Innovative use of residual biomass from agricultural processes. Case study: Use of Oil Palm and Sugar Cane Bagasse fibers for the improvement of construction materials

Tolosa R^{1, 3}, Solarte J¹, Restrepo D¹, Ramírez V³, Rojas D³, Arias N^{1, 2}, Giraldo O^{1, 2}, Cardona C¹

¹ Group of Chemical, Catalytic and Biotechnological Processes. Universidad Nacional de Colombia, Manizales

² Nanostructured and Functional Materials Laboratory. Universidad Nacional de Colombia, Manizales

³ Architecture and Urbanism School. Universidad Nacional de Colombia, Manizales

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Introduction

Currently, a green conscience has been generated, which is focused on an environmentally friendly economy, based on the use of natural resources that provide renewable raw materials, which are of great importance in the field of construction, as it helps Mitigate the environmental impact it generates in many ways. Natural fibers are an invaluable resource for biodegradable and supporting environmental conservation.

Natural fibers are complex, filamentous structures of plant, animal and mineral origin. By their physical, chemical and mechanical characteristics, have very diverse applications. Its use has been very remote, since times as old as the humanity itself [1]. Fibrous plants are of great importance, after food for their social and economic influence. About 35 million tons of fiber are harvested each year from a wide range of plants and animals. Many of these have been used in the manufacture of new materials [2].

With the appearance of synthetic fibers such as glass, carbon, aramid and polypropylene, the natural has been significantly displaced. The success of the synthetic has been due to its easy access and effectiveness, however the natural fibers have a good mechanical strength, light weight, and are a renewable resource par excellence, since at the end of their life cycle, they are biodegradable.

Plant fibers are found in roots, stems, leaves, fruits and seeds. These can be extracted by different chemical or mechanical processes such as: Rust, fermentation, steam explosion, kraft, and alkaline method, among others.

The vegetable fibers have a more complex morphology and chemical composition, whose main components are: Cellulose, hemicellulose and lignin [3]. These are called as the botanical components of a plant. Plant material is commonly called cellulose. However, it is actually composed of different components, the main three being lignin (26-32%), cellulose (40-45%) and hemicellulose (26-32%) and minority compounds, tannins, waxes, Alkaloids and inorganic elements representing less than 3% ash [3]. This composition varies according to the type of plant, its vegetal origin and maturity.

Lignin is a macromolecule of complex structure in charge of binding, as an adhesive, the individual cells (hereinafter called fibers) of the plant tissues. Therefore, the destruction of these bonds between the fibers is necessary. This process can commonly done by applying mechanical, thermal extraction, chemical energy or a combination thereof, in order to obtain individual fibers [4]

The processes of fiber extraction vary according to the type of plant, its plant origin and maturity. For which it is of great importance the knowledge of the different methods consulted in literature.

Methodology

Fiber extraction

The extraction process of the fiber was performed using a cylinder fitted with a 2 hp motor. The lignocellulosic material was cut into approximately 2-3 cm strips to facilitate extraction. Strips of material were passed continuously through the cylinders, starting with a separation between them of approximately 1 cm, and this step is repeated several times while decreasing the gap between the cylinders until the gap reaches 1 mm, thereby enabling the constant separation of the fibers. After this step, the fibers were dried at room temperature for 48 h and then manually cut into small pieces of 0.5 and 2.5 cm, with the goal of studying the influence of the fiber factor to the mechanical properties of the composite material [5].

Scanning Electron Microscopy.

Micrographs were obtained using a JEOL JSM 5910LV microscope with secondary electron (SEI) detectors, operated at 15 kV in high-vacuum mode. For this analysis, 3 mg of the synthesized material was placed onto carbon ribbon. The set was subjected to vacuum from 100 to 50 mTorr, and then gold was sputtered for 120 s to coat the sample to minimize the effects of charging in the scanning electron microscopy (SEM).

Structural characterization.

The X-ray diffraction patterns for the evaluated lignocellulosic materials were acquired in a equipment RIGAKU MINIFLEX II using radiation of Cu K α at 30 kV and 15 mA with an

acquisition speed of 2° (2 θ) min⁻¹, a sampling amplitude of 0.02° (2 θ) and a rank of 5° a 40° (2 θ).

The percentage of crystallinity (%Crys; relationship between the crystalline part and amorphous part of cellulose) was calculated by the method reported by Segal *et al* [6]. Through the formula

$$\% Crys = \frac{(I_{002} - I_{am}) * 100}{I_{002}}$$

Where I_{002} is the intensity of the diffraction peak of the plane (002), which is located approximately $2\theta \approx 22.6^{\circ}$ for cellulose I and Iam is the intensity of the baseline measured at $2\theta \approx 19^{\circ}$ for cellulose I.

Results

Figures 1 and 2 show images using scanning electron microscopy. Which were taken to the section of the palm, there can be observed that the diameter of the fibrilos is variable. However it is on the order of less than 15 μ m and the wall which is formed by cellulose bound by lignin and hemicellulose, is less than 4.35 μ m. In addition, it should be noted, as will be indicated below, that the fiber agglutinates several fibrils, which have the appearance of micro tubes. This morphological configuration is what gives plant resistance.



Figure. 1. SEM Cross section of oil palm, diameter fibrils

Figure. 2. SEM Cross section of oil palm, wall thickness

Figure 3 shows a larger increase of the microscope structural configuration of the section. There, the micro tubes that make up the plant material are observed with a higher level of detail, a configuration that is similarly repeated in the majority of the natural fibers reported in the literature.



Figure 3. SEM Cross section of oil palm, wall thickness

A similar case is observed in the section of cane bagasse, which is presented in Figure 4. It is observed that the micro tubes of the bagasse are larger than those formed in the palm, being of the order of almost 140 μ m, as seen in the approach made in Figure 5. It is also noted that the wall is much thinner than that observed in the palm section.



