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Mechanical processing in alkaline solution of coconut fiber for formation of micro fibrils

Processamento mecânico em solução alcalina da fibra para formação de micro fibrilas

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Abstract
Vegetable fibers are abundant and ecologically aligned materials with the environment, both in production and final disposal. They are promising materials for use in engineering applications, being able to replace conventional materials and materials from non-renewable sources, such as the formation of composites. For the formation of high-performance composite materials, the compatibility between their constituents must be as high as possible, in order to result in strong chemical interactions between them. Coconut vegetable fiber in its natural form has a typical morphology that, in addition to being irregular, is covered by a fatty layer, thus preventing possible contact between the cellulosic fiber structure and other materials. Many treatments and processes have been studied and presented to the scientific community, resulting in significant modifications for the formation of new materials. Treatment with sodium hydroxide revealed that it is possible to partially modify the external structure of the fiber and that it can also dissociate the fiber into a nanostructure by solubilizing lignin. This work was dedicated to evaluating the processing of coconut fiber in sodium hydroxide solution and ball mill, in order to modify the conditions of the vegetable fiber. The results revealed that, depending on the processing time, partial modification of the fiber occurs, with changes in color and formation of fibrils. Fibrils are branched extensions that can increase the conditions of contact with new materials.
Keywords: Cellulose. Coconut fiber. Alkaline treatment. Ball mill.

Resumo
As fibras vegetais são materiais abundantes e ecologicamente alinhadas com o meio ambiente, tanto na produção quanto no descarte final. São materiais promissores para o uso em aplicações de engenharia, podendo substituir os materiais convencionais e de fontes não renováveis, como por exemplo, a formação de compósitos. Para a formação de materiais compósitos de elevado desempenho é necessário que a compatibilidade entre os seus constituintes seja a máxima possível, de maneira a resultar em fortes interações químicas entre ambos. A fibra vegetal de coco na sua forma natural, apresenta uma morfologia típica que além de irregular é coberta por uma camada gordurosa, impedindo assim um possível contato entre a estrutura da fibra celulósica com outros

materiais. Muitos tratamentos e processos foram estudados e apresentados na comunidade científica, resultando em modificações significativas para a formação de novos materiais. O tratamento com hidróxido de sódio, revelou que é possível a modificação parcial da estrutura externa da fibra e que também pode dissociar a fibra em nano estrutura pela solubilização da lignina. Esse trabalho, se dedicou a avaliar o processamento da fibra de coco em solução de hidróxido de sódio e moinho de bolas, de forma a modificar as condições da fibra vegetal. Os resultados revelaram que, a depender o tempo de processamento, ocorre a modificação parcial da fibra, com a modificação na cor e formação de fibrilas. As fibrilas são prolongamentos ramificados que, podem aumentar as condições de contato com novos matérias.
Palavras-chave: Celulose. Fibra de coco. Tratamento. Alcalino. Moinho de Bolas.

1. Introduction

In a production and consumption system, where the population grows by 1.1% per year (2015-2020), after having reached 2.3% at the end of the 1960s, the productive sector needs to be attentive to find solutions to manufacturing of products and consequently the respective raw materials, to maintain the situation of the production and consumption system that we have adopted. (GU *et al.*, 2021)

In this context, sources of non-renewable raw materials are on the verge of scarcity, due to the exhaustion of the extraction of deposits. On the other hand, renewable materials are a unique opportunity and alternative, in order to enable the system to remain in the current condition in which it has been established to this day.

Vegetable fibers, by the very nature of being included in the permanent carbon cycle, are renewable and have interesting physical and chemical properties from an engineering perspective. Vegetable fibers are materials composed mainly of lignin and cellulose in addition to significant amounts of hemicellulose. They are abundant, have natural and pollution-free production, are relatively cheap, biodegradable and contribute to the renewal of the life cycle of the entire ecosystem (THAKUR *et al.*, 2021)

The formation of composite materials requires a harmonious and strong interaction between their constituents, in order to guarantee maximum union and the sum of their mutual properties. In the search for the application of polymer composites with plant fibers, many works have been dedicated to increasing the interaction of the fiber with polymer matrices, aiming for higher performance composites (FORNARI JUNIOR, 2017; JAGADEESH *et al.*, 2021; SATHISH *et al.*, 2021).

An interesting alternative in the formation of composites with higher mechanical properties is the use of cellulosic nanostructures, originating from natural macro fiber. Nano fibers are aligned and more perfectly packaged structures, presenting fewer defects on their surface and greater properties, mainly mechanical (ANUSIYA and JAIGANESH, 2022; FAHMA *et al.*, 2021). Fibers in their micro and nanometric dimensions demonstrate properties that are often superior when compared to macro fibers. In the nanometric dimension, the plant fiber is organized more perfectly, with a more homogeneous morphology and a reduction in the number of surface reliefs and depressions. Furthermore, the aspect ratio is an important factor in improving the properties of composites made with fibers (VALLITTU, 2015).

