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PROGRAMA INTEGRADO (UNESP, USP E UNICAMP) DE PÓS-GRADUAÇÃO EM BIOENERGIA

CHARACTERIZATION AND APPLICATION OF LIGNIN FROM BANANA PSEUDOSTEM AS A CHELATING AGENT FOR HEAVY METAL REMOVAL

ROSANGELA ALMEIDA MAIA

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Tese apresentada ao Instituto de Pesquisa em Bioenergia de Rio Claro, Universidade Estadual Paulista, como parte dos requisitos para obtenção do título de Doutor em Ciências.

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"If your dreams do not scare you, they are not big enough"

Ellen Johnson Sirleaf

ABSTRACT

Lignocellulosic biomass has emerged as a promising alternative to traditional fossil sources and petrochemical products. As the most abundant renewable raw material in nature, lignocellulosic biomass is primarily composed of cellulose, hemicellulose, and lignin. Banana crops represent a significant potential source of lignocellulosic biomass, producing substantial amounts of residues (pseudostem, leaves, and peels) that are largely underutilized. Despite a gap in the literature, the high lignocellulosic content of these residues suggests considerable potential for exploration and utilization. This thesis investigates the composition, structure, and potential applications of lignin extracted from banana pseudostem. Initially, related studies on lignocellulosic biomass and banana residues were reviewed. Subsequently, the chemical characterization of lignin extracted from banana pseudostem and leaves was performed using the Milled Wood Lignin (MWL) methodology, analyzed through various techniques. The results indicate that both pseudostem and leaf lignins are of the HGS type, with a high predominance of S units and a low proportion of H units. NMR analysis revealed that the primary interunit linkage in these lignins is the β-O-4 ether bond. Furthermore, the pseudostem lignin exhibits a higher concentration of phenolic hydroxyl groups and a lower degree of condensation compared to leaf lignin. Additionally, the findings suggested that lignin from both sources is associated with polysaccharides via lignin-carbohydrate complexes (LCC) and suberin components. The influence of extractives and their partial removal, combined with hydrothermal and dilute acid pretreatments, on lignin structure and its potential application for heavy metal adsorption was also explored. The results show that extractives significantly impact lignin’s structural complexity. The partial removal of extractives, coupled with pretreatment, facilitates the formation of new lignin-carbohydrate associations through physical deposition and chemical bonding, leading to a more complex lignin structure. Adsorption experiments highlighted the potential of banana pseudostem lignin as an adsorbent for Pb(II) ion removal. The Langmuir model best described the adsorption process, indicating monolayer adsorption. Kinetic analysis suggests that adsorption predominantly occurs through chemical interactions, as described by the pseudo-second-order model. Finally, the valorization of lignin fractions for the production of lignin nanoparticles (LNPs) and their role in forming supraparticles, a macro-enhanced structure with potential for heavy metal removal, was examined. The results demonstrate that the hydroxyl group content and molecular weight of lignin strongly influence nanoparticle morphology and production yield. In supraparticle formation, those formed using compact nanoparticles exhibited higher Pb(II) adsorption capacity. Adsorption studies confirmed that supraparticles followed a monolayer adsorption mechanism (Langmuir model), while kinetic analysis indicated a mixed physisorption-chemisorption process. Overall, the findings of this thesis enhance our understanding of lignin's structure-property relationships and its potential for high-value applications, particularly in environmental remediation. By elucidating the effects of extractives and pretreatment on lignin structure and demonstrating its practical use in heavy metal adsorption, this research contributes to the development of sustainable materials derived from agricultural residues. This work underscores the potential of banana pseudostem lignin for innovative applications and provides a foundation for future research aimed at maximizing the utilization of biomass waste.

Keywords: Pretreatments. Banana pseudostem. Lignin. Colloidal lignin. Heavy metal removal.

