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Composites of Portland cement paste and sugarcane bagasse fibers: structure-property relation and Weibull statistics

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Abstract

The main objective in this investigation is to study the structure-property relation of fiber-reinforced composites fabricated with Portland cement paste and sugarcane bagasse fibers, which is a residual biomass resulting from the cleaning, preparation, and extraction of sugarcane juice. Pastes of 1.0 and 2.0wt% of fibers were manufactured with water to cement ratio (W/C) of 0.6. The mechanical properties were determined through three-point flexion tests and the microstructural characterization was carried out through Scanning Electron Microscopy (SEM) tests. A Weibull distribution analysis for flexion damage was performed. Density, compressive strength, and ductility graphs were included. An analysis of the influence of capillaries pore size of the fiber over the flexural strength was made. Results show that the fiber content improves the compressive strength, 9.7MPa, 8.8MPa and 11.5MPa for cement paste with 0, 1 and 2 wt% fibers contents, respectively. These composites can be used as alternative green materials for houses in areas were sugarcane bagasse has poor or none waste valorization.

1. Introduction

Cementitious matrix composite materials reinforced with natural fibers have become promising materials for acceptable mechanical performance as a construction material in multiple applications, using their ability to dissipate energy through their failure mechanisms [1-4]. The rational use of vegetable fibers can be an alternative solution for the production of durable, more sustainable goods and green construction materials [3-5]. Natural fibers reinforcements are classified into three types depending on the source: animal, mineral, and vegetable origin [6]. In comparison with the synthetic fibers that are commonly used, vegetable fibers are recyclable, low cost, low density and biodegradable, although their use is limited due to their relatively low mechanical properties compared to synthetic fibers [7]. However, because of their low cost and density, natural fibers can be used in greater volume and therefore obtain composite materials with mechanical properties comparable to those reinforced with synthetic fibers [7].

Different researchers have analyzed natural fiber as reinforcement of cement matrix in diverse properties including tensile strength, fatigue, impact, ductility, and hardness have been reported [8], with very promising results. Portland cement paste with bamboo, luffa, and other fibers are currently used in non-high strength structural applications [9-14]. On the other hand, significant losses in long-term mechanical performance have been observed in plant fiber-reinforced composites after natural or accelerated aging, due to the degradation of cellulose fibers in the cementitious environment. [10-13]. The strength and rigidity of natural fibers depends on the cellulose content and the orientation of the microfibers in the cell wall [14]. These properties are also influenced by the particular fiber extraction methods and the environmental conditions of growing and storage [6].

Some studies established that natural fibers of plant origin have a negative effect on the hydration of cement compounds [15]. It has been observed a delayed setting time and a reduction of the heat of hydration in cement compounds reinforced with sugarcane bagasse fibers [15], which was attributed to the water-soluble sugars formed as a result of the alkaline hydrolysis of the lignin and the partial solubilisation of the hemicellulose contained in these fibers. Other studies support this argument for the case of Portland cement reinforced with bamboo fibers, in which there was also a delay in the setting time, also associated with high amounts of sugar in the fibers [16].

The dissolution of these soluble sugars produce calcium compounds in the cement matrix, which reduce the hydration temperature of the cement and delay the formation of hydration products [16]. A similar delay in setting time was observed in cement composites reinforced with hemp fibers [17], but this was attributed to the presence of pectin contained in these fibers, which acted as a growth inhibitor of calcium silicate hydrates. Other studies also revealed this delay in the setting of wood-reinforced cement compounds [17] and is attributed to the carbohydrates and hemicellulose contained in it. This effect on the setting delay caused by plant fibers could be reduced with the addition of a pozzolana [18] and could also be reduced by using pre-treated fibers containing low amounts of lignin in cement compound [18].

Bagasse is the name for the residue of the sugarcane after its juice has been extracted, which is one of the largest agriculture residues in the world [19-22]. Bagasse contains short fibers, water and small amounts of soluble solids. The bagasse/stalk ratio by mass is around 30% [23]. Sugarcane bagasse has reasonable good tensile strength and modulus, therefore having the potential to be used as reinforcement in composites [24]. On average, sugarcane bagasse fibers have a tensile strength in the range of 170–290 MPa and a modulus of elasticity in the range of 15–19 GPa [25]. Another research presented a tensile strength of 170–180 MPa and a modulus of elasticity of 17–19 GPa [26]. The chemical composition of the sugarcane bagasse fibers has a great influence on the mechanical properties of the compound material, mainly because cellulose, hemicellulose and lignin [27] are responsible for the bonding behaviour of the fibers in the cement paste [14, 28]. Bagasse fibers are also hydrophilic, which means it can be traced in the presence of hemicelluloses and the hydroxyl group in the cell walls, and the high content of these hydroxyl groups in cellulose increases the properties of moisture absorption [13]: Therefore, the presence of hydroxyl groups causes plant fibers to have a variable dimensionality induced by moisture absorption [14]. The moisture content in vegetable fibers has a significant effect on their mechanical properties and on the performance of the composite material [15]. The use of sugarcane bagasse fibers shows improved mechanical properties such as tensile strength, flexural strength, elastic modulus, hardness, and impact strength. It also meets the requirements to be a biodegradable, recyclable and reusable material [29].

