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**PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS BIOLÓGICAS
(MICROBIOLOGIA APLICADA)**

**CONTRIBUTION OF XYLAN TO PHYSICOCHEMICAL AND BIODEGRADATION
PROPERTIES IN STARCH-BASED BIOPLASTICS.**

JOÃO VICTOR CARPINELLI MACEDO

**Rio Claro
Julho - 2021**

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JOÃO VICTOR CARPINELLI MACEDO

Dissertação apresentada ao
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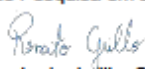
TÍTULO DA DISSERTAÇÃO: CONTRIBUTION OF XYLAN TO PHYSICOCHEMICAL AND BIODEGRADATION PROPERTIES IN STARCH-BASED BIOPLASTICS

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I dedicate this work to all my
family and to everyone who
helped me get to where I
am.

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ABSTRACT

Plastic has served society very well for decades, being able to present itself as a light and moldable material, mechanically and thermally resistant, inert to chemical products, electrical insulator, in addition to having interesting optical characteristics such as its transparency. Such characteristics, associated with the low price of its raw material, a residue from the processing of petroleum until then little used, made it crucial for the industry and the market, which consolidated its presence in society in different countries. However, as it is a resistant material, remaining for centuries without fully decomposing, and for having a large production in order to meet the needs of a consumer society, it began to accumulate in the environment over the years since its creation. Its abundance in the environment causes damage to life in different biomes, especially marine environments, in addition to having residues that can be toxic and reach humans through the food chain. A possible solution is biodegradable materials that use renewable raw materials such as proteins, lipids, and polysaccharides, the so-called bioplastics. In this context, the present study aimed to produce bioplastics based on starch with different proportions of xylan extracted from sugarcane bagasse and glycerol as a plasticizer. For this, their physicochemical properties (solubility, hydrophilicity, moisture, water barrier, opacity, crystallinity, and mechanical resistance) were evaluated. The biodegradation of the bioplastic was determined by buried and CO₂ released. As a result, it was observed that the increase in the proportion of xylan made the bioplastics more soluble in water, susceptible to enzymatic attack by microorganisms, and opaque. Through of the contact angle test, the bioplastic with starch and plasticizer showed lower affinity to water compared to the one including xylan, which among them the proportion of 25 % (w/w of the polysaccharides) was the least hydrophilic. Bioplastic with 5 % (w/w of polysaccharides) xylan showed better crystallinity followed by starch. As for its mechanical strength, the bioplastic with 10 % xylan (w/w of polysaccharides) owned the highest tensile stress (2.56 MPa), but the presence of xylan considerably reduced its elongation at break (ranging from 16 to 37 %) in relation to the starch bioplastic (208.8 %). In general, after 30 days of burial, the bioplastics had practically no fragments visible to the naked eye, and the rate of CO₂ production together with the increase in cell count after the biodegradation period demonstrate that there was an effective catabolic action of the microorganisms in the bioplastics. Thus, the 5 % xylan bioplastic showed the best physicochemical results when obtaining a biodegradable material.

Keywords: Bioplastic, xylan, starch, biomass, biodegradable.

RESUMO

O plástico serviu muito bem à sociedade durante décadas, podendo se apresentar como um material leve e moldável, mecanicamente e termicamente resistente, inerte a produtos químicos, isolante elétrico, além de possuir características ópticas interessantes como sua transparência. Tais características, associada ao baixo preço da sua matéria prima, um resíduo do processamento de petróleo até então pouco utilizado, fizeram com que ele se tornasse crucial para a indústria e o mercado, o que consolidou sua presença na sociedade em diferentes países. Contudo, por ser um material resistente, permanecendo séculos sem se decompor totalmente, e por ter uma larga produção a fim de atender as necessidades de uma sociedade consumista, ele passou a se acumular no meio ambiente ao longo dos anos desde sua criação. Sua abundância no meio ambiente acarreta prejuízos à vida em diferentes biomas, especialmente os ambientes marinhos, além de possuir resíduos que podem ser tóxicos e chegar ao ser humano através da cadeia trófica. Uma possível solução são os materiais biodegradáveis e que utilizam matéria-prima renovável como proteínas, lipídeos e polissacarídeos, os chamados bioplásticos. Neste contexto, o presente estudo objetivou produzir bioplásticos baseado em amido com diferentes proporções de xilana extraída do bagaço de cana-de-açúcar e glicerol como plastificante. Para isso, suas propriedades físico-químicas (solubilidade, hidrofiliicidade, umidade, barreira à água, opacidade, cristalinidade, e resistência mecânica) foram avaliadas. A biodegradação do bioplástico foi determinada por enterramento e liberação de CO₂. Como resultado, foi observado que o aumento na proporção de xilana tornou os bioplásticos mais solúveis em água, susceptíveis ao ataque enzimático de microrganismo, e opacos. Por meio do ensaio de ângulo de contato, o bioplástico apenas com amido e plastificante apresentou menor afinidade à água, comparado aos compostos por xilana, que dentre eles a proporção de 25 % (m/m de polissacarídeos) foi a menos hidrofílica. O bioplástico com 5 % (m/m de polissacarídeos) de xilana apresentou melhor cristalinidade seguido pelo de amido. Quanto à sua resistência mecânica, o bioplástico com 10 % de xilana (m/m de polissacarídeos) foi o que possuiu maior tensão de tração (2,56 MPa), porém a presença de xilana reduziu consideravelmente sua elongação na quebra (variando de 16 a 37 %) em relação ao bioplástico de amido (208,8 %). No geral, após 30 dias enterrados os bioplásticos já não apresentavam praticamente nenhum fragmento visível a olho nú, e a taxa de produção de CO₂ juntamente com o aumento da contagem de células após o período de biodegradação demonstram que houve uma efetiva ação catabólica dos microrganismos nos bioplásticos. Sendo assim, a concentração de 5 % de xilana foi a que mostrou melhores resultados físico-químicos ao obter um material biodegradável.

