LIGNIN EXTRACTION FROM COCONUT FIBER USING THE SODA-ANTHRAQUINONE METHOD

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ABSTRACT

Lignin, a biopolymer with great potential in the circular economy, representing about 25% of the composition of lignocellulosic biomass, has proven to be economically viable due to its abundance, sustainability, and versatility. In 2021, Brazilian coconut fiber stood out as the fifth largest production, with lignin levels that may exceed the values found in the literature, highlighting its robust structure. However, the lack of specific methods for lignin extraction in biomass emphasizes the need to evaluate and adapt existing methods, such as the soda-anthraquinone method. Comparative FTIR analysis between coconut fiber lignin and commercial lignin revealed the preservation of the same functional groups, validating the effectiveness of the proposed method for isolating lignin in coconut fiber.

Keywords: Lignin. Extraction. Soda-anthraquinone. Coconut fiber.

1 INTRODUCTION

Lignin is recognized as the second most abundant and active organic polymer on the planet, playing an essential role in the formation of rigid chemical structures and the protection of hemicellulose and cellulose[1]. Its versatility makes it an applicable component in various sectors of the economy, including cosmetics. Additionally, it is used in food packaging due to its ability to form covalent bonds between polymer chains, providing flexibility and stability. In the pharmaceutical industry, it plays a fundamental role in the formation of hydrogels with desirable characteristics, such as biocompatibility, biodegradability, and water retention capacity. Its composites have antioxidant and antimicrobial properties, being employed in the manufacturing of drug delivery systems and in promoting wound healing, making it a valuable raw material for biomedicine[2].

Lignocellulosic biomass is an important source of lignin, considered the most abundant renewable source in the world, with a lignin content ranging from 10 to 25%[3]. In Brazil, coconut production ranked fifth in the world in 2021, and approximately 80 to 85% of the coconut's weight corresponds to the mesocarp, a part especially rich in lignin. This high concentration of lignin gives coconut fiber greater rigidity compared to other agricultural waste fibers[4,5].

The wide range of applications for lignin and its abundance in coconut fiber highlight the feasibility of developing effective methods to isolate this biopolymer. This approach not only allows for more efficient use of natural resources without competing with the food industry but also contributes to waste reduction and environmental impact. There are two main methods for this purpose. The first method involves the dissolution of carbohydrate fractions, leaving lignin as an insoluble residue, using techniques such as the Klason and enzymatic acidolysis methods. The second method involves the removal and dissolution of lignin, leaving carbohydrates as insoluble residues, as seen in the process with ionic liquids and sulfonation-based alkali[6]. Although the lignin extraction process with soda-anthraquinone, which falls under the first removal method, has been widely used since 1853 in grass biomass[7], there is limited data on its application in specific biomasses such as coconut fiber.

This study aims to evaluate the efficiency of lignin extraction from coconut fiber using soda anthraquinone. For this purpose, a comparison of the chemical structures of the lignin extracted from coconut fiber and lignin from commercial sources will be conducted. This analysis will be carried out through Fourier-transform infrared spectroscopy (FTIR) with the primary purpose of identifying any alterations that may impact the polymeric properties of lignin.

2 MATERIAL & METHODS

The coconut fiber was initially characterized by adapting the methodology used for characterizing sugarcane bagasse (NREL), given the absence of a specific method in the literature for this biomass[8].

The pulping process, the initial stage in lignin extraction, employed the adapted methodology developed at LABPETROL of the Federal University of Rio Grande do Norte (UFRN)[9]. In this process, 10 g of biomass were mixed with 130 mL of

soda/anthraquinone solution (1:13 w/v ratio) in a 600 mL beaker. The mixture was heated to 60 °C for 30 minutes with constant stirring. Subsequently, the product was transferred to a stainless-steel reactor heated in an oven at 160 °C for 1 hour. Finally, the crude soda/anthraquinone pulp was filtered to obtain the black liquor, rich in lignin.

To obtain the solid lignin, an adapted methodology from Oliveira[10] was employed, 100 g of black liquor and H_2SO 6 mol.L^{-1} were added to a beaker until reaching pH 2. The mixture was subjected to constant magnetic stirring for 1 hour at 60°C. The system was then filtered, and the retained material was washed with 1% HCl solution until reaching pH 1, completing the precipitation. The obtained lignin was washed to neutral pH and dried in an oven for FTIR analysis (Fourier-transform infrared spectroscopy). Additionally, for comparison purposes, a commercial lignin (Alkaline) from the TCI brand was also analyzed.

