INCORPORATION OF REUSED SUGARCANE FIBRES APPLIED IN THE TREATMENT OF EFFLUENTS CONTAMINATED WITH ENGINE OIL AS REINFORCEMENT OF CEMENTITIOUS COMPOSITES

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Abstract

This work investigates the reutilisation of natural (SCB) and modified sugarcane bagasse fibres with aminopropyltriethoxysilane (MSCB) in cementitious composites. The modified fibres were used in the treatment of effluents contaminated with oil-engine. A full factorial design was used to identify the effects of fibre type (natural and modified sugarcane), fibre length (0.6 and 1.2 mm), fibre amount (1 and 2wt%), and fibre condition (before and after oil filtration) on apparent density, water absorption, apparent porosity, ultra-pulse velocity, dynamic modulus, flexural strength and modulus. SCB fibres led to increased apparent density compared to MSCB fibre reinforced composites. The MSCB fibres contributed to reduce the porosity of the composite, leading to higher mechanical properties. The reduced area of MSCB fibres increased the amount of cementitious phase per unit volume which increases the sample strength. Longer fibres (1.2 mm) have a higher surface area, leading to a higher concentration of fibres per unit volume, increasing water absorption by 17%. The amounts of fibres had no significant effect on the mechanical and physical responses. Samples made with 2wt% MSCB fibres of 0.6 mm in length achieved promising results for non-structural applications in civil engineering.

1. INTRODUCTION

The construction industry requires significant amounts of raw materials and energy for its development, generating a significant amount of pollution (CO2 emissions are an urgent risk) and waste. The search for sustainable solutions includes the correct use of industrial and agroindustry materials. New approaches to efficient projects are related to the development and use of natural building materials [6].

Gatto *et al.* [1] reported several advantages in composites with natural fibres, such as reduced apparent specific gravity, higher porosity, satisfactory tensile and impact strength, greater fracture control, and ductile failure. Further benefits are biodegradability of fibres, reduced costs and damages to manufacturing machinery when compared to synthetic fibres [2]. On the other hand, cement-based matrices are fragile and may present small cracks, even under small tensile loads or under deformation. The incorporation of fibres can 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 and impact strength [3-4]. Cementitious composites with reinforcement of natural fibres have been developed in several applications, such as in the automotive and building industries, as a substitute for conventional materials, with glass fibre being an example of fibre replacement [5].

A significant amount of natural fibres (hemp, straw, linen, bamboo, cork, etc.) has been applied as reinforcing phase to conventional matrices (cement, mortar, sand, plaster, concrete, etc.) for the design of composite materials. Many factors can affect the properties of fibre-reinforced composites, including fibre geometry, distribution, orientation, shape and length, as well as, the properties of individual phases (matrix and fibres), contact type, phase mixing ratio and manufacturing, etc. [7]. Most studies found in the literature investigated only a few significant parameters, such as fibre amount, length and type, to estimate composite performance [8].

Metha and Monteiro [9] reported an inverse relationship between porosity (volumetric fraction of pores) and mechanical strength in solids. In a multi-phase material, the porosity of each component or phase within its structure is a limiting factor for composite resistance. Although the water/cement ratio is the most important factor in determining matrix and transition zone porosities, in addition to the mechanical strength of cementitious composites, other factors such as densification, curing conditions (hydration degree of cement), aggregate dimensions and mineralogy, additives, sample geometry and moisture, and load type may have a significant effect on the mechanical properties.

Reis and Carneiro [5] highlighted the advantages of natural fibres, which include low cost, lightweight, high availability, easy recycling, good thermal and acoustic insulation, moderate mechanical properties and reduced CO_2 emissions. On average, the production of natural fibres as composite reinforcements require 60% less energy than the production of glass fibre. Besides reinforcing cementitious composites, natural fibres can also be used as absorbing material. Scarcity of water resources poses a challenge to the sustainable development of industrial and agricultural activities. The production of significant amounts of industrial effluents and the awareness of the impact of their effects on nature have forced the industry to adopt new environmental policies [10-11]. One method widely applied in last decades for the treatment of water contaminated with oil is the use of absorbent materials, which easily remove and recover the oil. Many materials can be used at this stage, being natural organic products of living organisms, also known as biosorbents, advantageous because of the high availability and low cost. Some examples are sawdust, cotton, peat, sugarcane bagasse, straw and corn cob. They are highly capable to float and are biodegradable.