The construction sector is a major contributor to environmental pollution, mainly in the emission of greenhouse effect gases [30], which is why it is important to look for new materials that reuse by-products or introduce renewable materials. The mechanical properties of the sugarcane bagasse fiber composites have shown their potential to be used in building materials with suitable properties [31, 32]. Regarding the durability of cement composites reinforced with vegetable fibers, it is well-known that natural fibers are susceptible to deterioration in the cement matrix due to the water absorbed and the weakening of fiber in the alkaline environment [33]. This weakening is mainly due to the dissolution of lignin and hemicellulose that bind the cells of the individual fibers with the alkaline solution deteriorating the capillaries [18]. The degradation is enhanced by the depolymerisation of the fibers induced by alkaline hydrolysis because the glucose molecules that are bound break down and the length of the molecular chain is shortened [33, 34]. The speed of degradation depends on the fibrous morphology and crystallinity of these fibers, and it is reported that the higher the crystallinity of cellulose, the slower the rate [35]. The early carbonation and the reduction of alkalinity through the partial substitution of cement by non-densified silica smoke, was effective to prevent the deterioration of natural fibers of plant origin in cement compounds [36]. In general, the reduction of the soluble alkali content of the cement, the depletion of the portlandite through pozzolanic reactions and the partial replacement of the cement by supplementary cementitious materials, reduces the alkalinity of the cement and therefore helps to preserve the durability of the fibers [36]. Other works establish that treating vegetable fibers by submerging it in polyester, improves its mechanical properties, increases the adhesion of the fibers to the matrix, and prevents the degradation processes [1, 37].

In this work, a study on the flexural strength of Portland cement reinforced with sugarcane bagasse fibers is presented. An effective washing and drying to the fibers extracted from the bagasse was conducted in order to clean the sugar from its surface, and results showed that not further chemical treatment or surface modification was required to incorporate successfully the fibers in the composite, since the overall properties were acceptable besides the increase in the fibers contents. Diverse characterization including Weibull statistics analysis was included.

2. Material and Methods

2.1. Raw

White Ordinary Portland Cement (WOPC) from Holcim S.A., Colombia (max. 6.0 wt% MgO, and max. 3.5 wt% SO_3) was used in this research in combination with sugarcane bagasse fibers. WOPC has been used in order to easily identity the fibers contents and because the results are useful to the regular ordinary Portland cement since the chemistry and reactions are quite similar. The fibers of the sugarcane bagasse was obtained from the waste that results after the extraction of the juice from the cane. In Colombia, sugarcane juice is a popular beverage sold with ice and lemon juice elsewhere, as shown in Figures 1a, b, and c. This bagasse is not used and typically is just placed in landfills decreasing the waste site life. The fiber was separated from the bagasse and then actively washed with soap and water , this in order to remove the residual sugar. Figure 1c shows the sugarcane bagasse, while Figure 1d shows the fiber of the sugarcane after washed and dried. Figure 1 is important because shows the context in which the waste is generated and that any manufacturing process could be more sustainable.

