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Effect of Ultrasound and Mechanics Agitation on Coconut Fiber in Alkaline Solution to Obtain Microfibers

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ABSTRACT: Vegetable fibers are important and promising sources of raw materials for composite materials due to their significant mechanical properties and environmentally friendly nature compared to synthetic fibers. Their abundance and the potential for cleaner production processes further enhance their attractiveness for the development of new composite materials. In addition, cellulosic nanostructures can be obtained from the decomposition of plant fibers.

The use of nanofibers in advanced materials represents a promising approach, given their enhanced properties, such as higher polarity, improved mechanical strength, and superior aspect ratio. However, the extraction of vegetable nanofibers remains challenging, particularly due to the high energy requirements and the intensive use of chemical agents.

This study presents the extraction of cellulosic nanofibers from coconut fiber using a low-energy process and an alkaline sodium bicarbonate solution. The effects of mechanical agitation and ultrasonic treatment in an alkaline medium were investigated and compared. The results indicate that both processes led to a reduction in the original fiber mass, confirming the production of nanofibers. Furthermore, mechanical agitation achieved a 30% higher yield compared to ultrasonic treatment.

INTRODUCTION

The global population has experienced significant growth, and it is estimated that it will reach approximately 10 billion by 2050 (Maja & Ayano, 2021; Leridon, 2020). Consequently, the demand for resources is expected to increase accordingly, as the production of goods is directly linked to population needs and consumption patterns. Modern society relies heavily on the extraction of both renewable and non-renewable natural resources to promote comfort and well-being through industrialization (Amri, 2019; Kedir & Hall, 2021). However, most raw materials used in the manufacture of consumer goods are still derived from non-renewable sources, such as minerals and metals.

In recent years, society has shown a growing awareness regarding the exploitation of natural resources, reflecting increased concern for environmental preservation. In this context, both industrial and governmental sectors have sought solutions to ensure the continuity of production systems while minimizing environmental impacts. Research and development efforts have advanced in this direction, focusing on the creation of new materials that can be integrated into industrial applications, thereby maintaining production efficiency and meeting the demand for raw materials (Adebayo et al., 2022; Ali, 2021). The scientific community has devoted considerable effort to developing materials with improved properties that are also environmentally sustainable (Karimah et al., 2021).

Plant-based resources represent an abundant and versatile source of raw materials for various technological applications, including textiles, pulp and paper, and coatings. In particular, plant biomass offers significant potential for the development of innovative materials and technologies, such as nanostructured materials (Munteanu & Vasile, 2019; Sameen et al., 2022).

One of the main challenges in this field is the development of advanced materials that meet industrial performance requirements while reducing environmental impact. In this context, composite materials have gained considerable attention. These materials are formed by combining two or more constituents, resulting in enhanced properties compared to their individual components (Fornari Junior, 2017; Hsissou et al., 2021).

Plant fibers are important resources that can be exploited for the production of composite materials. Their production is clean and low-cost, in addition to presenting numerous interesting chemical and physical properties, and can be applied to new materials with advanced technology (MARTINS & SANCHES, 2019; BUI et al., 2020; HIDALGO-SALAZAR et al., 2020). The use of plant fibers to form composites is a promising way to meet the industrial demand for materials and at the same time align due care with the environment.

Research into new technologies using plant fibers has achieved the science of nano materials. Cellulose nano fibers, for exemplo, offer exceptional mechanical properties, mainly due to their structure in gauge dimensions (ARUN et al., 2022; MOHAMADI et al., 2022). Cellulosic nano fibers have different properties from macro fibers, due to a significant

number of characteristics. They have, for example, an aspect ratio with the diameter of the fiber reduced in relation to its length. This modification reduces the morphological imperfections of its surface, reducing the chance of defects. The condition of the nano fiber is also changed, increasing its polarity and angular orientation. Dimensions and chemical composition are relevant factors, which will significantly influence the final properties of these structures or respective composites (OGUNBIYI et al., 2022; FORNARI JUNIOR, 2017).