Annunciado *et al.* [12] reported that natural fibres are example of low cost absorbents from renewable sources that may represent a sustainable alternative in reducing oil contamination. Sawdust, cellulose, rice husk, sugarcane bagasse, corn cob and straw, cotton, etc. are abundantly produced in Brazil. However, most of these fibres are discarded due to lack of suitable applications. They represent a suitable alternative for oil adsorption, due to its high absorption, low cost, no need for regeneration, and the contribution of the cellulosic nature to the degradation the oil in aqueous systems [13].

Although natural fibres can be an alternative for oil absorption, they have limitations because these fibres 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 properties of lignocellulosic materials. However, the hygroscopic properties of the samples can be reduced by superficial modifications [14]. One possible approach is to replace the hydroxy functional group with hydrophobic groups through chemical reactions.

This work investigates the use of sugarcane bagasse fibres in pristine (SCB) and modified fibre with aminopropyltriethoxysilane (MSCB) applied in the treatment of effluents contaminated with engine oil as reinforcement of cementitious composites. The use of natural fibres for effluents treatment has been described by Gatto *et al.* [1] and Guilharduci *et al.* [16].

2. MATERIALS AND METHODS

2.1. Materials

The cement used was the Portland cement (ASTM III) supplied by Cauê Company (Brazil). The sand particles were supplied by *Omega Mining Company (*Brazil). The sugarcane bagasse (SCB) fibres were sourced by *Cachaça Coqueiro* Stiller (Brazil). Aminopropyltriethoxysilane (APTS, 98%, $(H_2NCH_2CH_2CH_2)$-Si-$(OCH_2CH_3)_3$) was purchased from Dow Corning and was used without further purification. The other analytical grade reagents were obtained from Merck and were used as received.

2.2. Methods

The factors analysed were the type of fibre in pristine (SBC) and modified condition (MSCB), fibre length (0.6 and 1.2 mm), fibre amount (1 and 2%wt as a replacement of aggregates), and treatment conditions (before and after oil absorption), resulting in a Full Factorial Design 2^4 type, as shown in Table 1. The fibres with absorbed oil were prepared with the effluent collected at *Del-Rei Rectifier Company (*Brazil). One gram of fibres was immersed in 100 ml of effluent under stirring during 24h. Subsequently, the fibres were filtered and dried at room temperature [16]. Table 1 presents the factors investigated and their respective levels.

Table 1: Factors and experimental levels (2^4 = 16 conditions)

Factors	***Levels***	
Type	SCB	MSCB
Length	0.6 mm	1.2 mm
Amount	1%wt	2%wt
Condition	Before oil absorption	After oil absorption

The Design of Experiment (DoE) and Analysis of Variance (ANOVA) were performed via Minitab 18. The samples were prepared according to the standard NBR 7215 [17]. They were manufactured with mortar matrix composed of one part of cement and three parts of standard sand (20% of fine aggregate, 50% of medium aggregates and 30% of coarse aggregates) and water/cement ratio of 0.55. The mixture was inserted into prismatic moulds of 40 mm square cross-section area and 160 mm length. An electromagnetic vibrator was applied for 8 minutes to ensure a uniform batch. The samples were identified and covered by plastic film to prevent loss of moisture. As recommended by NBR 5738 [18], the samples were demoulded after 7 days and kept at room temperature. Samples were tested after 28-days cure under three-point bending load according to ASTM C348-14 [20] at a cross-head speed of 0.5 mm/min to obtain flexural strength (σ_f). The flexural test was conducted in a universal testing machine Shimadzu AGX with 100 kN load using the integrated software Trapezium X. The static modulus of elasticity (E_s) was then calculated based on ASTM C580-02 [20]. The pulse velocity (ν) was obtained using a portable ultrasonic non-destructive digital indicating test (PUNDIT) equipment in accordance with ASTM C597-09 [21]. The dynamic modulus of elasticity (E_d) was obtained based on ASTM C348-14 [19] standard. Physical properties, such as water absorption (W_{abs}) apparent porosity (P_a), and apparent density (ρ_a) were also determined using British standard BS 20545-3 [22].