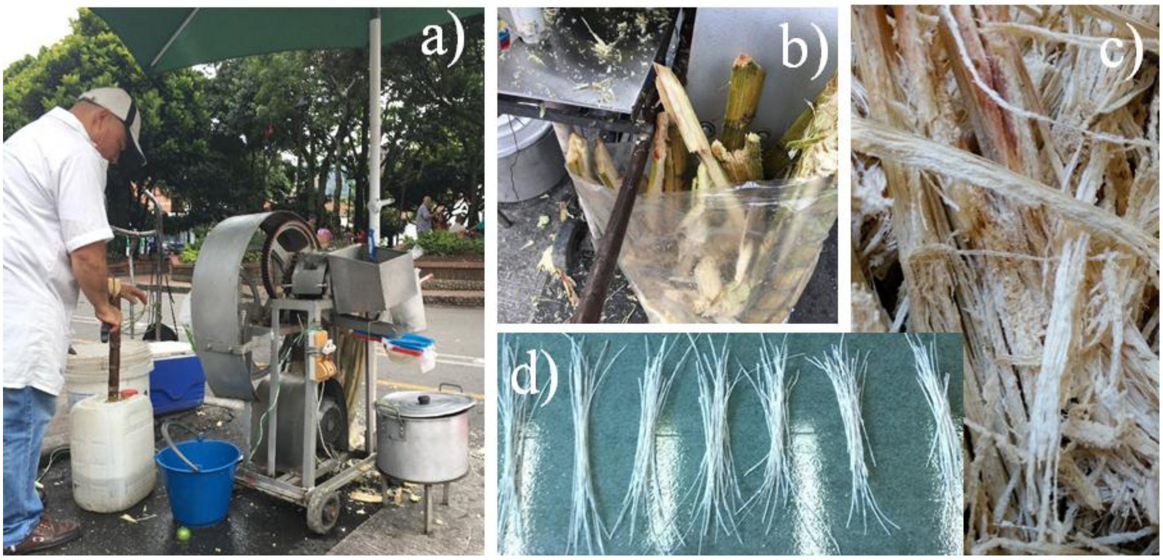

Figure 1: Extraction of fibers from sugarcane bagasse, a) machine used for the juice extraction from sugarcane for beverage; b) raw sugarcane for the machine; c) detail of the baggage; d) selected and cleaned fibers from c).

Portland cement paste reinforced with sugarcane bagasse fiber is investigated, with W/C = 0.6 ratio. The samples were manufactured at 20°C, with cement paste reinforced with 0.0, 1.0 and 2.0wt% of fibers. A total of 20 samples were manufactured for each formulation for mechanical and density tests. Samples were manually poured in moulds, and after 28 days of curing at room temperature, they were released and storage open to air for 2 days. Then, the sharp corners of samples were polished with sand paper in order to reduce potential cracking damage. The manufactured samples were 149 mm long, 10 mm high, and 14 mm wide.

2.3. Characterization tests

Flexural tests were conducted in a universal Shimadzu apparatus at a cross head speed of 1mm/min. A JEOL JSM – 6490 scanning electron microscopy (SEM) was used to observe the microstructure of the samples. Weibull statistics were conducted over 20 samples per composition in order to determine the variability accounted by the Weibull modulus and the corresponding failure probability

3. Results and analysis

Figure 2a shows stress-strain curves for 3 of the tested samples, which reveals multi-peak curves mostly associated with fibers failing and de-bonding from the matrix. Stress (σ) in MPa and strain (ϵ) in mm/mm, were calculated with equations (1) and (2)

$$\sigma = \frac{3}{2}\frac{PL}{bh^2} \quad (1)$$

Strain was calculated with the equation (2):

$$\epsilon = 6\frac{\delta h}{L^2} \quad (2)$$

where P is the Load in KN, L is the length in mm, b is the width in mm, h is the height in mm, and δ is the deflection in mm.

Figure 2b show that the flexural strength increases with just 1.0wt% of fibers, and even more when fibers are 2.0wt%. This increase is enough to justify the use of these fibers when flexural loads are applied. More than 2.0wt% is hard to introduce due mainly to the high volume of the fibers (1.0 and 2.0wt corresponds to about 7.5 and 15v/v% of fibers), which limits the cement amount to wet and therefore work effectively as binder in the composite, which can generate a lot of voids with the corresponding negative results for the strength. However, thermal and noise insulation properties could be greatly improved with higher fibers contents, besides the lower strength. Figure 2c summarizes the ductility of the samples. As the fiber content increases, the ductility increases, which is the main advantage of having fibers in the cementitious matrix and was the main goal of this composite manufacturing. Figure 2d shows the density values for each formulation. As expected, as the fiber content increases, the density decreases with a near linear trend. Sample reinforced with 1.0wt% fiber decreases its density in 0.05 gr/cm^3 and sample with 2.0wt% decreases in 0.16 gr/cm^3, both with respect to the pure cement paste. Although this density reduction is known elsewhere to reduce the mechanical strength (mainly due to fiber agglomeration and to an increase in porosity as a lack of matrix impregnating the fibers), as explained before, the porosity can be also beneficial for lightweight composites and low thermal conductivity applications.