Plant fibers are made up of countless bundles of micro fibers which in turn are also made up of countless bundles of nano fibers. The bundles are packed by lignin, a sealant that ensures the compaction and fixation of the physical structures of the cellulose. Therefore, the entanglement of the nano beams that will form the micro beams and finally form the macro fibers needs to be deconstructed, to obtain the individualized nano structures and consequently, separated from the initial structure. This operation of obtaining nano fibers requires, in addition to time and energy, a specific technique. (FORNARI JUNIOR, 2017; LEÃO *et al.*, 2012)

Fibrils are specific structures that can be created or generated from the original cellulose fibers. They consist of derivations of the fiber, which are formed by partial detachment from the original structure, but which are attached at one end, in order to create branches from the

macroscopic fiber. The formation of fibrils in the fiber is a factor that can probably favor the construction of a composite with better properties. The presence of fibrils provides a greater condition of interaction with the polymer matrix, due to the increase in the number of micro fibers, the possibility of increasing the number of fiber-matrix contacts and the greater number of directions. Furthermore, the aspect ratio that the fibrils have contributes to increasing the mechanical resistance of the composite. (COUGHLIN, 2021; BANVILLET *et al.*, 2021; WATHÉN, 2006)

This work was dedicated to evaluating a process of fibril formation in natural coconut fiber from green coconut fiber, discarded after removing the water contained in the fruit. The process was designed to consume a low amount of energy and generate the least amount of waste. The work brought together the mechanical action of the ball mill in synergy with the chemical action of the aqueous sodium hydroxide solution.

The fibers were processed for ninety minutes, alternating between rest and agitation. The processes were repeated and the fibers were evaluated in relation to the physical change in the fibrous structure. The results revealed that at all stages of the process there was a positive action of reducing and modifying the fibers, consequently forming micro-fibrous structures with the increase in the number of processes. With the aid of image analysis, the fibers showed microfiber formation from the third processing onwards. Infrared characterization demonstrated changes in some bands depending on the number of processes the fiber was subjected to. UV-Visible analysis revealed a quantitative increase in lignin proportional to the number of processing.

2. Methodology

2.1 Coconut Fibers

The fibers were extracted from healthy, green coconuts, from the UNA region, Bahia. The coconut fruits were washed in clean water, cut to remove the water contained in the fruit and the hard fibers were separated from the soft fibers contained in the fruit's mesocarp, using a mechanical defibrillator. Figure 1 shows the parts of the coconut fruit and the fibers in the mesocarp. The separated fibers were stored in the shade for several days for dehydration. Before the experiment, the fibers were dried in an oven with air circulation at 130 ± 5 ºC for 60 minutes. Five grams of dry fibers were weighed on a digital scale with four decimal places, Bioprecisa brand, model FA 2104 N.

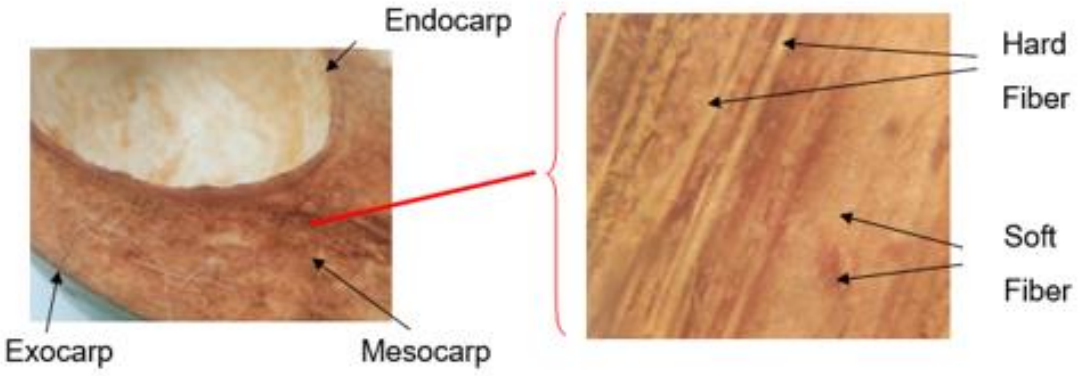


Figure 1 – Parts of the coconut fruit and the mesocarp with hard and soft fibers

2.2 Processing

The fibers were placed in the mill jar, together with 200 ml of 0.5 molar NaOH aqueous solution. The fibers remained in the solution for 60 minutes and were then processed in a ball mill for 30 minutes. For processing, 35 alumina spheres with a diameter of 22 mm, a 5-liter jar and a rotation speed of 90 rpm were used. Processing called A consisted of subjecting the fiber to one process (resting/grinding), processing B to two processes equal to process A... until processing F,

which corresponds to the repetition of five processes A. The fibers were separated of the solution through filtration with filter paper, washed in distilled water for four procedures with 500 ml each. After filtering, the fibers were dried in an oven at 130 ± 5 ºC for 60 minutes. NEON brand sodium hydroxide P.A. and distilled water were used. Figure 2 shows the open ball mill with the respective spheres, coconut fiber and alkaline solution before starting processing.