3. RESULTS AND DISCUSSIONS

The statistical test investigated whether the factors analysed, such as type of fibre (SCB and MSCB), amount of fibre (1 and 2%wt), fibre length (0.6 and 1.2 mm), and fibre condition (with and without absorbed oil) could affect the results of evaluated responses. A statistical analysis was performed using the P-Values of ANOVA to verify if there is any interference of the factors analysed. The P-Values underlined in Table 2 indicate the significant factors within a 95% confidence level. The reported values of R^2 also indicate that the quality of model adjustment was satisfactory for all variables. The assumption of data normality is an additional requirement to validate the use of hypothesis tests and analysis of variance. The normality of the experimental data was verified using the Anderson-Darling test. This test indicate that the data follows the normal distribution if the P-Value is greater than 0.05. In this case, all data follow a normal distribution (see Table 2), validating ANOVA model.

Table 2: Analysis of Variance results – P-Values

	Factors	ρ_a	W_{abs}	P_a	ν	E_d	σ_f	E_s
Main	**Type of Fibre (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>
	Amount of Fibre (A)	<u>0.009</u>	<u>0.000</u>	<u>0.000</u>	0.530	0.172	0.126	0.100
	Length of Fibre (L)	0.279	<u>0.000</u>	<u>0.000</u>	0.101	<u>0.032</u>	<u>0.002</u>	0.267
	Condition of Fibre (C)	0.571	<u>0.005</u>	<u>0.000</u>	0.083	0.173	0.586	0.262
Interactions	**T*A**	<u>0.000</u>	<u>0.000</u>	0.762	<u>0.000</u>	<u>0.000</u>	0.188	<u>0.000</u>
	T*L	<u>0.023</u>	<u>0.000</u>	<u>0.000</u>	0.349	<u>0.021</u>	<u>0.047</u>	<u>0.002</u>
	T*C	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	0.846	0.834	<u>0.009</u>	0.884
	A*L	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	0.213	0.278	0.604	0.866
	A*C	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	0.866	0.787	0.868	<u>0.000</u>
	L*C	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	0.088	0.455	<u>0.030</u>	<u>0.001</u>
	T*A*L	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	0.526	0.142	<u>0.016</u>	0.335
	T*A*C	<u>0.004</u>	0.185	<u>0.000</u>	<u>0.000</u>	<u>0.033</u>	0.084	<u>0.000</u>
	T*L*C	<u>0.000</u>	<u>0.001</u>	<u>0.000</u>	0.161	0.113	0.209	<u>0.000</u>
	A*L*C	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	0.521	0.790	0.128	0.521
	T*A*L*C	<u>0.000</u>	<u>0.000</u>	<u>0.000</u>	<u>0.001</u>	<u>0.020</u>	0.072	0.695
R^2 (%)		99.03	99.89	99.89	82.48	81.17	79.52	93.12
Anderson Darling (P-value>0.05)		0.879	0.643	0.881	0.890	0.755	0.091	0.721

3.1 Physical Properties

The apparent density data ranged from 2.25 to 2.37 g/cm³. Benmansour *et al.* [6] investigated the use of palm fibres in different lengths (3 and 6 mm) and amounts (5, 10 and 15wt%) in mortar, obtaining values of density lower than 0.984 to 1.476 g/cm³. Aggarwal [24] used sugarcane bagasse fibres in mortar involving two fibre amounts (12 and 16wt%), obtaining a density variation between 1.55 and 1.65 g/cm³.

Table 2 reports that the type of fibre (treated and untreated) and fibre amount (0.6 and 1.2 mm) factors individually affect apparent density. They also influence the remaining factors, as noted in the interactions shown in Table 2. The individual plots are presented in Figure 1 to assess the effect of each parameter on the apparent density. The use of natural bagasse fibres increased composite apparent density when compared to treated fibre reinforced composites (MSCB – see Figure 1a). The substantial difference of 208% between the fibre densities (0.71 g/cm³ for SCB and 0.23 g/cm³ for MSCB) justifies this behaviour. Figure 1b reveals the increase in the amount of fibre from 1 to 2wt% led to reduced composite density, since the fibre density is smaller than the aggregate density.