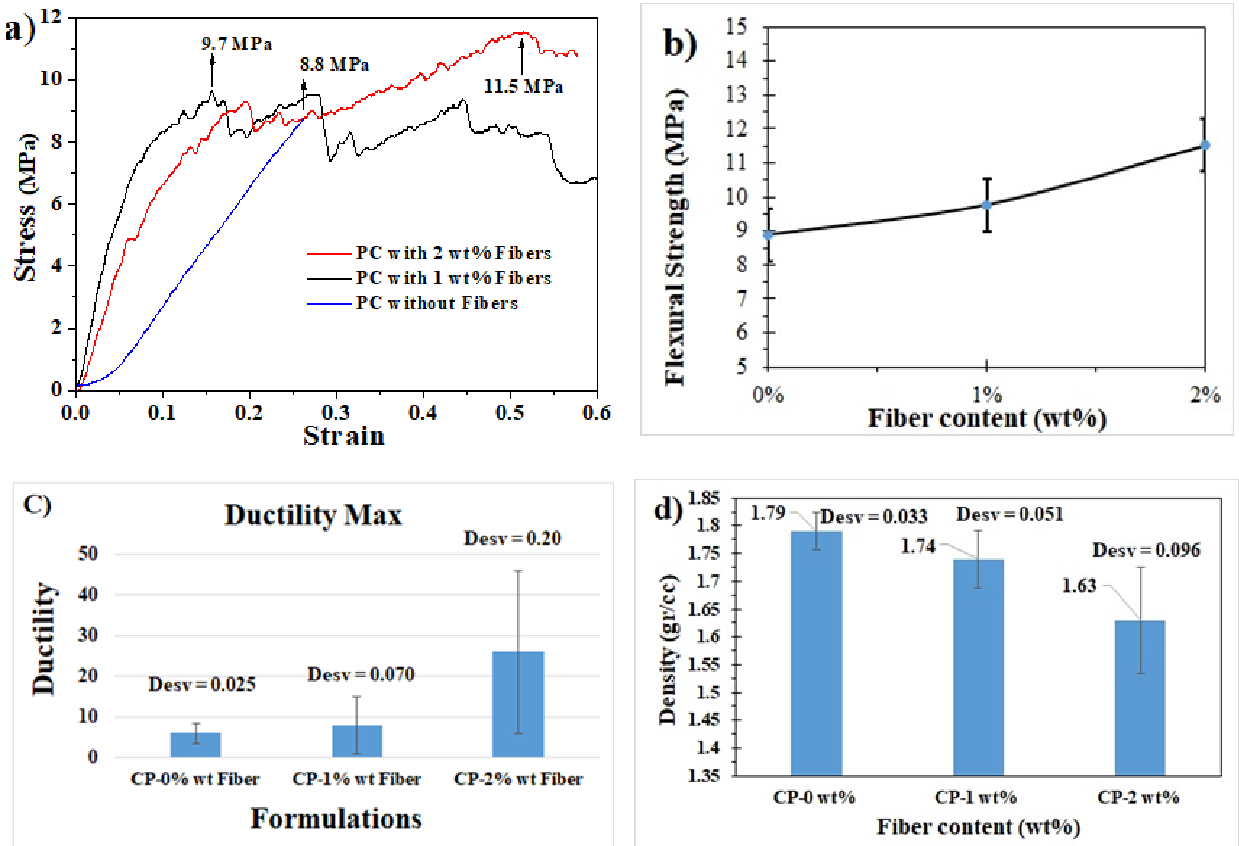

Figure 2: Flexural strength and density results for samples: a) typical strain-stress curves, b) flexural strength mean values, c) ductility, d) density.

Figure 3 shows an overview of the morphology of the fiber with scanning electron microscopy (SEM), all prepared after the cleaning process. Figures 3a, c, d and f, are focused on the external morphology of the fiber.

From images, fibers after the washing process still reveal the softer morphology of the fibers, the matrix working as the binder phase. Figures 3b and d reveal the microstructure inside the fiber, this is the shape and size of their capillary vessels, with an irregular distribution of their capillary vessels in both diameter and numbers in each fiber. Figure 3b and e evidences the high capacity of these fibers to accumulate water, which can has a tremendous effect on the mechanical properties of the compound and its performance, mainly decreasing their life in the long term [38], and is the reason why the bagasse has to be carefully prepared by a good cleaning and drying. ImageJ software was used to make an analysis of the images.