3 RESULTS & DISCUSSION

Table 1 presents the results obtained for the content of cellulose, hemicellulose, and lignin present in the coconut fiber.

Table 1. Contents of the main constituents present in raw coconut fiber.

Celullose (%)	Hemicelullose (%)	Lignin (%)	Ref.
36.90	13.94	49.66	This paper
43,40	19,90	45,80	11
35,30	6,20	42,10	12

As described in the literature, coconut fiber has a high lignin content, which significantly contributes to its stiffness and mechanical strength[12]. The abundance of this biopolymer, combined with the availability of coconut biomass, suggests that coconut fiber is an excellent raw material for lignin extraction processes, which can be used in obtaining high-value products.

Figure 1 shows the spectra of the commercial lignin (LIGCOM) and the lignin extracted using the soda-anthraquinone methodology (LIGext), as well as their respective functional groups.

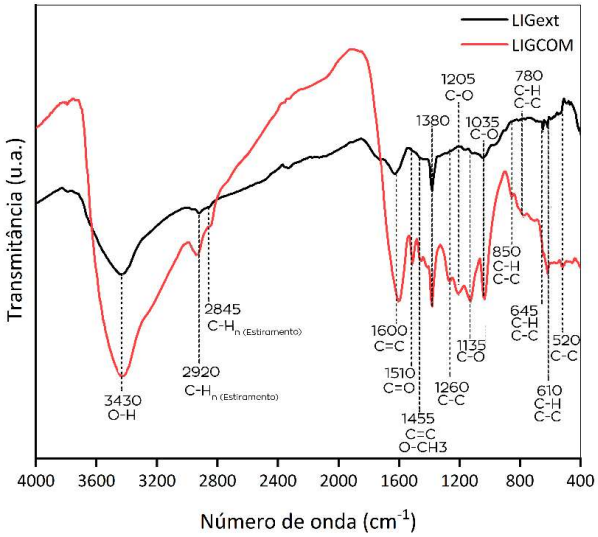

Figure 1 FTIR of commercial lignin (LIGCOM) and extracted lignin (LIGext).

Analyzing the FTIR spectrogram, it is noteworthy that the most intense band in both samples is located in the range of 3430-3200 cm^{-1}, indicative of the predominant presence of carboxylic acids and alcohols in the lignin structure. Furthermore, various groups with oxygen linkages, such as O-H, C=O, and C-O, representative of carboxylic acids, ethers, and various alcohols, are identified. The low absorption in the bands at 2920 cm^{-1} and 2845 cm^{-1}, observed in both the commercial and extracted lignin, is attributed to aliphatic C-H linkages, characterized by alkanes and alcohols[13].

Despite the similarity in their chemical structures, some differences in bands between the analyzed lignins can be observed in the spectrogram. Initially, differentiations in the bands at 1510 cm^{-1}, 1455 cm^{-1}, 1260 cm^{-1}, and 1205 cm^{-1} are highlighted, present only in the commercial lignin. These differences may be attributed to the purity of the commercial material compared to the extracted lignin, influencing the formation of more distinct bands and affecting the transmittance of the samples in the FTIR analysis[14].

The comparative analysis between the spectrogram of the commercial lignin and the extracted lignin reveals a variation in the intensity of the bands. The literature highlights that different extraction methods influence the chemical properties of lignin, affecting the intensity of the bands in FTIR[15]. Additionally, lignins originating from wood and non-wood biomass may exhibit distinct characteristics. The investigation of the specifications of the commercial lignin reveals an extraction process with sodium sulfite from trees with broad and sharp leaves. This differentiation in the process and raw material is associated with the discrepancy in the intensity of the bands between the two samples. However, the maintenance of the same functional groups of the commercial lignin in the lignin extracted by soda-anthraquinone indicates the effectiveness of this method in isolating lignin in coconut fiber.

4 CONCLUSION

Based on the characterization of coconut fiber, a significant presence of lignin was identified, aligning with literature that highlights the abundance of this polymer in this biomass. The fiber becomes a promising source of lignin, depending on the extraction methodology employed. The analysis of the spectrograms of coconut fiber lignin and commercial lignin revealed similarities in the preservation of functional groups, indicating that extraction with soda-anthraquinone does not substantially alter the chemical structure of lignin. Therefore, this approach proves to be a viable technique for extraction, potentially generating high-value molecules. Additional aspects, such as the delignification rate, should be considered in future studies to optimize process efficiency.

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ACKNOWLEDGEMENTS

Dedicated thanks to ÂNIMA, UFBA, UFRN, CNPQ, IBTR, and UNIFACS who made the research possible.