Mechanical performance of mortar mixes reinforced with hemp and fique fibers treated with sodium silicate

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Abstract. Natural fibers have been a replacement option for the plastic fibers used in mortar mixtures, given their ease of achievement and lower extraction cost, this gives them a strong advantage over their potential application as reinforcement. Hemp and fique were added to the mortar matrix, these fibers were superficially treated with sodium hydroxide at 1%, 2%, 5% and 10%. Subsequently, the fibers were immersed in a sodium silicate solution for 24 hours, extracting them and allowing them to dry at room temperature. The fibers were chemically characterized using infrared spectrophotometry, scanning electron microscopy, and gravimetric techniques. Mortar cubes and mortar beams were made evaluating their mechanical performance with the addition of 1% of natural fibers with and without sodium silicate treatment. The results showed that of the untreated fibers, that of hemp is the most resistant compared to that of fique, and the chemical impregnation treatments with sodium silicate improve the mechanical properties of the fibers, in this way the addition of reinforcement in the mortar and the treatment used to improve the mechanical properties of the fibers added in the mixtures improve the flexural strength compared to the control specimen.

1. Introduction
The interest in natural fiber-reinforced polymer composite materials is rapidly growing both in terms of their industrial applications and fundamental research. They are renewable, cheap, completely, or partially recyclable, and biodegradable [1]. In fact, fiber reinforced cementitious composites are generally characterized by higher tensile and flexural strength, tougher and more ductile post-cracking behavior and superior durability performance [2], so since the fibers promote a "bridging effect" through cracks in the binding material, they allowing a better response in the post-peak branch of stress strain relationships [3] In this way, to improve the resistance characteristics of the fibers, they must be subjected to an alkaline treatment and an impregnation with an inorganic compound in such a way as to improve their mechanical characteristics.

The use of natural fibers reduces weight by 10% and lowers the energy needed for production by 80%, while the cost of the component is 5% lower than the comparable fiber glass-reinforced component [4]. The building construction industry is not only a major consumer of energy, raw materials, and land; it also contributes immensely to environmental pollution, especially greenhouse gas (GHGs) emission. To improve sustainability in construction materials usage, the construction industry must embrace the reuse of industrial by-products and renewable materials in construction. [5] Incentive in tax reduction is
also another way to motivate different commercial units to design and make products with "Green" components.

The cost of natural fibers is relatively low as they are abundant and from renewable resources compared with other synthetic fibers. Therefore, such remarkable advantages of the natural fibers reinforced polymer composites enhance their commercial and research potentials. Accordingly, natural fibers and biodegradable polymers have become emerging materials in the composite community. Natural fibers have a potential as a substitute for artificial fibers derived from petroleum or materials of non-renewable origin in many applications, allow the development of ecological compounds that respect the environment, largely due to the biodegradability of natural fiber, weight light, relatively low cost, high specific resistance, natural abundance and then its supply, fast replenishment capacity (compared to non-renewable products), these are some of the strongest arguments to use them in the composites [6,7].

2. Materials and methods

Two types of natural fibers were used for the study, fique (Furcraea Andina) that is showed in the Figure 1(a) and the hemp (Cannabis Sativa L) in the Figure 1(b), a member of the Bromeliaceae family, native to South America, it was characterized and evaluated to determine their physical and mechanical characteristics. Fique was extracted mechanically from the leaves, while the hemp was extracted from the stem of the plant.

The two fibers were selected and guarded in the laboratory for the different physical and mechanical tests. The fibers were submerged in sodium hydroxide (NaOH) in four different concentrations (w/w), 1% w/w, 2% w/w, 5% w/w and 10% w/w, leaving them submerged in four different times at 1 hour, 2 hours, 8 hours, and 24 hours, to later wash each one with distilled water and leave them in oven at 30 degrees centigrade for 24 hours for drying. After this process, the fibers were immersed for 24 hours in sodium silicate (Na$_2$SiO$_3$), at a concentration of 68%, which after being baked in the oven at 30 degrees for 24 hours, were added to the cement mixtures.

![Figure 1. (a) Fique and hemp fibers treated with sodium silicate; (b) no treatment.](image)

To determine the effectiveness of the treatment on the fibers, infrared analysis by Fourier transformation (FTIR) was carried out using a Thermofisher equipment. The characterization was used to analyze the variation of the functional groups on the surface of the fiber after impregnation, using a Shimadzu FTIR, IR Prestige-21, with a resolution of 2 cm$^{-1}$, using the configuration attenuated total reflection (ATR), likewise, a The Thermo Scientific DXR SmartRaman spectrometer equipment was used, with which the response trials of the fiber treatments were carried out. Finally, a scanning electron microscope (SEM), JEOL 6000 Plus, was used at 15 KV, with which it was possible to determine the surface characteristics of the fibers without and with treatment.
Tensile tests were carried out following the protocol of ASTM D 3822 [8], in a polymer traction machine calibrated and conditioned for this test, obtaining force displacement curves. The speed used was established according to the length of the same at 1.5 mm/min, and 10 mm/min repetitions were carried out for each type of fiber. The diameter of the apparent cross section was determined with a ZEISS Primo Star/Primo Star iLED optical microscope, with a 4X objective, taking digital images with an AxioCamER camera conditioned to the equipment, and carrying out the analysis of the image with a software Zenlite 2012. For the elaboration of the test pieces that were tested in the tensile machine, the fibers were selected in such a way that they had similar diameters in such a way that the results were comparable.