Figure 2 - Mill jar at rest, with spheres, NaOH solution and coconut fibers

2.3 Description

The characterization of the NaOH solution was carried out for each type of processing. The solution was analyzed by Visible Ultraviolet (UV-Vis) electronic spectroscopy on a Nova Instrument Model Nova 1600 UV Spectrophotometer device. Samples of the solution used in processing were diluted 1/100 in 0.5 M NaOH solution.

3. Results

The fiber is the result of rigid cellulose and hemicellulose structures, protected and compacted with the help of lignin. Lignin has the fundamental role of holding the fibrous bundles together and at the same time filling the spaces between them. The alkaline solution, in turn, is capable of solubilizing the lignin contained in the fiber, when both come into contact (WEARN *et al.*, 2020). The removal of lignin from the fiber unprotects the packaging of the rigid cellulose structures, allowing their individualization. This work processed coconut fibers with a resting and stirring procedure in a ball mill in an alkaline solution, and the result showed significant changes in relation to fiber disintegration and changes in appearance. The fibers showed a decrease in weight at the end of each processing, corresponding to a linear and relatively constant behavior between processes B to F, as shown in figure 4. However, in the first process (A), that is, with natural fibers, the weight loss was approximately 35% of the total weight of the fiber, much higher than the average of the corresponding values between processes B to F. This value is associated with the layer of wax, pectin and fat that surrounds the fiber and the residual parts of soft fiber that remained attached to the surface of the fiber, due to the separation process by mechanical defibrillation. Two factors occurred to reduce fiber weight in the first processing. The first is due to the chemical action of the solution, which has the ability to remove fatty and soluble compounds from the surface. The second factor that promoted partial degradation of the fiber is related to comminution. The first contacts between the fiber and the mill balls act on the external parts of the fiber that are bonded to the fiber, such as, for example, parts of the soft fraction of the mesocarp of the coconut fruit. The chemical action of the solution certainly has a positive effect on this modification, however, in this first stage of processing, the action due to mechanical shear is quantitatively more significant (SITOTAW *et*

al., 2022). Figure 3 shows the natural fibers partially bonded with the soft fiber, and the fiber processed once (Processing A).

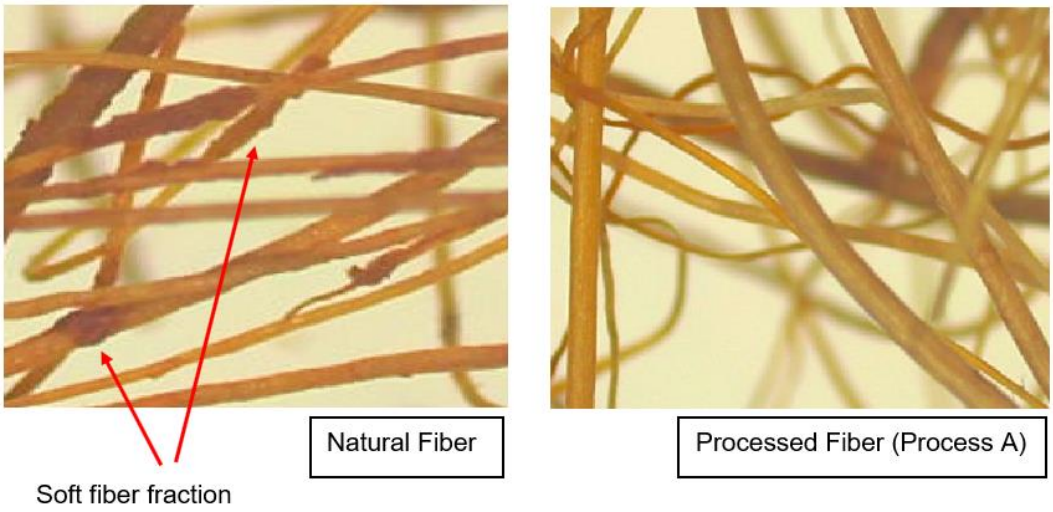


Figure 3 – Image of the natural fiber with the soft fraction and the fiber after process A

Between the second process in which the fiber was subjected and the last, the weight reduction occurred between approximately 5 to 9%. After removing the outer layer of the fiber, the removal occurs preferentially for the lignin contained in the fiber structure and the removal of the cellulosic nano beams (JANKER-OBERMEIER *et al.*, 2012).