Studies for technological applications of nano fibers are evaluating cell alignment (cytoskeleton remodeling), using topographic standardization for better cell alignment. Nano celluloses, in addition, have a great water retention capacity in addition to being biocompatible (LI et al., 2021). Nano cellulose is being studied for applications in high-performance materials, with the purpose of storing chemical energy in batteries. The nanometric structure serves as a precursor or guide for the orientation of specific morphologies, and involves a delicate thermodynamic and kinetic balance in the construction of these structures (LAMM et al., 2021). Nano cellulose fiber in a proportion of 3% by weight was added with carboxy methyl cellulose (CMC) and applied to the paper surface. The presence of CMC reduced the contact between the cellulose nanofibers and allowed a thicker and more homogeneous coating. Barrier properties were significantly improved with this technique. This was attributed to the homogeneous structure that was formed and the hydrophilic characteristics of nano cellulose (MAZHARI MOUSAVI, 2017). For application in paper packaging, with environmental concerns, the nano cellulose coating showed good properties especially against oxygen and fats, in addition to better mechanical resistance (KUMAR et al., 2016).

Studies for technological applications of nano e micro fibers are evaluating cell alignment (cytoskeleton remodeling), using topographic standardization for better cell alignment. Nano e micro celluloses, in addition, have a great water retention capacity in addition to being biocompatible (LI, 2021). Nano cellulose is being studied for applications in high-performance materials, with the purpose of storing chemical energy in batteries. The nanometric structure serves as a precursor or guide for the orientation of specific morphologies, and involves a delicate thermodynamic and kinetic balance in the construction of these structures (LAMM et al., 2021). Nano cellulose fiber in a proportion of 3% by weight was added with carboxy methyl cellulose (CMC) and applied to the paper surface. The presence of CMC reduced the contact between the cellulose nanofibers and allowed a thicker and more homogeneous coating. Barrier properties were significantly improved with this technique. This was attributed to the homogeneous structure that was formed and the hydrophilic characteristics of nano cellulose (MAZHARI MOUSAVI et al., 2017). For application in paper packaging, with environmental concerns, the nano cellulose coating showed good properties especially against oxygen and fats, in addition to better mechanical resistance (KUMAR et al., 2016).

The production and obtaining of cellulose nanofibers has been a subject of research interest for several researchers. However, obtaining nanofibers requires many efforts beyond the separation of nanoparticles in generally acidic or basic solutions and the filtration of the fibers. The process studied in this article involved opening coconut cellulose fibers to obtain cellulose microparticles, obtained using physical and chemical action and separated by decantation (DUFRESNE, 2013)

The coconut fruit is made up of three main parts or layers, the outermost layer being called exocarp, the middle layer mesocarp and the inner layer endocarp (CHUANHAO et al., 2020; DONG et al., 2023).

The fruit mesocarp is made up of the dry and wet fractions. The majority of the mesocarp by mass is made up of the wet fraction. This part represents 91% by weight of the fruit and is composed of water and dissolved salts. The dry fraction constitutes approximately 9% by weight of the fruit in natura, and is formed by cellulosic fibers, consisting of two distinct parts, called hard fiber and soft or sponged fiber (NGUYEN et al., 2016; LU et al., 2020; YANG et al., 2021). Figure 1 shows the parts of the coconut fruit and the mass percentage of the mesocarp.

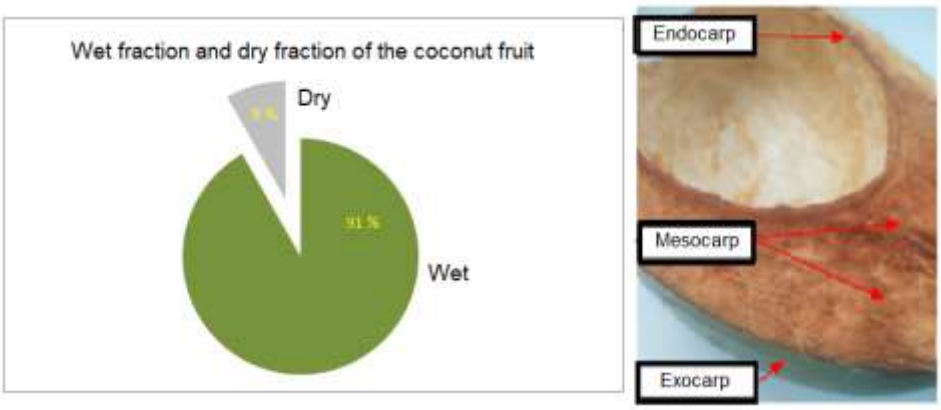

Figure 1- Percentage of constituents of the mesocarp and parts of the coconut fruit.