RESUMO

A biomassa lignocelulósica emergiu como uma alternativa promissora às fontes de fósseis tradicionais e produtos petroquímicos. Sendo a matéria-prima renovável mais abundante na natureza, a biomassa lignocelulósica é composta principalmente por celulose, hemicelulose e lignina. As plantações de bananeira representam uma fonte potencial significativa de biomassa lignocelulósica, produzindo quantidades substanciais de resíduos (pseudocaules, folhas e cascas) que são em grande parte subutilizados. Apesar das lacunas na literatura, o alto teor lignocelulósico desses resíduos sugere um potencial considerável para exploração e utilização. Nesse contexto, esta tese investiga a composição, estrutura e potenciais aplicações da lignina extraída do pseudocaule da banana. Inicialmente, estudos relacionados sobre biomassa lignocelulósica e resíduos de banana foram revisados. Subsequentemente, a caracterização química da lignina extraída dos pseudocaules e folhas da bananeira foi realizada utilizando a metodologia de Lignina de Madeira Moída (MWL), e analisada através de várias técnicas. Os resultados indicam que tanto as ligninas dos pseudocaules quanto das folhas são do tipo HGS, com uma alta predominância de unidades S e uma baixa proporção de unidades H. A análise por RMN revelou que a ligação interunidade principal nessas ligninas é a ligação éter β-O-4. Além disso, a lignina dos pseudocaules apresenta uma maior concentração de grupos hidroxila fenólicos e um menor grau de condensação em comparação com a lignina das folhas. Adicionalmente, os resultados sugerem que a lignina de ambas as fontes está associada a polissacarídeos via complexos lignina-carboidrato (LCC) e componentes de suberina. A influência dos extrativos e sua remoção parcial, combinada com pré-tratamentos hidrotérmico e ácido diluído, na estrutura da lignina, e o potential de aplicação das ligninas extraídas para adsorção de metais pesados também foi explorada. Os resultados mostram que os extrativos impactam significativamente a complexidade estrutural da lignina. A remoção parcial de extrativos, juntamente com o pré-tratamento, facilita a formação de novas associações lignina-carboidrato através de deposição física e ligação química, levando a uma estrutura de lignina mais complexa. Experimentos de adsorção destacaram o potencial da lignina dos pseudocaules de banana como adsorvente para a remoção de íons Pb(II). O modelo de Langmuir descreveu melhor o processo de adsorção, indicando adsorção em monocamada. A análise cinética sugere que a adsorção ocorre predominantemente através de interações químicas, conforme descrito pelo modelo de pseudo-segunda ordem. Finalmente, a valorização das frações de lignina para a produção de nanopartículas de lignina (LNPs) e seu papel na formação de suprapartículas, estruturas macro-enhanced com potencial para remoção de metais pesados, também foi explorada. Os resultados demonstram que o conteúdo de grupos hidroxila e a massa molecular da lignina influenciam fortemente a morfologia das nanopartículas e o seu rendimento. Na formação de suprapartículas, aquelas formadas utilizando nanopartículas compactas exibiram maior capacidade de adsorção de Pb(II). Estudos de adsorção confirmaram que as suprapartículas seguem um mecanismo de adsorção em monocamada (modelo de Langmuir), enquanto a análise cinética indicou um processo misto de fisisorção-quimissorção. Assim, as descobertas desta tese ampliam nossa compreensão das relações estrutura-propriedade da lignina e seu potencial para aplicações de mais nobres, particularmente na remediação ambiental. Ao elucidar os efeitos dos extrativos e do pré-tratamento na estrutura da lignina e demonstrar seu uso prático na adsorção de metais pesados, esta pesquisa contribui para o desenvolvimento de materiais sustentáveis derivados de resíduos agrícolas. Este trabalho destaca o potencial da lignina dos pseudocaule da bananeira para aplicações inovadoras e proporciona uma base para futuras pesquisas visando maximizar a utilização de resíduos de biomassa.

Palavras-chave: Pré-tratamentos. Pseudocaule de banana. Lignina. Lignina coloidal. Remoção de metais pesados.

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LIST OF ABBREVIATIONS

2D HSQC NMR: Two-Dimensional Heteronuclear Single Quantum Correlation Nuclear Magnetic Resonance spectroscopy

^{31}P NMR: Phosphorus-31 NMR Nuclear Magnetic Resonance spectroscopy

AP: Dilute acid pretreatment

BPS: Extractives-free banana pseudostem

BPS-AP: Extractives-free banana pseudostem subjected to dilute acid pretreatment

BPSE: Banana pseudostem (original biomass without pretreatment)

BPSE-AP: Banana pseudostem subjected to dilute acid pretreatment

BPSE-HP: Banana pseudostem subjected to hydrothermal pretreatment

BPS-HP: Extractives-free banana pseudostem subjected to hydrothermal pretreatment