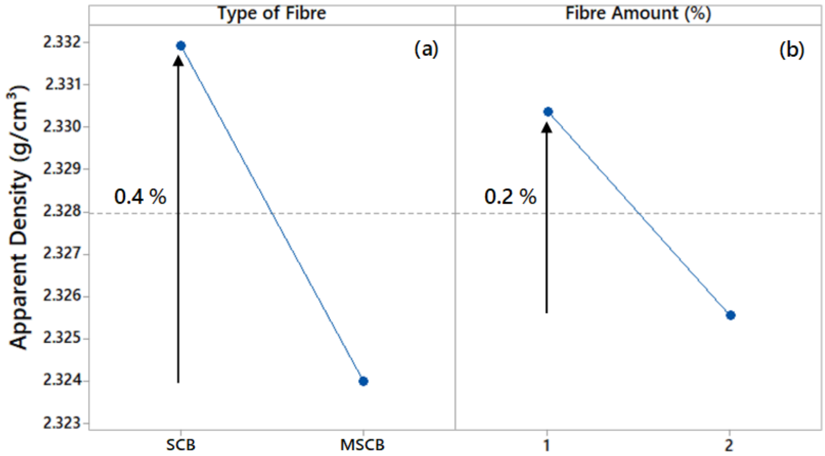

Figure 1: Main effect plots for mean apparent density.

The water absorption values ranged from 2.41 to 8.01%. Aggarwal [24] studied two amounts of sugarcane bagasse particles (12 and 16wt%) as reinforcement in cement composites, presenting a water absorption ratio between 12.5 and 14.5%. Filho [25] investigated sisal fibres reinforced composites at two lengths (25 and 50 mm) and three amount levels (2, 4, and 6%), reaching water absorption values of 7.73 to 11.47%. These findings indicate that the range of absorption found in this study is promising. The benefits of fibre inclusions are evident when the water absorption ratio for the reference condition (without fibres) is evaluated. A large absorption of 7.38% was obtained for the reference condition, being attributed to the excess water in the system. It is noteworthy that water can be absorbed when fibres are incorporated into the system. Table 2 indicates that four main factors were considered significant for the water absorption response. The influence of individual factors is shown in Figure 2.

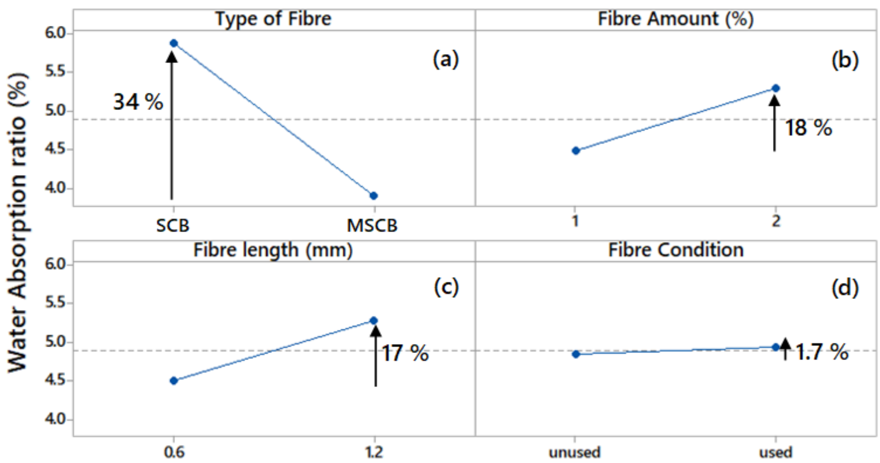

Figure 2: Main effect plots for mean water absorption ratio.

The apparent porosity varied from 5.21 to 15.95%. The findings reported by Filho [25] ranged from 14.87 and 21.48%. Teixeira [26] investigated composites reinforced with Curauá fibres of varying lengths (6 and 10 mm) and amounts (1 and 2%), with a resulting porosity between 28.16 and 31.90%. The results found in this work were significantly lower than those reported in the literature. The reference condition also presented higher apparent porosity (14.74%) than most of the conditions tested, similar to the results of water absorption. It is observed in Table 2 that all individual factors significantly affected the apparent porosity (Figure 3).