Two tests were carried out to evaluate the effect of the addition of the treated fibers, standard ASTM C 109 [9] and standard ASTM C 348 [10], they were used to measure the mechanical properties of compression and flexural mortar, respectively. For the tests, a universal Shimadzu UK500 machine was used, which allowed the taking of the test data. The sand used for the preparation of the mortars was normalized with a commercial sand (20-30) from the city of Sogamoso, Boyacá, Colombia, fulfilling the norm ASTM C 778 [11], the cement used was a cement of General Use commercial brand Argos, and the water cement ratio used was that necessary to produce a flow rate of 110 +/- 5 in the flow table, established in the standard ASTM C 230 [12].

3. Results
When observing the untreated fibers under the scanning electron microscope, the change in diameter of the fique fiber is noted, see Figure 2 with respect to the hemp see Figure 3, of these the diameter of the fique fiber is greater compared to the hemp fiber.

![Figure 2. SEM of fique fiber untreated.](image1)

![Figure 3. SEM of hemp fiber untreated.](image2)

In the fibers it is observed that the impregnation applied by the treatment with sodium silicate was effective in the two types of fibers, see Figure 4 and Figure 5, its adherence being greater in hemp fibers than in those of fique, the foregoing due to that hemp fibers have more microfibers available to adhere to the compound of the applied treatment. The treatments with sodium hydroxide increased the roughness of the fibers and therefore the adherence with sodium silicate was more effective in the fibers of fique and hemp.

Figure 6 and Figure 7 show the FTIR of the treated fibers, fique treated (FT) and treated hemp (HT) are observed, in the same way the untreated fibers, FUT and HUT. Through the analysis of the fibers by the FTIR technique on the fique fibers see Figure 4 and the hemp fiber see Figure 5, characteristic bands of the components of the natural fibers were evident at 3343 cm⁻¹ and 3314 cm⁻¹, that are attributed to the hydroxyl (OH) groups. The bands at 2897 cm⁻¹ and 2920 cm⁻¹ are attributed to the methyl functional groups (CH) and finally the bands 1028 cm⁻¹ and 1030 cm⁻¹ are attributed to the aromatic groups of the fibers, these groups are associated with the structure of the cellulose, hemicellulose, and lignin.

When comparing the FTIR spectra of the fique and hemp fibers, it can be observed that, when applying the alkalization treatment, the bands at 2897 cm⁻¹, and 2920 cm⁻¹ attributed to the -CH₃, CH
and C=O bonds disappear these links that are present in the lignin of vegetable fibers. For the band at 1730 cm\(^{-1}\), and 1734 cm\(^{-1}\) representing the carbonyl groups of the fibers associated with the hemicellulose and the pectin were removed by the alkaline treatment.

![Figure 4. SEM of fique fiber treated.](image)

![Figure 5. SEM of hemp fiber treated.](image)

![Figure 6. FTIR of fique fiber no treatment and treated.](image)

![Figure 7. FTIR of hemp fiber no treatment and treated.](image)

The results of the tension tests, made in the two fibers, the one with the highest strength value was the fique fiber see Figure 8, followed by the hemp fibers in Figure 9, with load values of 0.82 Kg and 0.52 Kg, respectively. To quantify the area, optical images of the cross-section of the fiber were used, and the net area of each of the study fibers was determined, these areas allowed the calculation of the stress. The percentages of elongation of the fibers were in the range of 6% to 9%, as observed in Figure 8 and Figure 9.

![Figure 8. Force-displacement curves fique.](image)

![Figure 9. Force-displacement curves hemp.](image)
From the evaluated percentages of sodium hydroxide in the fibers, an optimal time range of 7.5 hours was obtained with a percentage of NaOH solution between 1% and 2%, these results were obtained from a superficial analysis of the stresses of the fibers, depending on the concentration and the treatment time of the fiber.

The results of the compression tests, Figure 10, show that the addition of fique and hemp fibers without treatment decrease the resistance by 13.8% and 10.8%, respectively, the above compared to the control specimen. Similarly, the addition of treated fiber decreased the strength of the mortar in percentages of 11.9% and 18% for the hemp and fique fibers. In general, the fibers decrease the compressive strength of mortars as they induce more pores in the cement paste.

The results of the Flexural tests, Figure 11, show that the treated fibers (T) with respect to the untreated fibers (UT) tend to increase their adherence with the cement paste, for this reason, the percentages of the modulus of rupture of the beams of Mortars were favored by increasing their resistance by 8% and 7% for hemp and fique fibers, respectively. Venkateshwaran, et al. [4] found similar results with 4% improvement with banana/sisal fibers, this clearly shows the advantage of treatment with sodium hydroxide and the adherence of sodium silicate to natural fiber.

4. Conclusions
It was evidenced that the optimal treatment for alkalinization is 7.5 hours, with a percentage between 1% and 2% of sodium hydroxide for the fibers studied. The hemp fibers presented a better performance as an addition to the tested mortars, compared to the fique fibers, in the same way the analysis by Fourier transformation tests showed a good performance with the studied treatments, this reflected in the analyzed bands.

The results of flexural strength of mortar, evidence that the treatments to the vegetable fibers of fique and hemp can be favorable in the cement pastes to specifically improve flexural properties, values of 8% and 7% were the improvement results obtained from the hemp and fique fibers, respectively; Regarding the compression properties of the mortars studied, it was evidenced that there is a decrease in resistance in all the additions studied, with respect to the control specimen.

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