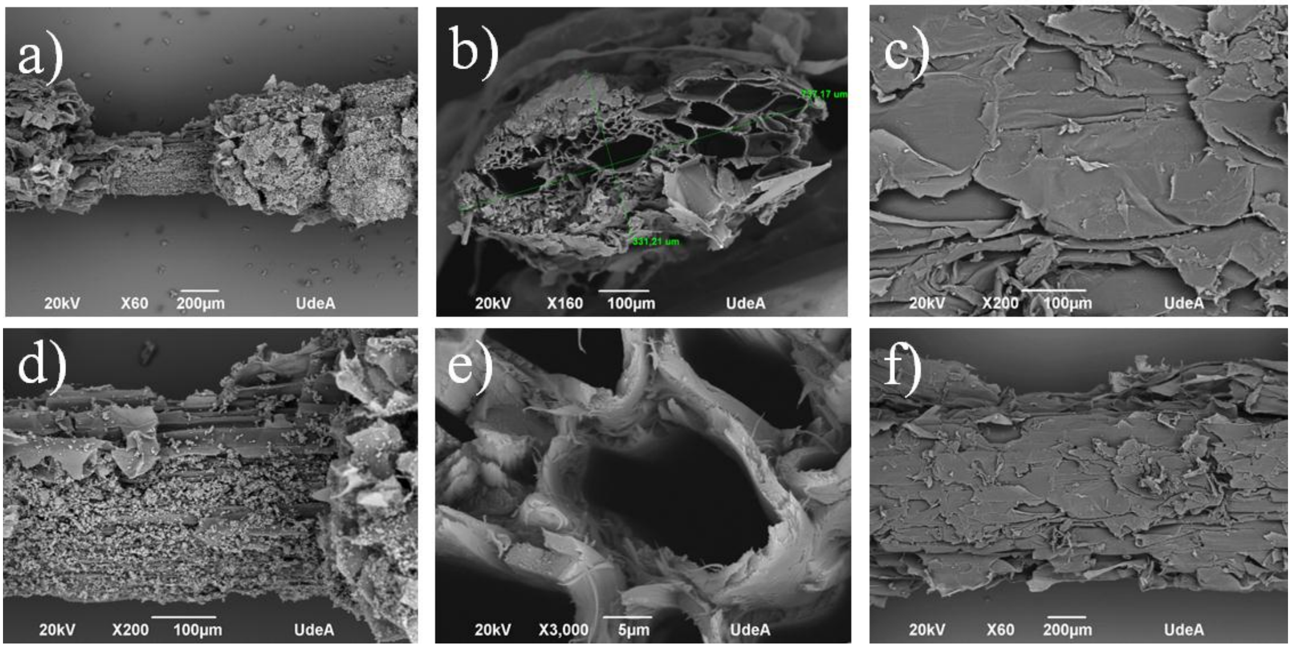

Figure 3: SEM images of sugarcane bagasse fibers after the cleaning process.

The diameter of each of the capillary vessel of the fiber was measured, then the probability distribution adjusted to a normal distribution was calculated, and their results are shown in Figure 4. The diameter distribution centers on 43µm, showing that is not fully a normal distribution. Their size distribution effect of these vessels on the mechanical properties of the concrete are hard to estimate and need to be further investigated, although due to the high heterogeneity of these in a single fiber, their control is almost impossible in a natural material as bagasse. However, if a bio-inspiring material is built, its control via precision manufacturing such as 3D printing, can take advantage of the optimal vessel diameters.

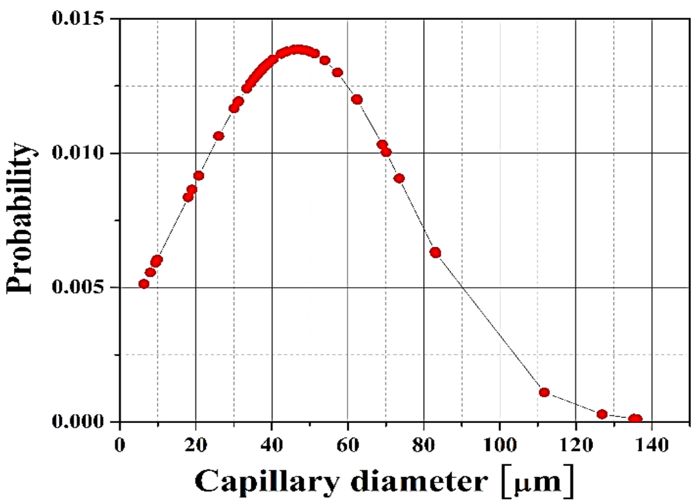

Figure 4*:* Pore (capillarity) diameter distribution for the fibers.

Figures 5a and b show the SEM images of the pure cement paste; while Figures 5c, d, e and f show cement paste reinforced with 1 and 2wt% sugarcane bagasse fibers. Figures 5a and b show the porosity of the pure cement paste. Samples with fibers revealed fibers well impregnated by the cement paste, although at the some lack of filling and micro-cracking is observed. Therefore, besides the reasonable good results in the strength, an optimized manufacturing process can significantly increase the strength values. It is also seen that as fibers content was increased from 1 to 2wt%, see Figures 5d and f, the porosity in the cement matrix was increased, and therefore higher fibers contents will be very detrimental for the mechanical stability of the composite.