The treated fibres (MSBC) tended to absorb more water, which led to reduced pores within the cementitious sample and reduced water absorption and porosity levels (Figure 2a and 3a). In addition, MSBC fibres present lower porosity than untreated fibres (SCB). Fibre porosity directly affects the composite water absorption and porosity after cement-hydration process, increasing these properties when a larger amount of fibre is used (see Figure 2b and 3b). Longer fibres (1.2 mm in length) exhibit higher superficial area compared to fibres of 0.6 mm in length, which increases fibre concentration per unit volume. Therefore, longer fibres increase the water absorption ratio and sample porosity by 17% and 13%, respectively (see Figures 2c and 3c), due to the higher volume of fibres within the sample. Figures 2d and 3d show a reduced variation for the fibre condition (unused and used fibres), however this effect is important to identify the dominance of the cementitious matrix over resulting properties. MSBC fibres were able to absorb less water in the system, leading to excess water in the sample, consequently increasing the porosity and water absorption of the cured samples.

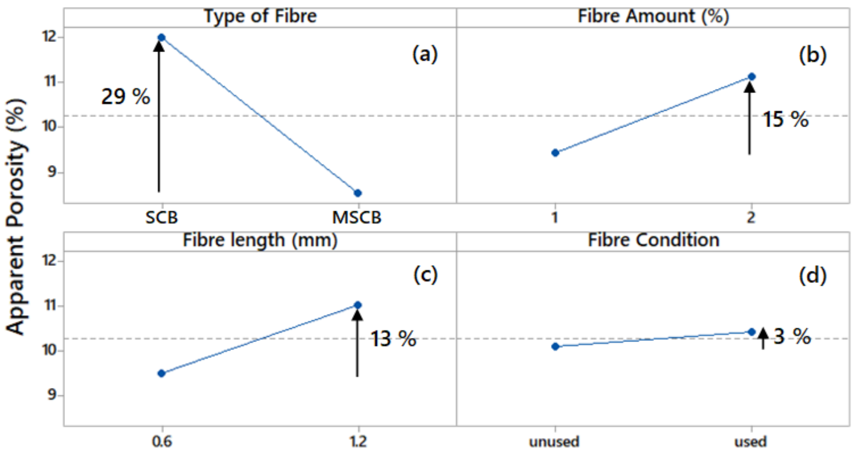

Figure 3: Main effect plots for mean apparent porosity.

3.2 Ultra-pulse velocity and Dynamic modulus

The pulse velocity data for composite samples varied from 352.97 to 393.17 m/sec. Valenciano [4] investigated cementitious composites with 2 wt% sugarcane bagasse fibres in varied lengths. The fibres were used untreated and with chemical treatment by immersion in 5% sodium silicate solution and in 30% aluminium sulphate. These samples presented pulse velocities between 1290 and 1880 m/s. The results found in the present work were significantly lower than the literature values, which can be attributed to different fibre lengths, amounts and chemical treatments, as well as the components ratio (cement:aggregates:fibre:water). The overall results, however, are reasonable when considering the result for reference condition without fibre inclusions (371.41 m/s). Table 2 indicates that only the main factor “Type of Fibre” significantly affects the response (see Figure 4). The ultra-pulse velocity of the composites increased when treated fibres (MSCB) were incorporated. This behaviour can be explained by the effect of the fibres on the matrix properties. The MSCB fibres exhibit higher water absorption, which consumes the water/cement ratio of the system, leading to a less porous cementitious sample, thus achieving a higher ultra-pulse velocity.

