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

**DEVELOPMENT AND COMPARATIVE CHARACTERIZATION OF
BIODEGRADABLE BIOPLASTICS BASED ON STARCH, PECTIN, CHITOSAN
AND BANANA PSEUDOSTEM-DERIVED XYLAN**

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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.

Orientador: Dr. Michel Brienzo
Coorientador: Dr. Eddyn Gabriel Solorzano Chavez

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“Nothing will work unless you do.”
(Maya Angelou, 1993)

ABSTRACT

The growing demand for sustainable alternatives to conventional plastics, together with the need to valorize agricultural residues, has stimulated the development of biodegradable materials from renewable resources. In this study, xylan was extracted from banana pseudostem and evaluated for the development of biodegradable films. Individual films based on starch, pectin, and chitosan were also produced using glycerol as a plasticizer and characterized for comparative purposes. Films produced from xylan alone were brittle and unsuitable for characterization; therefore, a xylan–chitosan composite film (80:20, w/w) containing gallic acid was developed and characterized. The physicochemical, mechanical, thermal, optical, and biodegradation properties of the films were evaluated. Chitosan films exhibited the highest tensile strength (33.56 ± 4.88 MPa) and elongation at break ($39.93 \pm 11.33\%$), whereas starch and pectin films showed tensile strengths of 19.92 ± 4.92 MPa and 23.52 ± 4.99 MPa, respectively. Pectin films exhibited complete water solubility (100%), while starch and chitosan films showed lower solubility ranges of 17.76–23.60% and 8.58–24.10%, respectively. The xylan–chitosan composite presented intermediate solubility (59.56–69.87%), high wettability, and low tensile strength (2.70 ± 0.64 MPa). Biodegradation assays revealed complete disintegration after approximately 20 and 34 days for pectin and starch films, respectively, while the composite remained visible for approximately 51 days and chitosan films for more than 65 days. Overall, the results demonstrate the potential for valorizing banana pseudostem through xylan extraction and show that the different polysaccharide-based films exhibit distinct properties, supporting their consideration for different potential applications.

Keywords: soil degradation; hemicellulose; renewable natural resources; polysaccharides; biomass.

RESUMO

O crescente interesse por alternativas sustentáveis aos plásticos convencionais, aliado à necessidade de valorização de resíduos agrícolas, tem estimulado o desenvolvimento de materiais biodegradáveis a partir de recursos renováveis. Neste estudo, a xilana foi extraída do pseudocaule de bananeira e avaliada para o desenvolvimento de filmes biodegradáveis. Filmes individuais à base de amido, pectina e quitosana também foram produzidos utilizando glicerol como plastificante e caracterizados para fins comparativos. Os filmes produzidos apenas com xilana apresentaram-se quebradiços e inadequados para caracterização; portanto, foi desenvolvido e caracterizado um filme compósito de xilana–quitosana (80:20, m/m) contendo ácido gálico. As propriedades físico-químicas, mecânicas, térmicas, ópticas e de biodegradação dos filmes foram avaliadas. Os filmes de quitosana apresentaram a maior resistência à tração ($33,56 \pm 4,88$ MPa) e elongação na ruptura ($39,93 \pm 11,33\%$), enquanto os filmes de amido e pectina apresentaram resistências à tração de $19,92 \pm 4,92$ MPa e $23,52 \pm 4,99$ MPa, respectivamente. Os filmes de pectina apresentaram solubilidade completa em água (100%), enquanto os filmes de amido e quitosana apresentaram menores faixas de solubilidade, entre 17,76–23,60% e 8,58–24,10%, respectivamente. O compósito de xilana–quitosana apresentou solubilidade intermediária (59,56–69,87%), alta molhabilidade e baixa resistência à tração ($2,70 \pm 0,64$ MPa). Os ensaios de biodegradação revelaram desintegração completa após aproximadamente 20 e 34 dias para os filmes de pectina e amido, respectivamente, enquanto o compósito permaneceu visível por aproximadamente 51 dias e os filmes de quitosana por mais de 65 dias. De modo geral, os resultados demonstram o potencial de valorização do pseudocaule de bananeira por meio da extração de xilana e mostram que os diferentes filmes à base de polissacarídeos apresentam propriedades distintas, permitindo considerar seu potencial para diferentes aplicações.

Palavras-chave: degradação do solo; hemicelulose; recursos naturais renováveis; polissacarídeos; biomassa.

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1 INTRODUCTION

A significant amount of plastic waste is produced globally each year, while only a small fraction is effectively recycled. Most plastic materials accumulate in landfills, are incinerated, or escape into the environment, particularly aquatic ecosystems. In marine environments, plastic pollution poses risks to wildlife and human health due to the generation and accumulation of microplastics, which can enter food chains and persist for long periods (Perreard et al., 2023; Lau et al., 2020). Because conventional plastics are primarily derived from fossil resources and exhibit high resistance to biological degradation, they may remain in the environment for centuries, aggravating environmental and economic challenges worldwide (Machado et al., 2018).