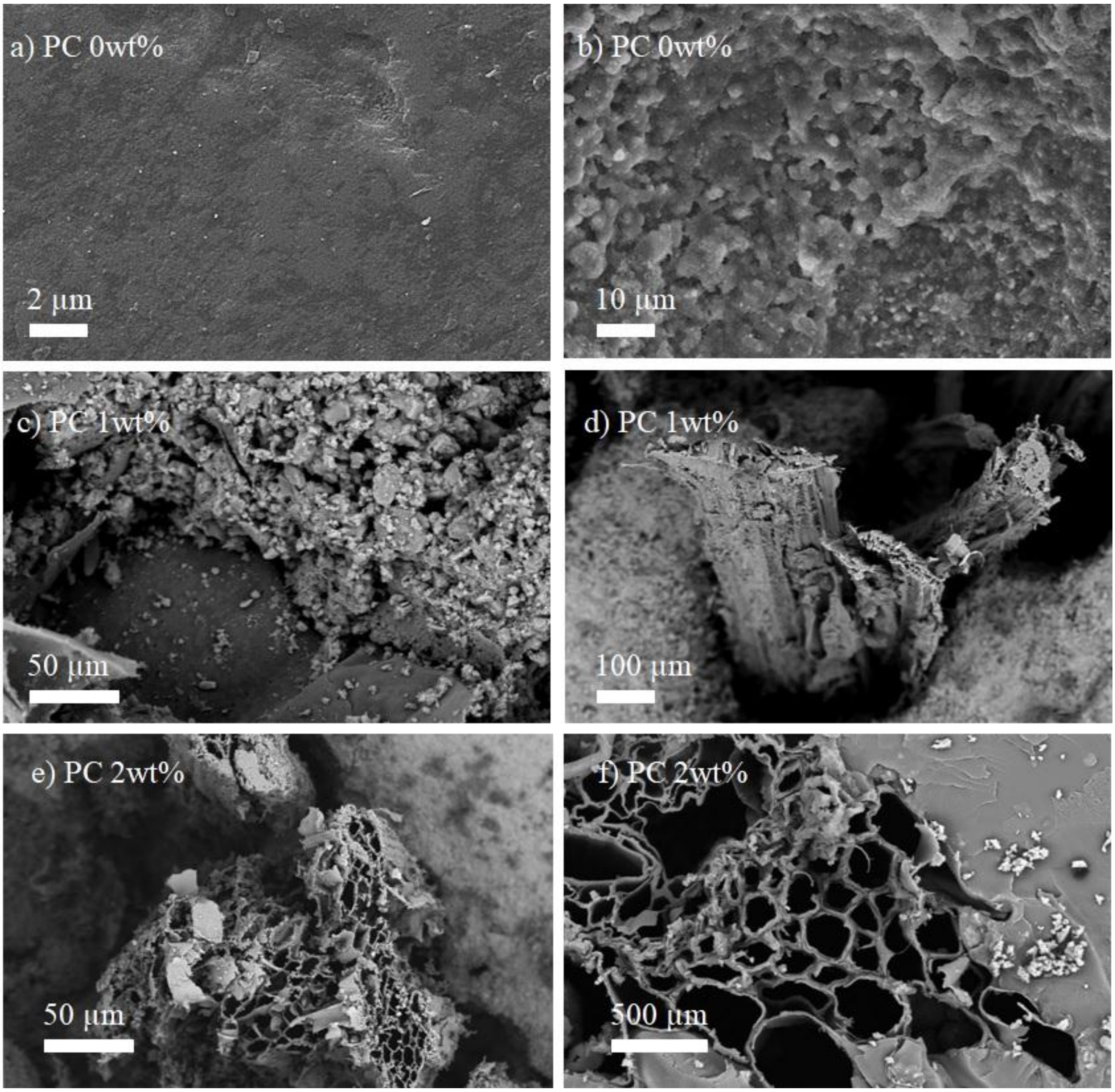

Figure 5: SEM images of cement paste reinforced with sugar cane fibers

Finally, the Weibull distribution data is presented in Figure 6. In this research, 20 samples were tested per composition, and these numbers were also summarized before with regular statistics, showing the mean and standard deviation in Figure 2.

The probability of failure is presented in Figure 6a, where the flexural strength varies from approximately 6.2 to 15 MPa for all formulations. This curve shows the increase in flexural strengths as the fiber content increases.