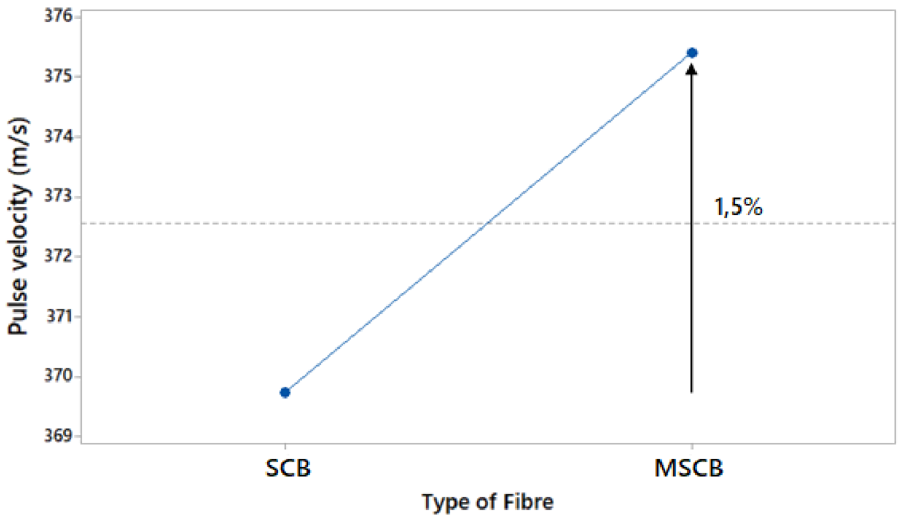

Figure 4: Main effect plot for mean ultra-pulse velocity.

The values of the dynamic elastic modulus ranged from 0.241 to 0.310 GPa. Valenciano [4] also reported the dynamic modulus of bagasse-reinforced cementitious composites, which ranged from 2.37 to 6.03 GPa. These values are again above the results obtained in the present work. It is noteworthy that the dynamic modulus of the reference condition was 0.27 GPa, which indicates an appropriate variation of the results. Table 2 shows that the factors “Type of fibre” and “Fibre length” significantly affect the dynamic modulus (Figure 5). As previously discussed, MSCB fibres tend to absorb more water in the sample, reducing the porosity of the cementitious specimens, which increases the ultra-pulse velocity. As a result, an increased dynamic modulus was reached when the composites were reinforced with MSBC fibres (see Figure 5a). The fibre length also affects the volume occupied by the matrix phase in the system. Shorter fibres led to larger amounts of matrix, leading to the increase of the dynamic modulus (Figure 5b).

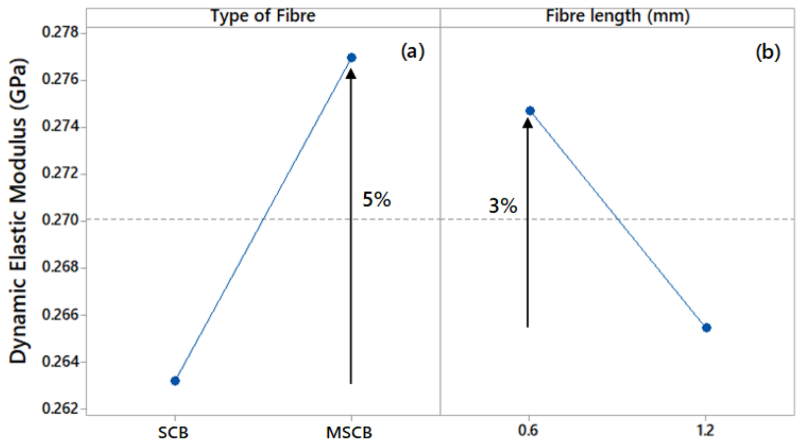

Figure 5: Main effect plots for mean dynamic elastic modulus.

3.3 Mechanical Properties

The flexural strength of cementitious composites varied from 4.56 to 7.78 MPa. These results are in agreement with the values reported by Sales [27], considering that the author has investigated reinforced composites with 8 and 12 wt% of bamboo pulp. The results of the flexural strength were reported as, respectively, 7.50 and 4.43 MPa. The type, amount, length and conditions of fibres have been reported as the main factors influencing flexural strength of reinforced cementitious samples. In this work, the fibre type and length were statistically significant (Figure 6), the fibre length being the factor that most influenced the flexural strength. The composites made with MSCB fibres provided reduced porosity (see Figure 3a) and thus achieving higher strength when compared to SCB composites (see Figure 6a). MSCB fibres have a lower superficial area than natural ones, which increases the amount of cementitious paste in the sample, leading to superior flexural strength. Shorter fibres (0.6 mm) also present lower superficial area, reaching higher composite strength (Figure 6b).

