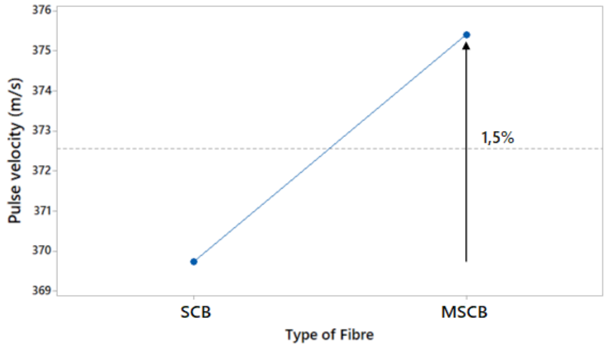

Figure 4. Main Effect Plot for Mean Ultra-pulse Velocity

Dynamic modulus data range from 0.241 to 0.310 GPa. Valenciano *et al.* reported the dynamic modulus of bagasse-reinforced cementitious composites ranging from 2.37 to 6.03 GPa[19]. In the present work, the dynamic modulus of the reference condition (without fibres) is 0.28 GPa (Table 1). Table 3 shows that two main factors, fibre type and fibre length, significantly affect the dynamic modulus (Figure 5). As previously discussed, MSCB fibres tend to absorb more water from the system, reducing porosity and increasing ultra-pulse velocity. As a result, an increased dynamic modulus is achieved when composites are made with MSBC fibres (Figure 5a). Fibre length also affects the volume occupied by the matrix phase in the system. Shorter fibres lead to larger amounts of cement volume, providing increased dynamic modulus (Figure 5b).

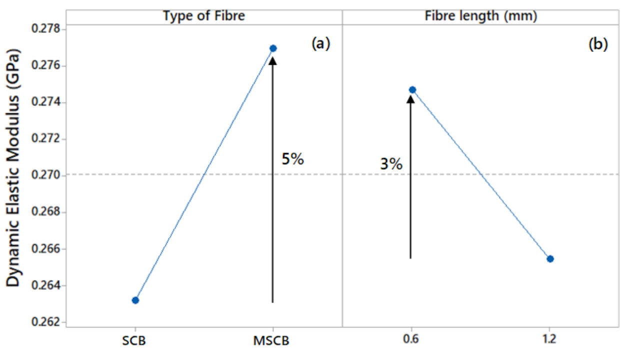

Figure 5. Main Effect Plots for Mean Dynamic Elastic Modulus

3.3 Mechanical Properties

The flexural strength of the cementitious composites ranges from 4.56 to 7.78 MPa. These results are in agreement with [20], which obtained 7.50 and 4.43 MPa for composites reinforced with 8 and 12 wt% of bamboo pulp, respectively[20]. In this work, fibre type and fibre length statistically affect the flexural strength (Figure 6), but the

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fibre length factor leads to a large contribution (11%). Composites made with MSCB fibres obtained not only reduced porosity (Figure 3a), but also increased flexural strength (7%) when compared to SCB composites (Figure 6a). MSCB fibres (9.3 m^2g^{-1}) exhibit a smaller surface area than SCB (9.6 m^2g^{-1})[11], contributing to a higher amount of cement paste, which increases flexural strength. In addition, functionalization with APTS can remove amorphous phase by increasing the amount of hydroxyl groups which can provide increased chemical bonding. Improvements in mechanical performance of APTS-treated natural fibre-reinforced cementitious composites have been attributed to increased adhesion to the fibre-matrix interface due to the formation of cement hydration products in the luminal cavity, known as cellulose fibre mineralization[21]. Due to their smaller surface area, shorter sugarcane fibres (0.6 mm) also contribute to increase the volume occupied by the matrix phase in the system, which is responsible for greater composite strength (Figure 6b).

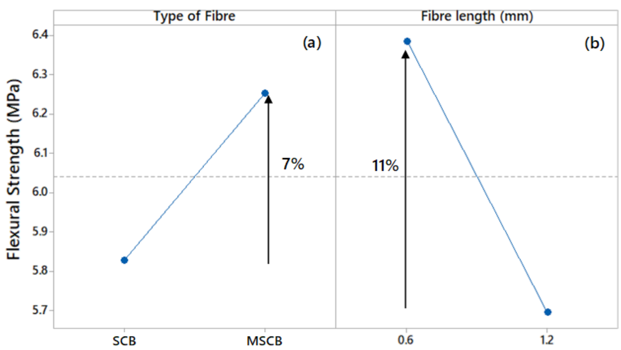

Figure 6. Main Effect Plot for Mean Flexural Strength

The flexural moduli range from 0.30 to 0.68 GPa. Picanço and Ghavami investigated *Curauá,* jute and sisal fibre reinforced composites with inclusions of 2 and 3 wt% and lengths of 15, 25, and 45 mm, obtaining flexural moduli from 18.24 to 29.33 GPa[22]. These findings are extremely superior to those obtained in this study. It is noteworthy that many other factors, such as densification, curing conditions, fibre type, length and amount can also influence the mechanical properties of the composite. The reference condition (without fibres) reached 0.42 GPa for the mean elastic modulus (Table 1). Table 3 reveals that the type of fibre factor significantly affects the flexural modulus (Figure 7). The flexural modulus of pristine fibre reinforced composites (SCB) is 8% larger than MSCB composites, being attributed to better fibre-matrix adhesion. Sugarcane fibres in pristine condition exhibit a rougher surface than the treated ones (see Figure 8), which contributes to improving the elastic modulus of the composites. It is worth of notice that chemical treatment with APTS is carried out to increase the fibre absorption characteristic, as reported by Guilharduci *et al.* [11] and not as an improvement in fibre-matrix interface. The use of APTS has led to a higher oil sorption capacity, being attributed to the polar amino terminal groups in the silane structure, which increases the hydrophilic character of the fibres [11].