DLS: Dynamic Light Scattering

FTIR: Fourier Transform Infrared Spectroscopy

GPC: Gel Permeation Chromatography

H_2SO_4: Sulfuric Acid

HNO_3: Nitric Acid

HP: Hydrothermal pretreatment

HPLC: High-Performance Liquid Chromatography

LPEs: lignin nanoparticles from SOE

LPEs-AP: lignin nanoparticles from SOE-AP

LPEs-HP: lignin nanoparticles from SOE-HP

LPs: lignin nanoparticles from SO

LPs-AP: lignin nanoparticles from SO-AP

LPs-HP: lignin nanoparticles from SO-HP

NaOH: Sodium Hydroxide

NCh: Nanochitin

PSO: Soda pulp derived from untreated BPS

PSO-AP: Soda pulp derived from BPS-AP

PSOE: Soda pulp derived from untreated BPSE

PSOE-AP: Soda pulp derived from BPSE-AP

PSOE-HP: Soda pulp derived from BPSE-HP

PSO-HP: Soda pulp derived from BPS-HP

SEM: Scanning Electron Microscopy

SO: Lignin isolated from untreated BPS subject to alkaline process

SO-AP: Lignin isolated from BPS-AP subject to alkaline process

SOE: Lignin isolated from untreated BPSE subject to alkaline process

SOE-AP: Lignin isolated from BPSE-AP subject to alkaline process

SOE-HP: Lignin isolated from BPSE-HP subject to alkaline process

SO-HP: Lignin isolated from BPS-HP subject to alkaline process

SPLEs-HP: supraparticles produced from LPEs-HP

SPLs: supraparticles produced from LPs

SPLs-HP: supraparticles produced from LPs-HP

TGA: Thermogravimetric Analysis

UV-Vis: Ultraviolet-Visible Spectroscopy

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CHAPTER I

STATE OF THE ART

1 GENERAL INTRODUCTION

The most significant contribution of this thesis lies in the detailed characterization of lignin derived from banana pseudostem, an underexplored lignocellulosic material. The limited knowledge in literature regarding its structure, chemical composition, properties, and potential applications motivated this study. The findings presented here contribute to bridging this gap, expanding the scientific understanding of this biomass, and potentially benefiting the agro-industrial sector in the future.

This work is structured into four chapters to ensure a clear and engaging discussion of lignin characterization and its potential application for heavy metal removal.

Chapter I presents a concise yet comprehensive literature review covering key aspects related to banana cultivation, as the raw material studied is the pseudostem. It also defines lignocellulosic materials, explores the macro-and microstructural characteristics of lignin, discusses pretreatment methods and lignin extraction techniques, and introduces adsorption systems, given that the primary application of the studied lignin is in heavy metal adsorption.

Chapter II focuses on the complete characterization of lignin from banana pseudostem using the milled wood lignin (MWL) methodology. This chapter emphasizes understanding the structural and chemical composition of lignin in its native state.

Chapter III explores how pretreatments, both hydrothermal and dilute acid, and the presence of extractives can influence the structural characteristics and properties of the lignin extracted from banana pseudostem by an alkaline process. Additionally, this chapter investigates the potential of the extracted lignins as chelating agents for heavy metal removal.

Chapter IV examines the application of the lignins obtained in Chapter III in the production of lignin-based nanoparticles. It also explores the use of these lignin nanoparticles in the formation of supramolecular, a macrostructure, investigating their potential in adsorption systems for the removal of heavy metals.

This thesis, therefore, not only provides a thorough characterization of banana pseudostem lignin but also explores its practical applications, contributing to the broader field of lignocellulosic biomass valorization.

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FINAL REMARKS

5 GENERAL CONCLUSIONS

This thesis explored various aspects of lignin extracted from banana pseudostem, addressing the existing knowledge gap in the literature regarding its structure, chemical composition, properties, and potential applications. The key findings of each chapter are summarized below, culminating in a comprehensive overview of the contributions and implications of the research.

Chapter II provided a detailed structural characterization of lignin from banana pseudostem and leaves using various analytical techniques. The results revealed that the lignin from pseudostem and leaves is of the H:G:S type, with a high predominance of S units and a low quantity of H units. The β-O-4' ether linkage was identified as the primary interunit bond in lignin, which also exhibited natural partial acylation. The pseudostem lignin demonstrated a lower degree of condensation compared to leaves lignin. NMR techniques confirmed that carbohydrates and suberin are linked to lignin by LCC (Lignin-Carbohydrate Complex) structures. The analysis suggested that pseudostem lignin is better suited for applications due to its chemical composition and the presence of linked polysaccharides and aliphatic acids.

Chapter III explored the influence of extractives and pretreatment methods (hydrothermal and dilute acid) on lignin structure extracted via alkaline process and its potential application as a chelating agent for heavy metal removal. The findings demonstrated that extractives significantly affect lignin composition, leading to the formation of more complex structures when combined with pretreatments. Furthermore, adsorption experiments confirmed that lignin from banana pseudostem exhibits promising potential as an adsorbent for Pb(II) ions, with adsorption behavior following the Langmuir isotherm model and pseudo-second-order kinetics. These results reinforce the potential of lignin-based materials for environmental remediation and water treatment applications.

Chapter IV investigated the formation of lignin nanoparticles and their assembly into supraparticles for heavy metal removal. The study indicated that the morphology of lignin nanoparticles is directly influenced by the amount of hydroxyl and carboxyl groups, as well as the molecular weight of lignin. Hollow nanoparticles were found to have enhanced mechanical strength due to their higher surface area and porosity, whereas compact nanoparticles exhibited higher adsorption capacity for Pb(II) removal. The adsorption experiments revealed that supraparticles formed with compact nanoparticles performed better in heavy metal removal, suggesting a trade-off between mechanical stability and adsorption efficiency.

Overall, this thesis provides valuable insights into the structure-property relationships of lignin from banana pseudostem, its extraction and modification processes, and its functional applications in heavy metal adsorption. It demonstrated the significant influence of extractives and pretreatment methods on lignin structure, impacting its chemical properties and potential applications. Additionally, the research elucidated the mechanisms underlying the formation of lignin nanoparticles and their utility in heavy metal adsorption, contributing to the development of new materials for environmental remediation.

The findings highlight that lignin from banana pseudostem holds [potential for value-added applications. Moreover, this work advances the scientific understanding of how various factors influence lignin structure and functionality, indicating that lignin structure interferes with and influences future applications of biomass.