Palavras-chave: Bioplástico, xilana, amido, biomassa, biodegradável.

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CHAPTER I: INTRODUCTION

The fossil-based plastic, that we conventionally use, assumed an important role in society in a way that it would be impossible to abruptly abolish its use, since it is present in our daily lives from the time we wake up to the time we go to sleep. Although plastic waste management policies can be carried out, the ideal level of recycling to contain the generation of waste, which tends to grow as the world population grows, is a difficult level to be reached. And the reasons for this are the demand for high investment in collection and sorting centers, the problem of the quality of recycled materials in relation to primary plastic and the most important factor, the cooperation of the population, because even if the first two items are resolved, will not be as effective if there is no awareness of the population. This last requirement is a more complex issue, because it is strictly related to the education and civility of each individual.

Biodegradable polymers produced from renewable sources have been seen as a promising strategy to replace (partially) the intensive use of plastic derived from petroleum due to the growing environmental awareness in recent decades, as this synthetic polymer remains in the environment for centuries. The use of polysaccharides has been studied and arouses the interest of researchers as they are molecules capable of forming long chains. The polysaccharides, can form a cohesive network due to inter and intra-molecular bonds, and can also be consumed and degraded by microorganisms resulting in small molecules common to the environment. In addition to being biodegradable, bioplastics have the raw material from renewable sources, and potentially toxic substances are not used in their composition. By relying on several hydroxyl groups, polysaccharides also have the ability to mix with other components (plasticizers and additives) improving their functionality.

Starch is generally used in bioplastic/film production because it is highly available and easy to extract, it has low cost, is biodegradable and biocompatible. It is still widely studied as edible bio-packaging because it has high energy value, in addition to forming films without odor, taste, color, toxicity, showing semipermeability to gases such as CO₂, O₂ and water vapor. However, native starch has a hydrophilic character and tends to form films with poor mechanical properties. Basically formed by two types of molecules, amylose (forms linear chains) and amylopectin (branched chains), both having a large amount of carboxyl groups capable of making hydrogen bonds.

In this context, xylan, a type of hemicellulose composed mostly by pentoses known as xylose, can present itself as a compatible sugar to starch, as it shares practically the same characteristics. Xylan can contribute to form bioplastic with greater mechanical resistance, in addition to theoretically preventing the retrogression process characteristic of starch, which consists of the loss of water molecules causing dryness. The interest in xylan also comes from the fact that it is a polysaccharide with potential for application in bioplastics that is still little used when compared to cellulose, even though it has considerable participation in the composition of plant biomass.

The study of starch in the preparation of biodegradable bioplastic is already well documented. However, from our knowledge, there are not study in the literature dedicated to understanding the contribution and correlation of xylan in bioplastic. In this context, the purpose of this study was to combine starch and xylan (extracted from sugarcane bagasse) to produce bioplastics. The contribution of the xylan concentration was evaluated in properties such as biodegradability, crystallinity, mechanics, opacity, water affinity, and water barrier.

CHAPTER II: OBJECTIVES

1. General objective

The main objective of this study was to evaluate the addition of xylan extracted from sugarcane bagasse in the composition of bioplastics in combination with starch. Physicochemical properties were determined and related to the biodegradation of the bioplastic.

1.1. Specific objectives

- Identify the better starch gelatinization conditions and glycerol concentration;
- Study the contribution of the xylan in the bioplastic compositions on the moisture, solubility, opacity, mechanical, crystallinity, and hydrophilicity, defining the most suitable proportion;
- Analyze the time needed for biodegradation of the bioplastic composed of xylan and starch by burial and CO₂ release.

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CHAPTER V: CONCLUSION

Researchers from companies and governmental initiatives around the world are trying to develop a material that is similar to plastic but that can be completely degraded. In this sense, the use of biopolymers/biomolecules from plant biomass is a renewable and environmentally beneficial option in the production of bioplastics, as in the case of the xylan used in this study together with starch. In relation to the bioplastic production, the gelatinization process contributed to its integrity, as well as its flexibility, which was also favored when the glycerol content was 20 % (w/w of polysaccharides), resulting in films that were easier to handle and remove from the drying plate. The association of polysaccharides generated homogeneous and intact bioplastics, indicating a good association between them. Moreover, xylan tended to increase the opacity, solubility, and hydrophilicity of bioplastics reducing their crystallinity and elongation at break. When buried, the bioplastics were almost completely degraded within a month, and in those where xylan was more present, CO₂ production peaked in fewer days, indicating an even more accelerated decomposition. Its properties may not make xylan bioplastics ideal candidates for use in food packaging and bags, for example, but in applications that do not require high mechanical strength but rather a fast biodegradation rate, they are an efficient option, such as in agricultural applications where some products or even the cultivation itself needs a covering that preferably can be degraded over time without generating potentially toxic waste and plastic waste.