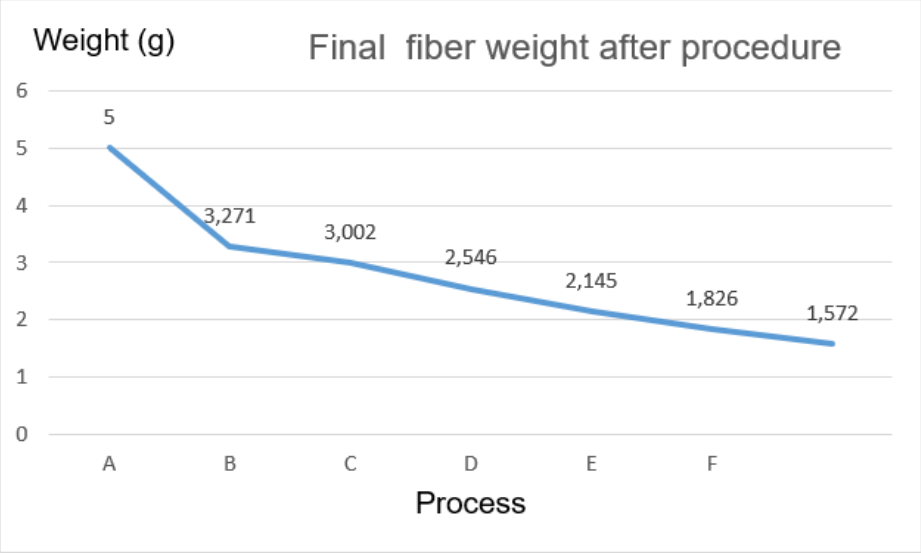


Figure 4 - Final weight of coconut fibers after undergoing procedures in alkaline solution and ball mill

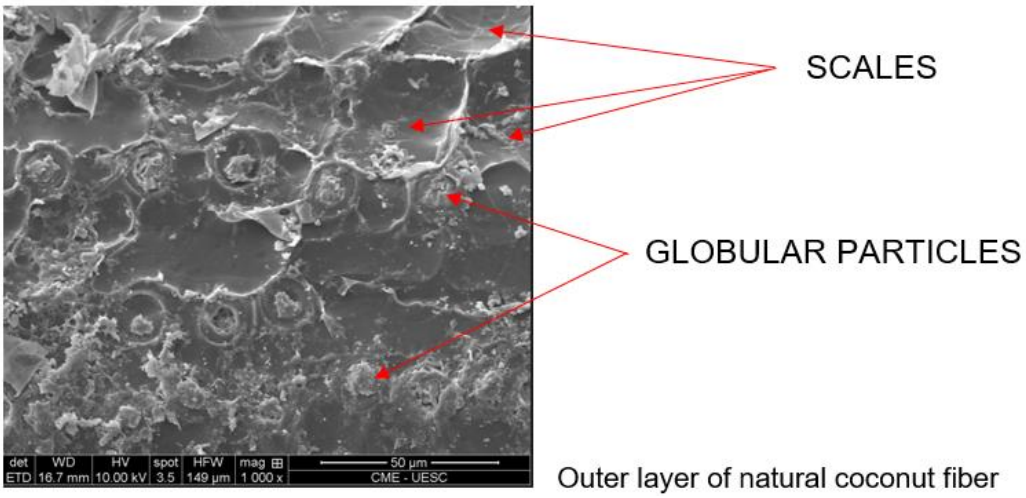
In the composition of natural fiber, lignin is closely linked to the set of fibers and presents some difficulty in coming into contact with the alkaline solution. However, the action of the ball mill is important in order to promote agitation/bending and force the fiber structure to come into contact with the solution. The action of the ball mill is to promote mechanical shearing of the fibers, forcing greater movement and facilitating contact with the solution. For this, physical contact of the fibers with the mill balls is necessary, however, this action is random and requires the most homogeneous distribution of fibers possible within the mill, in order to obtain greater efficiency of the shearing work through contact. between them. An irregular distribution of fibers and spheres

can promote agglomeration and segregation of fibers, making contact and mechanical action of the spheres in the mill difficult. The variation in relative weight loss that occurred between process types B to F was most likely a consequence of the irregular distribution of fibers within the ball mill (HERNÁNDEZ-VARELA *et al.*, 2021). Table 1 presents the weight values of coconut fibers subjected to the respective procedures.