The curve given in Figure 6b shows the linear equations to calculate the Weibull modulus (WM), which is defined by the slope of the line: 6.326, 4.788 and 7.737 for cement paste reinforced with fibers content of 0.0, 1.0 and 2wt%, respectively. Typically, the slope for the pure cement paste is higher than for cement with natural fibers, however, in this research, the opposite was found, which can be attributed to a correct selection of the fibers, which have good properties that clearly enhanced the matrix. The cleaning process must be also very important, as removed weakest parts, but also perhaps dissolved components that decrease the mechanical properties.

Figure 6c shows the variation of the WM with the fibers content. The highest value for the WM corresponds to the lowest variability. Therefore, the samples with 1wt% of fibers surprisingly were the composition accounting the highest variability, which is reflected in the positive concavity of the parabola (this curve is just for reference, as more point are needed in order to stablish a trend, which can vary a lot if other fibers are used. Conversely, sample with 2wt% of bagasse fibers are the best in terms of the WM, therefore corresponding to samples with the lowest variability. An important aspect of the WM is that it is a statistical parameter which establishes probable values at the macro mechanical level regardless of the micromechanical dynamics such as the interfaces and the interactions that can be made with them. The increase in WM can be associated with fiber elasticity, which increases the overall variability of each formulation. The values found in this research are typical for cementitious composites as presented elsewhere [38].