In the mesocarp, the hard fibers constitute the reinforcing component, providing structural support and contributing to the mechanical strength of the fruit. In contrast, the soft fibers, in combination with the hard fraction, play a key role in impact absorption. This softer portion is capable of dissipating mechanical energy due to its more elastic behavior. Additionally, it helps maintain a uniform distribution of the fibers while binding them together, thereby preserving the integrity of the mesocarp structure. The combined action of these two components ensures the protection of the fruit, particularly when it is subjected to impacts, such as falling to the ground (Ito et al., 2021). Figure 2 illustrates the mesocarp of the coconut fruit, highlighting the hard and soft (spongy) fiber regions.

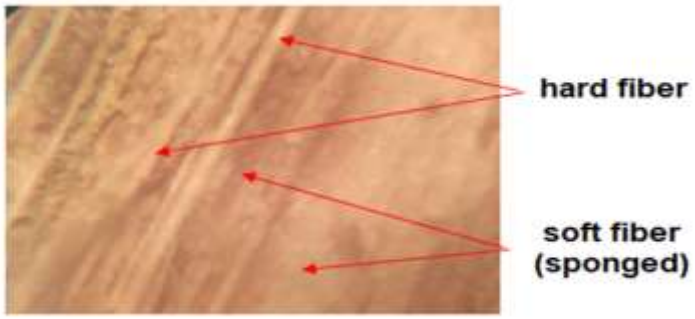

Figure 2- Photo of the coconut fruit mesocarp at 20x magnification, highlighting the hard and soft fiber.

The production of cellulosic microfibers is a complex process that requires specific treatments to achieve fibers at this scale. Cellulose nanofibers are fundamental structural elements that form micro- and macro-scale fibrous assemblies. These nanostructures consist of both crystalline and amorphous regions and are arranged in helical configurations, aggregating into bundles stabilized by lignin. Hemicellulose, a low-molecular-weight polysaccharide, is distributed between cellulose nanofibrils, preventing their aggregation and maintaining an adequate spacing between them.

Macrofibers are therefore composed of hierarchical assemblies of nano- and micro-scale fibrillar structures. Consequently, the deconstruction of these fibers requires overcoming the strong intermolecular interactions and organized architecture of the fibrous network (Fornari Junior, 2017).

This study investigates the effects of an alkaline sodium bicarbonate solution combined with ultrasonic and mechanical agitation on coconut fibers for microfiber production. The applied treatments altered the fiber structure and promoted the disintegration of the fibrous network, resulting in micrometric-scale structures. Different processing times were evaluated, with cycles of agitation and rest. All experiments were conducted in triplicate, and the results showed an average mass reduction of approximately 7% under the tested conditions.

METHODOLOGY

2.1 Fiber Samples

Coconut fibers obtained by mechanical processing from healthy fruits were dried in the shade and manually selected to ensure uniformity in terms of appearance and diameter. The fibers were manually cut into lengths of approximately 30 mm. The fibers were stored in an oven with air circulation at a temperature of 120 ± 5 ºC for 60 minutes. After cooling in a desiccator, the fibers were weighed in the amount of 5 g, using an analytical balance with five decimal places, from the brand Bioprecisa, model FA2104N.

2.2 Characterization

The fibers were characterized by scanning electron microscopy, using FEI Company equipment, model Quanta 250, and optical microscopy using a Tecnival magnifying glass. Infrared analyzes were carried out using Perkin Elmer model Spectrum 400 equipment.

2.3 Process

The processing of the fibers occurred with ultrasound or mechanical agitation in an aqueous solution of sodium bicarbonate at 8% w/w. It was carried out at regular intervals consisting of 60 minutes of rest in solution and 30 minutes of agitation. Each rest-agitation sequence constituted a period of fiber treatment, being named A (1 period), B (2 periods) ... up to E (5 periods), as illustrated in figure 3. The fibers after each period were washed in distilled water three times with a volume of 500 ml for each wash and separated from the solution using a paper filter. The fibers after treatment were dried in an oven at a temperature of 120 ± 5 ºC for 60 minutes and evaluated by gravimetry, according to the conditions already reported. For ultrasonic agitation, the Unique model Ultra Cleaner 1400 device was used and for mechanical agitation, the Servitech model CT 054 device was used. The mechanical agitation speed was 300 rpm.