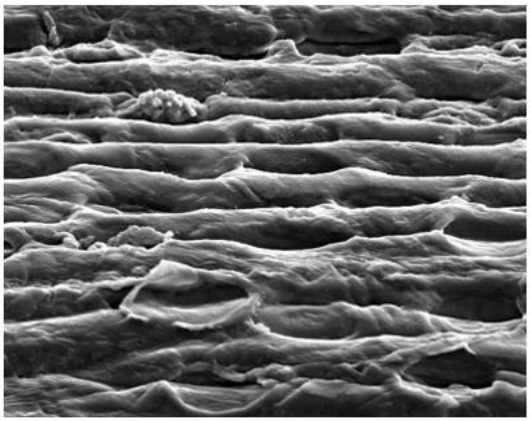
Table 1 – Weight values and percentage of fibers subjected to different procedures in alkaline solution

Processo	Peso inicial (g)	Peso final (g)	% em peso restante	% de peso perdido	% de perda relativa
A	5,000	3,271	65,42	34,58	34,58
B	5,000	3,002	60,04	39,96	5,38
C	5,000	2,546	50,92	49,08	9,12
D	5,000	2,145	42,90	57,10	8,02
E	5,000	1,826	36,52	63,48	6,38
F	5,000	1,572	31,44	68,56	5,08

When in contact with the solution, the extraction of the soluble fraction initially occurs in the external region of the fiber, rich in wax and pectin. Globular particles are also present in this region (FREITAS *et al.*, 2022). This region is the first to come into contact with the NaOH solution, and begin the dissolution of the soluble constituents. After solubilization of the layer covering the fiber, the cellulosic lignin structure is exposed. After this moment, the alkaline solution comes into contact with the cellulosic lignin structure and begins the solubilization of the lignin fraction and the solubilization of the hemicellulose, according to the literature. (JANKER-OBERMEIER 2012). Figure 5 shows the scanning electron microscopy image of the surface of the natural fiber and the surface of the fiber treated in NaOH solution for 5 hours at rest. The image of the fiber treated in NaOH solution shows the rigid structure of the fiber, composed of cellulose bundles and the grooves between these bundles. The voids correspond to the space that was filled by lignin, and which, due to natural protection, has the action of keeping the structures cohesive and distant from each other.



Outer layer of natural coconut fiber



Outer layer of coconut fiber after procedure in NaOH and partial removal of soluble substances

Figure 5 – Image of natural coconut fibers submitted in NaOH solution for 5 hours

As process times increase, the fibers decrease in diameter and at the same time partially fragment. The NaOH solution and the action of mechanical shearing by the ball mill chemically and physically degrade the fiber. Lignin is solubilized by the alkaline solution as the two come into contact. (MELRO *et al.*, 2020). The mechanical action of the mill acts on the fibers in two ways. Firstly, forced displacement or bending of the fiber structure occurs. This action helps in the movement and possible opening of the fibrous packaging, so that the alkaline solution can more easily come into contact with the internal structure of the fiber. The second action of the mill occurs by the total rupture of the fiber. When rupture occurs, two new areas of contact with the solution are generated, which correspond to the ends of the new fiber fragment. Figure 6 presents the schematization of the changes caused by the mechanical action of the ball mill.

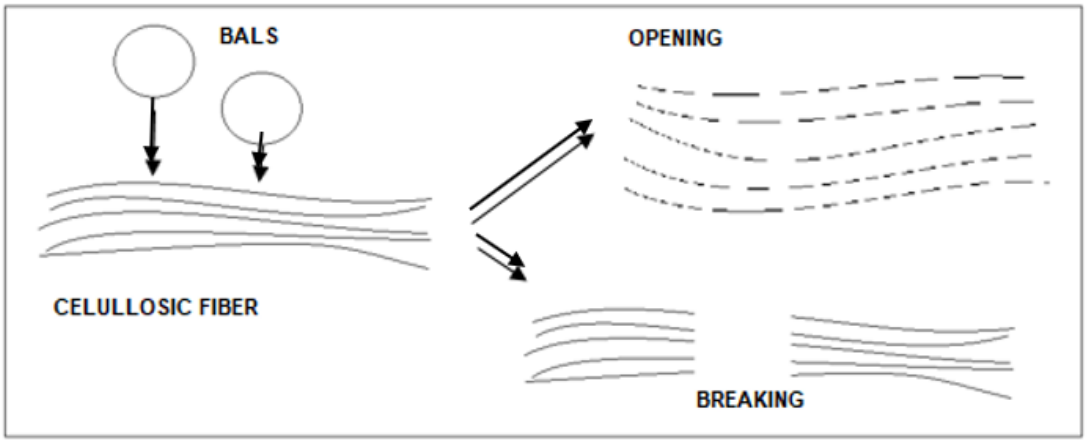


Figure 6 – Characterization of the sequence of changes in the cellulose fiber that suffers from the action of the spheres

The processed coconut fibers showed changes in appearance due to the increase in process time. From treatment A, the fibers decreased in color intensity, starting from brown to a light-yellow tone. In the shorter time processes (A and B) the changes are small, showing a small difference in light tone. From procedure C, the fibers appeared even lighter, due to the greater removal of lignin and consequently, the remaining structure being composed of more concentrated or isolated cellulose. Figure 7 shows the coconut fibers after undergoing procedures A to F.