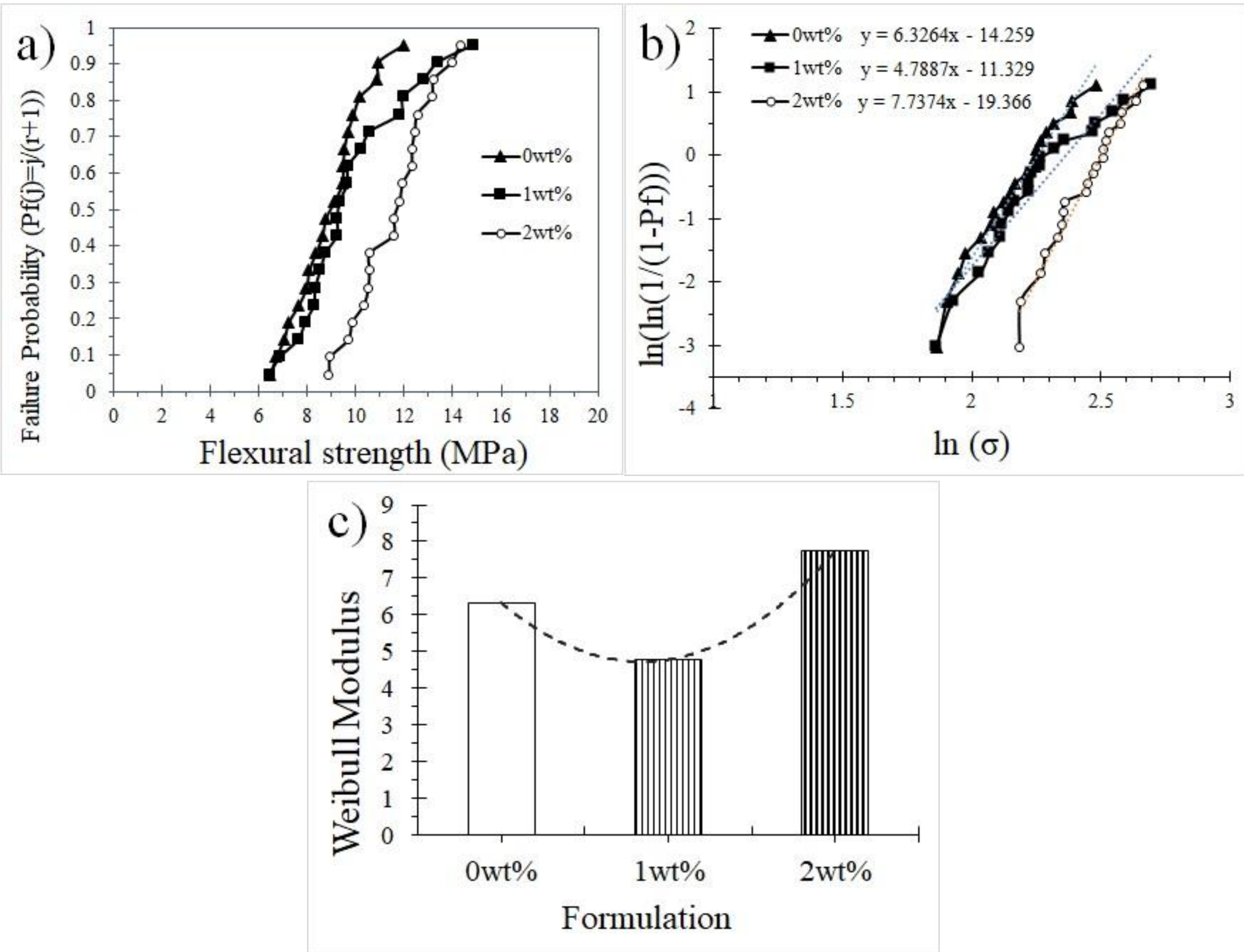

Figure 6: Weibull distribution results for the different samples

The first aspect found in this investigation is that the procedure followed to make the bagasse-cement composites was successful to increase the strength of the pure cement past, which open two conclusions regarding the inexpensive and simple process and also considering that no further fiber treatment was used. Results suggest that not further processing for these fibers is necessary to make these cement composites, which similarly has been observed for other natural fiber composites, where the process rather than improve the strength, in some cases can deteriorate the fibers, and therefore decrease their life and with this, the composite properties [9-12]. It is important to say that at the beginning of this research, some problems were faced regarding the long setting times, which led to a low strength cement. This issue was due to a small presence of sugar in the fibers, which is known to delay the setting time in Portland cement. The problem was solved by a very complete cleaning process using soap and water over the fibers [39] that guarantee they were free of sugar cane.

On the other hand, bagasse fibers occupy a high volume in the composite and this is the main reason because only up to 2wt% of this was used in this research. Thus, fiber content could not be exceeded 2 wt% because there is no workability, and it was not possible to manufacture a good composite. As expected, fiber content increase tends to decrease the mechanical strength, although can have beneficial properties in applications such as noise and thermal insulation. Since Portland cement is a hydraulic cement, typically water continue reacting and decreasing with time, and thus the pores can increase with time These pores have a complex interaction with the fiber diameter distribution and an unknown effect on the vascular vessels inside the fibers, which can be fully explore regarding thermal and noise insulation applications.

However, because the nature of the fibers makes them variable in diameters and properties regarding the environmental and soil conditions (pH, nutrients, etc), the conclusions have to consider the wide variability of results. Considering the results presented in this investigation, several of the emerging cementitious matrices can be improved with bagasse fibers, such as calcium aluminate cements (CAC), a cement of high projection as a construction material for its early strength and resistance to chemical attacks [41, 42]. The materials fabricated in this investigation could be used in applications that do not involve high loads (of the order of 10 MPa), or applications with materials exposed to high temperatures, potential fire hazards, or large buildings. These composites can be used in sidewalks, city furniture, facades, or decoration walls [42].

Discussion

The results found in the present investigation show a simple path to give a different value to part of the sugarcane bagasse which is produced in many countries worldwide, its fibers, used in a composite after an inexpensive cleaning process. These fibers can be utilized in construction materials such as concrete and other cementitious materials. The fibers not only can give ductility to the cement based materials, but also can make it lightweight and can reduce the cement consumption as well, which is a direct impact in the reduction of the CO_2 footprint for these materials.

Regarding the compressive strength results, the fiber content improves the compressive strength, 9.7MPa, 8.8MPa and 11.5MPa for cement paste with 0, 1 and 2 wt% fibers contents, respectively. These results and the corresponding Weibull analysis has not been reported before, and from particular interest is the fact that Weibull modulus has increased with the fiber contents, indicating a more reliable material, which is associated with the selection and preparation of the fibers.

Also, the mean value of the compressive strength increases, however, considering the error bars, the compressive strength seems to be similar. This is not a bad result at all for two things. First, the fact that strength is not significantly reduced with increasing the fiber content is very important, as the amount of fibers by volume is quite important. Therefore 1.0 or 2.0wt% of them in the cement means a lot of waste in the composite (1.0 and 2.0wt corresponds to about 7.5 and 15v/v% of fibers). Second and perhaps more important, is the possibility to reduce cement contents, which is also a positive contribution for the environment.

Conclusion

Portland cement-bagasse fiber composites were successfully made with 0, 1 and 2wt% of fibers, showing a significant increase in the bending and ductility values, upon the low fiber contents. Since no further treatment to the fibers was required, is concluded that is not required for the materials, conditions and applications discussed in this research. This made the process more economical, simple, and ecological.

The investigation revealed that in general with a higher fiber content, the flexural strength increases, density decreases, and variability did not show a clear trend.

This research also showed a solution for the sugarcane bagasse in Colombia, which is currently mostly put in landfills and therefore not only not used at all, but also decreasing the useful life of the landfill.

These fabricated composites can be used in applications that do not involve very high loads, exposure to fire, or long-term buildings. These can be sidewalks, facades, or decoration walls.

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