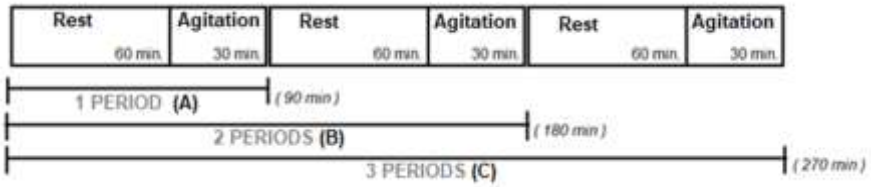

Figure 3: Fiber processing mode depending on resting and agitation times.

RESULTS AND DISCUSSION

The results of the ultrasonic treatment applied to the coconut fibers are presented in Table 1. All experiments were conducted in triplicate (Processes 1, 2, and 3), and only slight variations were observed among the replicates. These differences are attributed to the filtration, washing, and drying steps, which introduce some variability and make strict process control challenging.

After five treatment cycles, the final mass of the fibers was approximately 93% of the initial mass, indicating an average mass loss of about 7% over the course of the process.

Table 1- Gravimetric results of coconut fiber subjected to ultrasound agitation.

Periods	PROCESS 1	PROCESS 2	PROCESS 3	Weight reduction (%)		
	Fiber weight (g)			1	2	3
A	4,87	4,87	4,82	97,4	97,4	96,4
B	4,77	4,82	4,80	95,4	96,4	96,0
C	4,69	4,79	4,77	93,8	95,8	95,4
D	4,69	4,76	4,73	93,8	95,2	95,2
E	4,69	4,67	4,63	**93,8**	**93,4**	**92,6**

Natural coconut fiber is covered by a characteristic surface layer composed of waxes, fatty acids, and pectin, forming a distinct coating on the fiber. In this region, globular particles or protrusions are also observed, contributing to the overall surface morphology associated with this fatty layer. Figure 4 illustrates the outer surface of the coconut fiber (Fornari Junior, 2017; Ahmad et al., 2022).

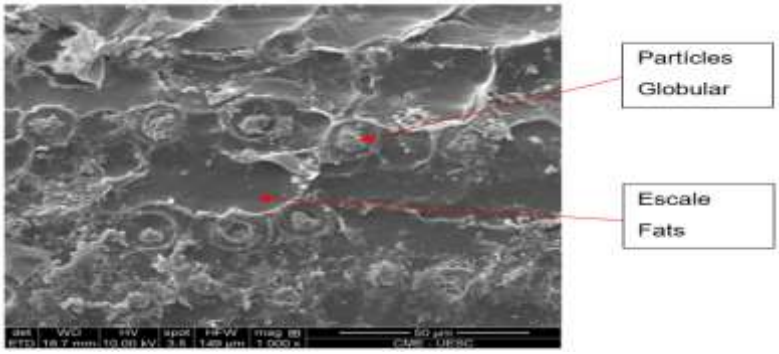

Figure 4-Image of coconut fiber with globular particles and greasy scale.

Figure 5 presents the mass evolution of the fibers as a function of time during the ultrasonic treatment. Processes 2 and 3 exhibited similar behavior in terms of mass loss, whereas Process 1 showed a more pronounced reduction up to the third cycle, followed by a more stable trend until the end of the experiment. This variation is likely associated with intrinsic differences in fiber characteristics, such as composition, size, and age, which may influence their susceptibility to modification during the treatment.

All processes showed a significant decrease in mass, reaching an approximate reduction of 7% relative to the initial weight. This reduction is primarily attributed to the removal of the outer layer of the coconut fiber, resulting from the combined action of the alkaline solution and ultrasonic treatment. Initially, the surface layer is removed, followed by partial lignin extraction and the progressive disintegration of fiber bundles.

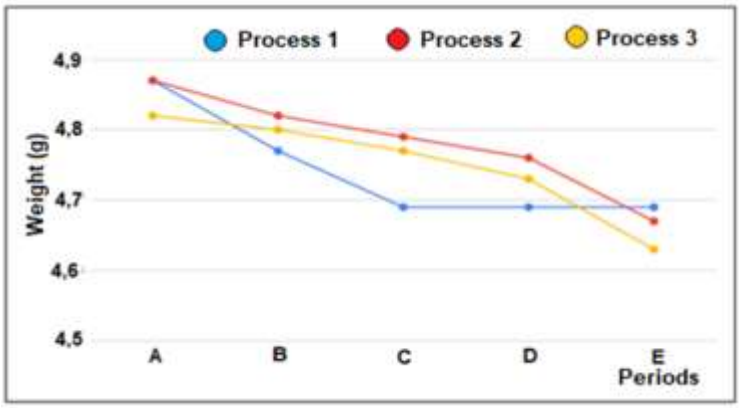

Figure 5-Graph of the weight of the fiber per period in sodium bicarbonate solution and subjected to ultrasound agitation.