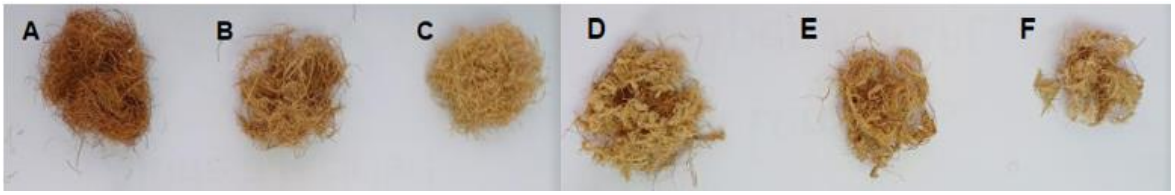


Figure 7 – Image of coconut fibers after undergoing the processes (resting / shaking)

The removal of lignin changes the condition of the cellulosic structure both physically and chemically. The absence of lignin allows the fiber to be exposed and at the same time more individualized. In chemical modification, the hydroxyls present in the cellulose chain are exposed. The fibrous structures will have a change in surface tension and the fiber will be partially altered, due to the fact that the hydroxyls are exposed to act and make bonds through hydrogen bonds.

In this exposed fiber condition, the sodium ion present in the solution may react with the hydroxyls of the fiber, if conditions are favorable, and promote chemical changes in the chain. The possibility of the hydroxyl hydrogen being replaced by sodium may occur, as shown in the reaction in figure 8 (SONG *et al.*, 2021; EGAL *et al.*, 2007).

These new conditions alter the energetic balance of the fibrous bundle and at the same time provide the opportunity for attractive forces to interact between the modified fibers.

Figure 8 – Reaction of cellulose and sodium hydroxide

The result of fiber processing produces a macroscopic agglomeration or clogging of the fibers, as shown in figure 9. After the third procedure (C) in NaOH solution, the smaller diameter fibers attract each other and with the removal of the aqueous solution, an agglomerated mass of fibers occurs. Figure 9 shows the fiber after six processes and with a partially agglomerated region. The enlarged image reveals that the fibers came together and compacted together, as a result of mutual attraction after removal from the solution.

Figure 9 – Images of agglomerated fibers after process F in alkaline solution and ball mill

The images of the fibers, natural and treated by processes A and B, are shown in figure 10. The fibers do not present significant changes for the process conditions to which they were subjected.

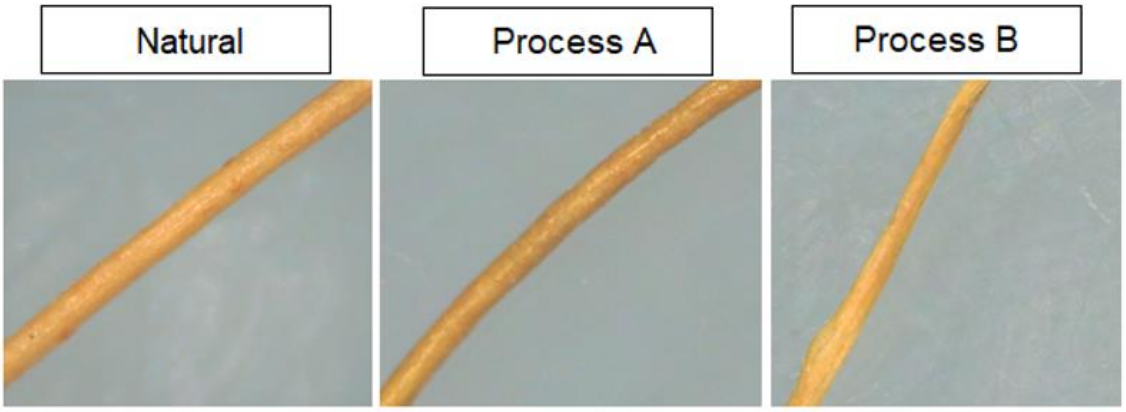


Figure 10 – Natural and processed coconut fiber (A and B)

From treatment C onwards, the number of fibers that presented fibrils was significant, corresponding to approximately 50% of the fibers. This treatment was able to initiate partial degradation of the cellulosic structure, forming a significant number of fibrils on the fiber surface. Fibrils constitute the partial separation of the fibrous structure and occurs at a specific point, in order to detach some fiber bundles and at the same time keep them originally connected to the main structure. The effect of the treatment appears in the superficial region of the fiber, as contact with the solution and mechanical shear initially occurs on the outside of the fiber. Figure 11 shows the fiber after process C and highlights the fibrils that formed on the surface.