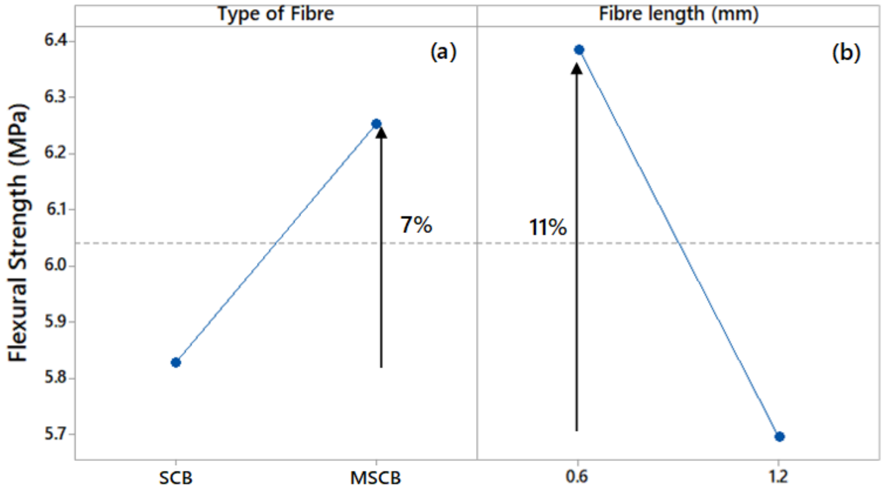

Figure 6: Main effect plot for mean flexural strength.

The flexural modulus ranged from 0.303 to 0.678 GPa. Picanço and Ghavami [28] investigated composites reinforced with *curauá*, jute and sisal fibres at 2 and 3wt% inclusions and lengths at 15, 25, and 45 mm. The authors found that the flexural modulus varied from 18.24 to 29.33 GPa. These findings are highly superior to those obtained in this work. 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 results obtained in this research show satisfactory agreement with the reference condition (0.50 GPa). Table 2 reveals that the type of fibre factor significantly affects the flexural modulus (Figure 7). Figure 7 shows that the natural fibre reinforced composites (SCB) achieved 8% higher flexural modulus than MSCB composites, being attributed to the fibre-matrix adhesion condition. The natural fibres exhibit a rougher surface than the treated ones (MSCB), which contributes to enhance the modulus of elasticity of the composites.

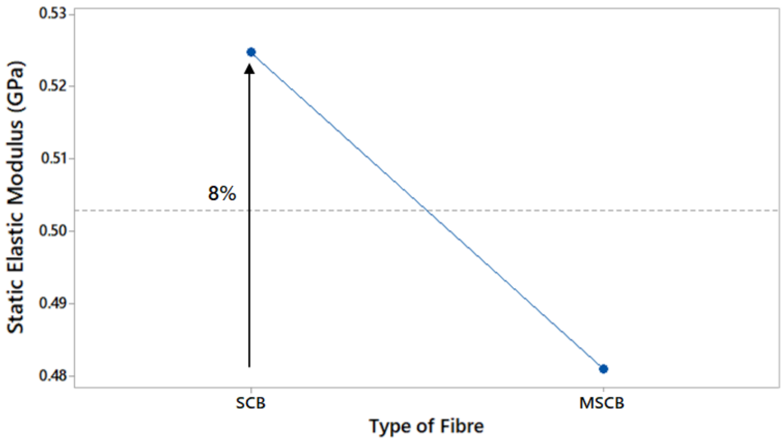

Figure 7: Main effect plot for mean flexural modulus.

4. CONCLUSIONS

This study investigated the reuse of natural fibres, after treatment with effluents contaminated with oil, as reinforcement of cementitious composites. A statistical analysis was conducted to evaluate the influence of the fibre length, amount, treatment and condition on the mechanical and physical responses. The main conclusions are as follows:

- The presence of oil in the fibres was not significant to affect the mechanical properties of the composites. It indicates that the reuse of absorbing fibres into cementitious products is feasible;
- The amount of fibre (1 and 2wt%) did not affect the mechanical properties of the samples. However, higher amounts of fibres led to increased porosity and water absorption, which may reduce the durability of cementitious products in outdoor environments. Since natural fibres are only suitable for indoor applications, the 2wt% fibre amount level is adequate in a context of increasing need for natural fibres in the construction industry;
- The type of fibre (natural and treated fibres) had a significant influence on the responses, with higher strength for MSCB fibres and higher stiffness for natural fibres. Due to the high functionality of treated-fibres (MSCB) in the effluent treatment, it can be concluded that both types can be used in non-structural engineering applications.;
- Fibre length (0.6 and 1.2 mm) factor affected the physical and mechanical properties by reducing sample permeability and increasing the flexural strength of composites with 0.6 mm fibres by approximately 11%.

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

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