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All processes showed a significant gravimetric reduction, reaching approximately 7% of the initial mass. This reduction is primarily attributed to the removal of the outer layer of the coconut fiber, resulting from the combined action of the alkaline sodium bicarbonate solution and ultrasonic agitation. Initially, the surface layer is removed, followed by partial lignin extraction and the progressive disintegration of the fiber bundles.

The fibrous bundles that constitute the cellulosic structure are protected by a lignin coating, which acts as a physical barrier while also binding the structural fibers together. However, exposure to the alkaline bicarbonate solution promotes the partial dissolution of lignin, leading to its transfer into the aqueous medium. This process disrupts the fibrous organization, exposing the bundles to the surrounding environment and facilitating their individualization.

Figure 6 shows the alkaline solution obtained after each treatment cycle. The color intensity of the solution increases progressively with the number of cycles, indicating a higher concentration of dissolved constituents. This coloration is associated with the removal of surface components, such as waxes and fatty substances, as well as lignin.

The partial dissolution of lignin weakens the rigid structure formed by cellulose and hemicellulose, promoting the breakdown of the fibrous network and enabling the formation of micro- and nanoscale fibrillar structures. In this context, ultrasonic agitation plays a crucial role, significantly enhancing fiber deconstruction and contributing to the reduction in mass and the generation of cellulosic nanostructures

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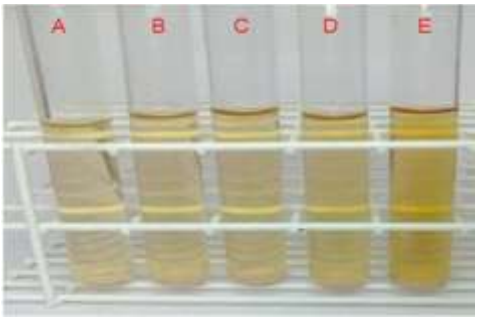

Figure 6-Aqueous sodium bicarbonate solution after ultrasound processing of coconut fibers in each period.

After ultrasonic treatment, some fibers underwent significant structural changes, becoming micronized into small particles that precipitated in the solution. These precipitated particles, obtained after period E, were analyzed using scanning electron microscopy (SEM). This stage consisted of five cycles of 60 minutes of rest followed by 30 minutes of ultrasonic agitation, totaling 450 minutes of processing time.

The results revealed substantial modification of the original coconut fibers, including the progressive separation of fibrous nanobundles. The structure exhibited partial individualization into finer fibrillar elements, with diameters on the nanometric scale (approximately 3 nm), indicating that

the applied conditions were sufficient to initiate the deconstruction of the fibrous network.

The partial removal of lignin, which is responsible for maintaining the compact structure of the fibers, facilitated this separation process. However, complete individualization was not achieved at this stage. The fibers remained interconnected at their base, forming a brush-like morphology, characterized by individualized fibrils at the top and bonded structures at the bottom.

Figure 7 shows SEM images of coconut fibers after ultrasonic treatment in an alkaline solution at a magnification of 500X.

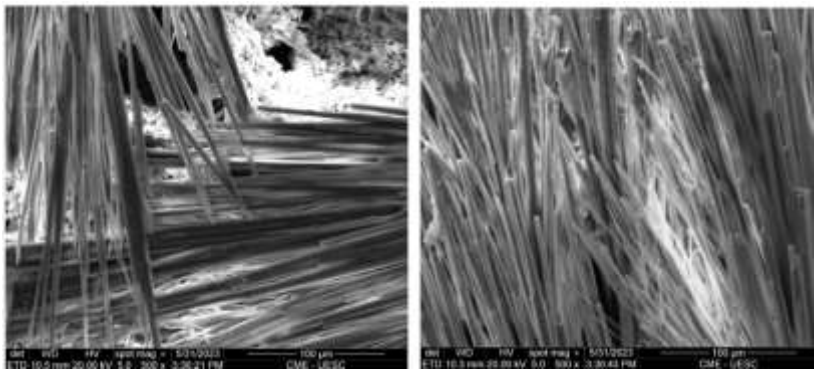

Figure 7- Scanning electron microscopy of coconut fiber subjected to ultrasound agitation in alkaline solution

The process employing mechanical agitation was also evaluated using a gravimetric approach. The results, presented in Table 2, were obtained from triplicate experiments. Across all processing periods, a consistent reduction in fiber mass was observed. At the end of the treatment, the fibers exhibited an average mass loss of approximately 10% under all evaluated conditions.