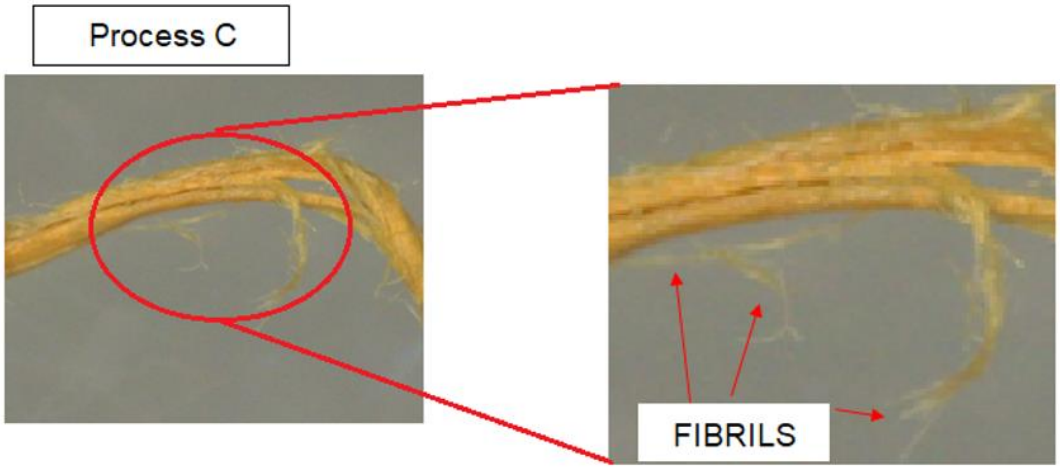


Figure 11 – Coconut fiber after undergoing process C, with initial formation of fibrils

The increase in the number of processing processes, which correspond to processes D, E and F, promoted a greater number of fibril populations in coconut fibers, as shown in figure 12. Processing is capable of destroying the fibrous structure and randomly fragmenting parts of the fiber. fiber. The chemical action of the solution is sufficient to interact with the lignin, causing its dissolution and deconstructing the original structure of the fiber (ACHANYA *et al.*, 2021; SIRVIÖ & HEISKANEN, 2020). The physical action through shear promotes the rupture of the fiber through the impact of the balls. The mechanical shock forces the fibrous bundle to move and partially rupture. Both chemical and physical energy lead the fiber to partial collapse, generating a population of fibrils from its surface.

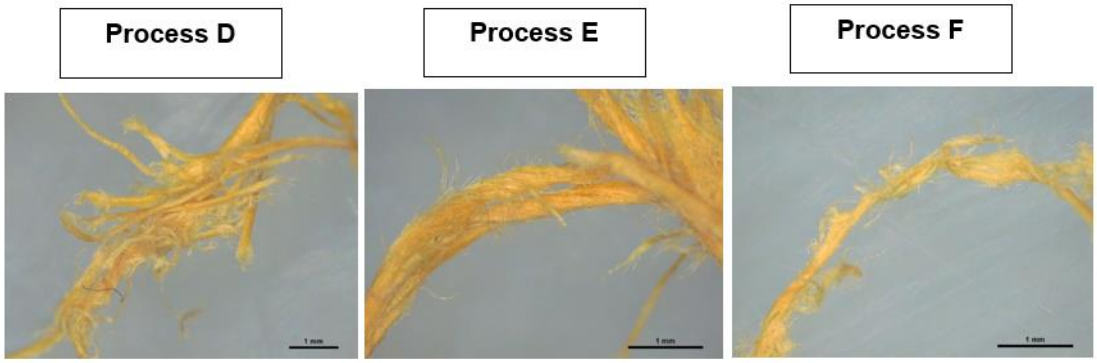


Figure 12 – Coconut fibers processed in D, E and F, with the respective fibrils

To characterize the lignin contained in coconut fiber, the literature records the spectrum with an absorption wavelength around 280 nm, which is the record for the uncondensed phenolic group (IBRAHIN *et al.*, 2007) (SARI, 2023). However, wood lignin extracted in hot alkaline solution produced a significant increase in hydroxy-phenolic and hydroxy-aliphatic groups and registered the peak at 360 nm for that treated in alkaline solution. The increase is attributed to the breaking of the α and β bonds of the ether and time and temperature were factors that influenced the change in properties. For lignin in neutral solution, the peak at 300 nm was recorded. (MANCERA *et al.*, 2011). The GOLDSCHMID study records a change in the displacement of the Uv-Vis absorption of the aromatic group, which is attributed to the ionization of the substituent groups, and which can occur in alkaline solution. Bands recorded at 280 μ, characteristic of compounds such as vanillin and acetovanilone, were shifted to 350 μ in alkaline solution, with a more than two-fold increase in intensity (GOLDSCHMID, 1953). The lignin from coconut fibers treated in alkaline sodium hydroxide solution recorded the band at 275 nm for UV-Vis analysis. The treatment was carried out at an ambient temperature of 25 ºC with the NaOH solution dissociating in an aqueous medium into hydroxyl and sodium ions. The pH of the solution is calculated and equal to 0.3, being classified as a strong base. The temperature and pH conditions used in this work did not prove to be effective enough to modify the hydroxy-phenyl and hydroxy-aliphatic groups, even in the presence of mechanical shear in the ball mill.