In response to these concerns, increasing attention has been directed toward bioplastics derived from renewable resources as alternatives to petroleum-based plastics. Polysaccharide-based materials are particularly attractive because of their abundance, renewability, biocompatibility, and film-forming ability. Starch, chitosan, pectin, and hemicelluloses can be processed into biodegradable films for applications including food packaging, edible coatings, biomedical materials, and other single-use products (Kumar and Thakur, 2017; Atiwesh et al., 2021). Among these materials, starch is widely available and can form continuous films (Pooja et al., 2024), while chitosan and pectin exhibit favorable film-forming and functional properties, including biodegradability, antimicrobial activity, and biocompatibility (Jin et al., 2024; Yüksela et al., 2024).

Hemicelluloses, particularly xylan, have also attracted interest as renewable feedstocks for biodegradable materials. Xylan is one of the main polysaccharides in lignocellulosic biomass and can be obtained from agricultural residues. The use of such residues contributes to biomass valorization and circular bioeconomy strategies by converting underutilized resources into value-added materials. Banana pseudostem, generated in large quantities during banana cultivation, is a lignocellulosic residue with potential for valorization and use as a renewable source of raw materials (Abe et al., 2021; Sewsynker-Sukai et al., 2025).

Despite the advantages of polysaccharide-based films, their individual properties may limit their applications. Starch-based films are generally sensitive to moisture and may exhibit limited mechanical performance, while chitosan requires

acidic conditions for solubilization. Pectin films may also show high water solubility, and pure xylan can produce brittle films with poor mechanical integrity and flexibility (Mali et al., 2005b; Elsabee and Abdou, 2013). Therefore, polymer blending, plasticization, and incorporation of functional additives are commonly used to improve film performance.

Among these approaches, polymer blending enables the combination of complementary properties from different biopolymers. Xylan–chitosan systems have attracted interest because the combination of these polymers can improve the applicability of xylan-based films compared with the use of pure xylan (Schnell et al., 2017; Schnell et al., 2021). In this context, starch, pectin, and chitosan can also serve as relevant reference materials for evaluating the behavior of xylan-based films and distinguishing the effects associated with the intrinsic properties of each polymer.

Therefore, this study investigated the production and characterization of individual biodegradable films based on xylan extracted from banana pseudostem, starch, pectin, and chitosan, using glycerol as a plasticizer, as well as a xylan–chitosan composite film. The physicochemical, mechanical, thermal, optical, and biodegradation properties of the films were evaluated to assess their potential applications. Emphasis was placed on the valorization of banana pseudostem through xylan extraction and on the development of a xylan–chitosan composite as a strategy to overcome the limitations of films produced from pure xylan.

6 CONCLUSIONS

This study demonstrated the potential of xylan extracted from banana pseudostem for the development of biodegradable materials and comparatively evaluated films individually produced from starch, pectin, and chitosan, as well as a xylan–chitosan composite film.

Films produced from pure xylan showed high brittleness and insufficient integrity for further characterization, even after glycerol addition. However, the incorporation of chitosan enabled the production of a xylan-based composite film with sufficient integrity for handling and characterization, demonstrating that polymer blending can be an effective strategy to overcome the limitations observed for pure xylan films.

The comparative characterization showed that the properties of the films were strongly influenced by the composition of each polysaccharide system. Chitosan films exhibited the highest tensile strength and elongation at break, whereas starch films showed intermediate mechanical performance, low moisture content, and moderate water solubility. Pectin films exhibited complete water solubility and the fastest degradation in soil. In contrast, the xylan–chitosan composite presented higher moisture content and opacity, high UV-blocking capacity, the lowest visible light transmission, high surface wettability, intermediate water solubility, and lower mechanical resistance than the chitosan films.

The different degradation profiles observed among the films further demonstrated the influence of polymer composition on their behavior. Pectin and starch films showed faster disintegration, whereas chitosan films presented greater resistance to degradation. The xylan–chitosan composite exhibited an intermediate degradation profile, demonstrating that the incorporation of xylan into a composite system can modify the behavior of the resulting material.

Overall, the results demonstrate that no single formulation presented superior performance for all evaluated properties. Instead, each polysaccharide-based film exhibited specific advantages and limitations that should be considered according to its intended application. The successful incorporation of xylan extracted from banana pseudostem into a composite film also demonstrates the potential for the valorization of this agricultural residue as a renewable raw material for the development of biodegradable materials.

Future studies should investigate strategies to further improve the mechanical performance and water resistance of xylan-based films, including the evaluation of different polymer ratios, plasticizers, crosslinking approaches, and functional additives. In addition, the potential applications of the developed formulations could be further assessed according to their specific performance profiles, particularly in sustainable material and packaging-related applications.

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