This reduction occurred in two main stages. Initially, the outer (waxy) layer of the coconut fiber was removed, followed by partial lignin dissolution and the formation of nanofibrillar structures. In comparison with the ultrasonic treatment, mechanical agitation proved to be more effective, resulting in a higher mass reduction (approximately 7–10%). This indicates a greater degree of lignin solubilization and, consequently, a higher yield of fibrous particles.

Mechanical agitation enhances the interaction between the fibers and the alkaline solution by promoting shear forces and continuous mixing. This dynamic contact facilitates the constant renewal of the reactive medium at the fiber surface. As a result, a synergistic effect between chemical and mechanical actions is achieved, leading to a more efficient fiber deconstruction process, with an improvement of approximately 40% compared to ultrasonic treatment.

Table 2-Gravimetric results of coconut fiber subjected to mechanical agitation.

Periods	PROCESS 1	PROCESS 2	PROCESS 3	Weight reduction (%)		
	Weight fibers (g)			1	2	3
A	4,80	4,75	4,73	96,0	95,0	94,6
B	4,74	4,69	4,69	94,8	93,8	93,8
C	4,72	4,66	4,62	94,4	93,2	92,4
D	4,68	4,56	4,56	93,6	91,2	91,2
E	4,52	4,50	4,48	**90,4**	**90,0**	**89,6**

Fibers subjected to mechanical agitation in a sodium bicarbonate solution exhibited a progressive decrease in mass as a function of treatment time. By the end of the process, all tests converged to similar final values. Process 1 showed a lower mass loss up to the penultimate stage compared to Processes 2 and 3. This difference can be attributed to variations in the intrinsic properties of the fibers, such as chemical composition, size, and age, which likely influenced the rate of lignin removal and the separation of fibrous bundles.

Overall, the results converged to an approximate total mass loss of 10%. Figure 8 presents the mass variation observed during the mechanical agitation process.

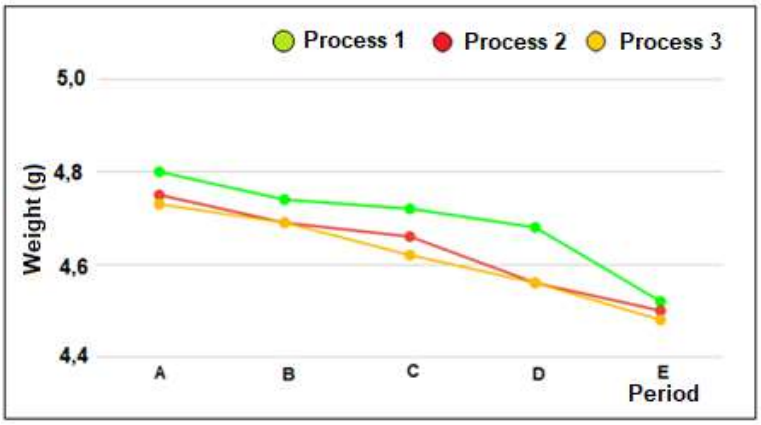

Figure 8-Graph of fiber weight per period in sodium bicarbonate solution and subjected to mechanical agitation.

Tables 1 and 2 summarize the fiber mass loss observed after five processing periods. During the first period, approximately 2.5% of the fiber mass was lost under ultrasonic agitation, whereas mechanical agitation resulted in a 5% reduction. These results indicate that mechanical agitation is more effective than ultrasonic treatment. The observed mass loss during the first period primarily corresponds to the removal of the fiber's outer layer, which consists of waxes, fatty substances, and pectin.