Figure 4. SEM Cross section of bagasse, diameter fibrils

This morphological difference supports the hypothesis that the smaller cell size and greater wall thickness, give a greater structural capacity to the palm fiber. Likewise, the thicker the wall indicates that there is a greater presence of cellulose, and of lignin and hemicellulose as binding material.



Figure 5. SEM expanded from cross section of bagasse.

The use of SEM proves to be an invaluable tool, since it allows to establish that the greater capacity that the fiber can contribute to the composite material will be given when the fiber reaches a size that approaches the one of the micro tube, maintaining an important relation of thickness of wall. Because this would increase their mechanical behavior.

On the other hand, the alkalinity of the matrix degrades the fiber, as explained Tonoli *et. al* [4]., the high alkalinity of the water raises the pH by degrading the fiber and decreasing the useful life of the composite material. Tonoli also notes that to improve the interface between fiber and matrix, techniques such as mechanical treatment of the pulp, chemical modification of the surface, use of additions, reduction of the A / mC ratio, autoclaved curing and cementant matrix carbonation are used .

Considering the above, it decided to apply the technique of mercerization, since as noted Sampieri, *et. al.*[7], it is possible to remove the lignin, hemicellulose and improves the adhesion of the fiber with the matrix, which contributes to increase the mechanical behavior and durability of the composite material by the union of the fiber with the matrix.

In this work, it was also sought to establish, following the procedure indicated by Tolosa *et al* [5], the fixation of manganese to the fiber, with which it is expected to provide an additional coating that increases the adhesion of the fiber to the matrix, which is made with cement base hydraulic. It is also intended that this treatment increase the durability of the composite material.

In Figures 6 and 7, the texture of a fiber of bagasse and palm, without any treatment, is shown. There it is possible to observe how its texture is smooth. This characteristic limits the adhesion of the fiber to the matrix, since the fiber could present pulling due to lack of





fixation to the matrix.

Figure 6. SEM bagasse fibre.

Figure 7. SEM palm fibre.

To evaluate this effect, the fiber was treated with NaOH and KMnO₄. Figure 8 shows the images by scanning electron microscopy of the cane bagasse fibers obtained with the mercerization technique, without coating with NaOH y KMnO₄.



Figure 8. SEM cane bagasse fibers obtained with the mercerization technique

It can be seen that the diameter differs along the fiber, in addition it can be seen how the mercerization is favorable, since it leaves a rough surface, which is appropriate, since as indicated above it improves the adhesion to the matrix and increases the mechanical capacity of the composite material.

Figure 9 shows the images by scanning electron microscopy of the palm fibers obtained with the mercerization technique, without coating with NaOH y KMnO₄.



Figure 9. SEM cane bagasse fibers obtained with the mercerization technique

It can be observed when comparing with the fiber that did not have this treatment, as it increases the roughness of the same, this aspect is key because it allows to determine that its adhesion to the compound will be greater.

Finally, Figure 10 shows the bagasse fiber coated with NaOH and KMnO₄, where it can be observed how the treatment increases the roughness of the fiber with respect to the fiber without mercerization and the mercerized. This is evidence of the adhesion of the coating.



Figure 10. SEM bagasse fiber coated with NaOH and KMnO₄.

Figure 11 shows the palm fiber coated with NaOH and KMnO4. As with bagasse, it can be stated that the treatment increases the roughness with respect to the fiber without mercerization and the mercerized fiber.



Figure 11. SEM bagasse fiber coated with NaOH and KMnO₄.

By way of conclusion, it is possible to indicate that the structure of the fiber improves with the treatments and that, based on what is reported in the literature, the fiber in the final condition, coated with NaOH and $KMnO_4$, will improve the mechanical behavior of the composite, Flexion and impact, as well as the material's ductility and durability.

Now, in order to establish whether the fiber is effectively coated with manganese, the results of the X-ray diffraction made on the cane and palm bagasse fibers to which the mercerization treatment and the same fibers were applied by applying the treatment with NaOH y KMnO₄.



Figure 12. Comparative X-ray diffraction pattern for cane bagasse before and after coating



Figure 13. Comparative X-ray diffraction pattern for African palm before and after coating Table 1. Percentage of crystallinity for the evaluated materials

Material	I ₀₀₂ Cellulose I	I _{am} Cellulose I	% Crystallinity cellulose I
Palm	860	548	36.28

Covered palm	1006	721	28.33
Bagasse	776	512	34.02
Covered bagasse	775	487	37.16

The results show a decrease in crystallinity for the coated African palm possibly due to destruction of the amorphous part of the cellulose structure whereas for cane bagasse the results indicate an increase in crystallinity.

Conclusions

The method of extraction with Sodium Hydroxide (NaOH) is effective to break the lignin chains and facilitates the obtaining of fibers of greater length, less radius and free of impurities, taking into account the times necessary to avoid the degradation of the same.

The coating of cane bagasse and oil palm with Potassium Permanganate (KmNO4) and Sodium Hydroxide (NaOH), increases the roughness with respect to the mercerized and mercerized fiber, allowing adhesion to the concrete paste, it also prevents the alkaline components of the cement from attacking and degrading the fiber.

The experimental exercise with Scanning Optical Microscopy (SEM), helps to determine the greater capacity that the fiber can contribute to the composite material will be given when the fiber reaches a size that is close to that of the micro tube, maintaining an important ratio of thickness of Wall, because this would increase its mechanical behavior

The use of natural fibers poses a focused view towards green construction, with the use of natural resources available, generating labor, contributing to an impact on social, cultural and economic aspects.

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