Electronic spectroscopy called ultraviolet (UV-Vis), in the ultraviolet region (200-400 nm) of the visible (400-800 nm), is a technique that makes it possible to identify organic substances such as lignin, for example. The technique is based on the wavelength that a given sample is capable of absorbing/reflecting. The peaks are recorded as absorbance, when energy is absorbed by the sample, and transmittance when referring to the amount of radiated energy that passed through the sample. Figure 13 presents the results of UV-Vis spectroscopy. The absorbance peaks registered the band at 275 nm for the samples tested, characterizing, according to the literature, that lignin was present in the alkaline solution. For each processing, starting from process A, the amount absorbed in the analysis was greater, characterizing a gradual and proportional increase in the number of fiber processing. (OCHONMA *et al.*, 2020)

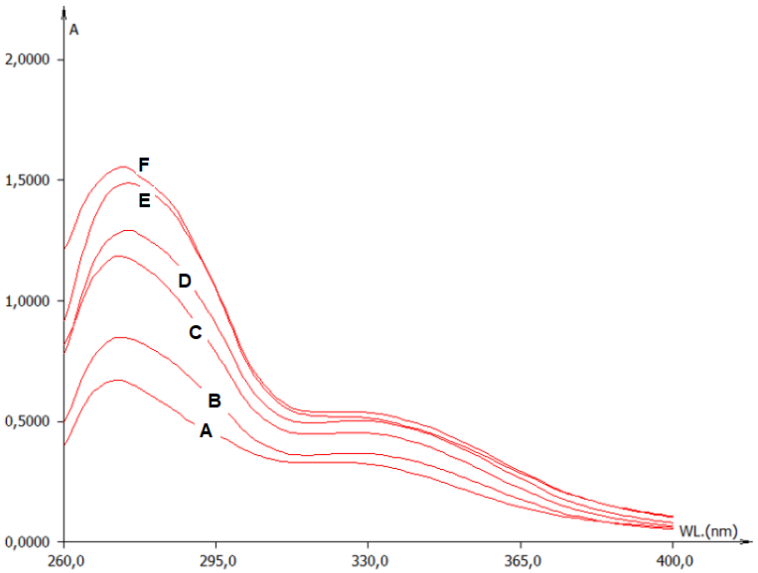


Figure 13 - UV-Vis analysis with peak at 275 nm for each fiber processing (A – F)

Figure 14 shows samples of NaOH solutions after due processing. All samples presented the same color, differing only in their tone or intensity. For processing A, the solution presented a light-yellow color and increased in color proportionally as the number of coconut fibers was processed (SARI *et al.*, 2022).

Figure 14 – Coloring of NaOH solutions after processing with coconut fiber

4. Conclusion

The coconut fibers were processed in a ball mill with a 0.5 mol/L NaOH solution, with different processing times, with each stage of the process corresponding to 60 min of rest followed by 30 min of shearing in a ball mill and in solution. In the first processing that the fibers underwent, there was a loss of approximately 35% of the initial weight, due to fiber degradation. This decrease was attributed to some factors related to the solubilization of the outer layer of the fiber, composed of fatty substances and pectin, the partial fragmentation of the fiber and the separation of fragments of the soft part of the coconut fruit mesocarp that were present in the original fiber. The number of fiber processing steps was replicated six times, and from the second processing to the sixth processing, the decrease in fiber weight showed a linear and decreasing behavior, varying between 5 and 9%. The fibers changed from brown to yellow from the first processing, and the aqueous NaOH solution changed from a light color to a darker tone. The fibers processed after the third process (C) present a mutual attraction, forming a cluster of fibers. This was attributed to a change in polarity in the cellulose chain, due to greater exposure of hydroxyls. From the third processing onwards, coconut fibers demonstrated a considerable number of fibrils. Characterization using an ultraviolet visible spectroscopy technique recorded the NaOH solution containing soluble coconut

fiber extracts at 275 nm, indicating lignin as the predominant substance present. The technique recorded an increase in absorbance intensity as the number of processing steps also increased.

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