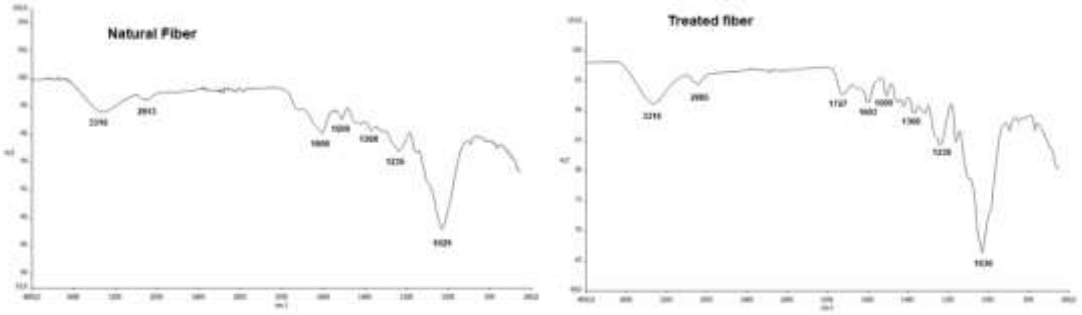

Figure 9-Attenuated infrared total reflectance spectra of natural and treated coconut fibers

Figure 9 presents the attenuated total reflectance Fourier-transform infrared (FTIR-ATR) spectra of natural and ultrasound-treated coconut fibers in 8% w/w alkaline sodium bicarbonate solution. The analysis recorded characteristic absorption bands of the fiber components and highlighted differences induced by the treatment.

3316 cm⁻¹ – The absorption band at 3316 cm⁻¹, associated with O–H groups, was more pronounced in the treated fibers, indicating an increased exposure of hydroxyl groups within the cellulose chains. Alkaline treatment effectively removes lignin and the fatty coating, revealing the cellulosic structures in their pure form, which are rich in O–H groups (Mittal & Chaudhary, 2019).

2885–2913 cm⁻¹ – Absorption peaks at 2885 cm⁻¹ (treated fibers) and 2913 cm⁻¹ (natural fibers) correspond to the C–H stretching of aliphatic molecules. The shift in peak position indicates that alkaline treatment alters the vibrational environment of these groups (Sathish, 2018; Sistons et al., 2022).

1510–1560 / 1730 cm⁻¹ – Carbonyl groups (R_2–C=O), abundant in cellulose and hemicellulose, were detected in these regions. Treated samples showed stronger bands at 1510 cm⁻¹ and 1727 cm⁻¹, reflecting the increased presence of cellulose and hemicellulose after partial lignin removal. Natural fibers exhibited a band at 1509 cm⁻¹. These results align with previous pyrolysis studies, where organic groups (C=O, C–O–C) are mainly released from hemicellulose and cellulose, while lignin contributes more CO and CO_2.

1421 cm⁻¹ – This band, characteristic of the C–H vibrational mode in the aromatic lignin ring, was preserved in both natural and treated fibers, indicating that sodium bicarbonate treatment maintains the aromatic structure of lignin (Araújo et al., 2023).

1368 cm⁻¹ – Assigned to C–H bonds in cellulose and hemicellulose, this peak was observed in both treated and natural fibers. In contrast, previous studies report its absence in coconut fibers treated with 5% NaOH for 24 hours, due to hemicellulose removal (Freitas et al., 2022).

1030 / 1238 cm⁻¹ – Peaks at 1030 cm⁻¹ and 1238 cm⁻¹, associated with C–O groups of cellulose and lignin, were present in both treated and untreated fibers, indicating that the treatment did not significantly alter these bonding environments (Vidal et al., 2018; Gama et al., 2021).

Figure 10 shows the optical analysis of coconut fibers during the ultrasound-assisted alkaline treatment. In the natural fiber (Figure 10 – Natural Fiber), the external layers appear uneven and partially cover the fiber. After the first period of treatment (Figure 10 – Period A), the fiber surface

becomes smoother and more uniform, with the outer layer largely removed, revealing the underlying fibrous bundles.

By the second period (Figure 10 – Period B), partial rupture of the fibrous bundles is observed. Ultrasound agitation in alkaline solution disrupts the bundle packing, facilitating partial lignin dissolution and loosening the fibrous network. From period C through period E, the fibers continue to exhibit a smooth and homogeneous surface, interspersed with partial ruptures of the bundles, reflecting progressive deconstruction of the fibrous structure. Figures 10 – Periods C, D, and E illustrate this morphological evolution.