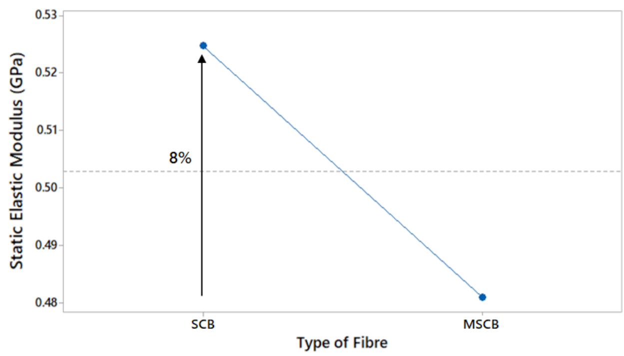

Figure 7. Main Effect Plot for Mean Flexural Modulus

It is noteworthy that the dynamic modulus (Figure 5a) achieves higher values for composites made with MSBC fibres, whereas the static modulus (Figure 7) is 8% higher when using SCB fibres. The velocity of ultrasonic pulses (UPV) traveling in a solid depends on the density and elastic properties of the material. A decrease in UPV corresponds to an increase in porosity and permeability, which confirms a region of low compaction, voids or damaged material [26]. In this context, it is possible to state that the condition of interfacial adhesion between fibres and cement paste does not affect UPV measurements, consequently revealing divergent elastic behaviour in static and dynamic modes.

3.4 Microstructural Analysis

A Hitachi TM 3000 microscope is used to observe the morphology of sugarcane bagasse fibres in pristine condition (Figure 8a,b) and after treatment with APTS (Figure 8c,d). Images are taken in backscattered mode, with an acceleration voltage of 5kV at magnification levels of ×100 and ×500. SCB fibres reveal a rougher surface compared to MSBC fibres. APTS functionalization is able to remove impurities, wax and amorphous phase, such as lignin and hemicellulose, from the fibre surface which makes it smoother [21, 23, 25]. The higher surface roughness revealed by SBC fibres may also be responsible for increasing the elastic modulus when compared to MSBC composites, as shown in Figure 7.

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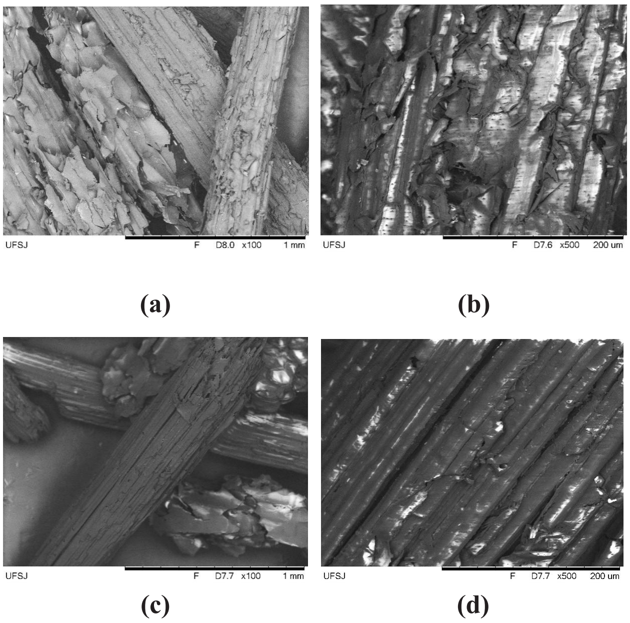

Figure 8. Backscattered Electron Images of Sugarcane Fibres (a, b) in Pristine Conditions and (c, d) After Treatment

4. Conclusion

The main conclusions are described as follows:

i.The amount of sugarcane fibres (1 and 2wt%) does not affect the mechanical properties of the composites. However, higher fibre amounts lead to increased porosity and water absorption, which may affect the durability of cementitious products.

ii.Fibre type significantly affects mechanical responses, achieving higher strength for treated fibres (MSBC) and higher stiffness for untreated ones (SBC).

iii.The fibre length factor affects the physical and mechanical properties. Reduced porosity and water absorption and increased flexural strength and stiffness are obtained especially when 0.6 mm fibres are used.

iv.The presence of oil in the fibres does not affect the mechanical properties of composites. This indicates that the reuse of absorbing fibres in cementitious products is feasible.

v.The dynamic and elastic moduli reveal different findings, being attributed to the inefficiency of the UPV to measure the interfacial adhesion condition between the fibres and the cement paste.

vi.Functionalization using APTS is able to remove amorphous phase, obtaining a smoother fibre surface compared to pristine sugarcane bagasse.

Cementitious composites made with 2 wt% of 0.6 mm MSCB fibres, pre-used in effluent treatments, are considered promising in non-structural applications for the construction industry.

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