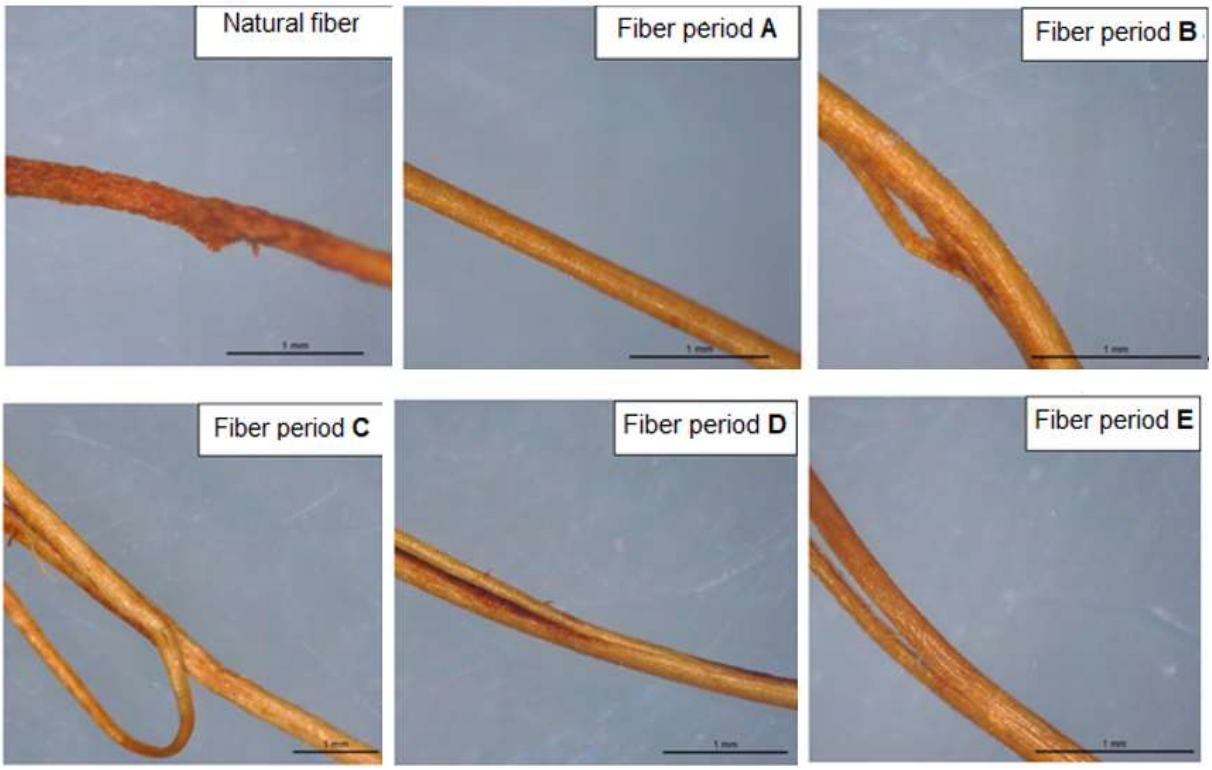

Figure 10-Optical micrograph of natural coconut fiber and after treated with ultrasound agitation at different periods (A, B, C, D and E).

CONCLUSION

The production of cellulose microfibers was successfully achieved through a combination of chemical and physical treatments, demonstrating that macrofibers can be deconstructed into smaller fibrous structures with relatively low cost and minimal environmental impact. Coconut fibers were treated with an 8% w/w alkaline sodium bicarbonate solution at room temperature, using either ultrasonic or mechanical agitation. The processes were identical in terms of time, solution, and temperature, differing only in the mode of agitation, and all experiments were performed in triplicate.

In both treatments, the fibers exhibited progressive mass loss from the first period onward. Ultrasound-treated fibers showed a final weight reduction of approximately 7% after five periods, while mechanically agitated fibers exhibited a 10% reduction. Mechanical agitation was more effective, likely due to enhanced chemical action facilitated by solution renewal and shear forces in a turbulent regime.

FTIR-ATR analysis revealed that sodium bicarbonate treatment altered the chemical characteristics of the fibers, with increased peak intensity observed at 3316, 1730, and 1510 cm^{-1}, corresponding to hydroxyl and carbonyl functional groups. Scanning electron microscopy confirmed that the fibrous structure partially opened and separated into nanofibrils after 450 minutes of processing. Optical microscopy demonstrated the gradual removal of the outer layer and progressive modification of fiber morphology during each period. Notably, after the second period, partial rupture of the fibrous structure indicated the combined effect of ultrasonic agitation and alkaline treatment in facilitating fiber deconstruction.

Overall, the study confirms that coconut fibers can be efficiently converted into micro- and nanoscale fibrous structures through environmentally friendly, low-energy processes, with mechanical agitation providing the highest efficiency in mass reduction and fiber deconstruction.

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