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Characterization And Development of Nut Grass (Cyperus rotondus L.) Biodegradable Plastic

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ABTRACT

Plastic pollution has profoundly affected Earth's ecosystems, posing a grave threat to the balance of nature. Conversely, Cyperus rotundus Linn, commonly known as nut grass, is one of the world's worst weeds, infesting a wide range of crops and reducing yields by 20 to 90 percent. Notably, the starch granules in its tubers have a significant amylose concentration of 26.73 percent and a yield of 24.1 percent on a dry weight basis. This study aims to characterize and develop a biodegradable plastic using starch extracted from nut grass tubers. The method and formulation of Marichelvam (2019) were used for bioplastic production, with five treatments and three replicates each. Each treatment had different nut grass starch compositions (10 g, 7.5 g, 5 g, and 2.5 g) combined with glycerol, citric acid, gelatin, and distilled water. The study's findings revealed that Treatment 4 was the most effective in terms of elongation at break (35.6580%), water absorption (41.875%), and biodegradability (84.45266%). Moreover, Treatment 3 exhibited the highest tensile strength at 4.76504 MPa, while Treatment 2 had the highest density of 1.41193 g/mL. All treatments experienced significant weight loss between 260°C and 350°C. The FTIR spectra revealed the presence of C-H stretches at 2926.03/cm, 2857.05/cm, 1455.83/cm, 1409.83/cm, 1365.55/cm, 1238.82/cm, 925.52/cm, 860.85/cm,
822.05/cm, 810.55/cm, and 761.69/cm, indicating the starch structure present in all bioplastic samples. Based on the results, it is concluded that the most effective formulation for producing bioplastic from nutgrass tuber starch is Treatment 4, with a 5 g nut grass starch composition

Keywords: *Plastic, Nut Grass, Tubers, Starch, Bioplastic*

INTRODUCTION

Plastic has permeated every aspect of human lives, revolutionizing industries, increasing convenience, and improving countless products. Yet, beneath its seemingly boundless utility lies an expanding crisis that threatens the planet where we live.

Currently, plastic pollution has emerged as one of the most pressing global challenges, profoundly affecting the Earth's ecosystems and posing a grave threat to the delicate balance of nature. Wherein, there are over 300 million tons of plastic produced globally and one of the major contributors is plastic packaging designed for single use (Vasarhelyi, 2023).

These plastics designed for disposable water bottles, food containers, and shopping bags are used briefly but can persist in the environment for hundreds of years. Most of these are improperly discarded, littering streets, parks, natural landscapes, and ending up in landfills.

According to the survey conducted by the Global Alliance for Incinerator Alternatives (GAIA), Filipinos used almost 48 million shopping bags everyday adding up to more than 17 billion a year excluding the 165 billion amount of transparent plastic bags or mostly known as "labo" bags across the country.

Every day, the equivalent of 2,000 garbage trucks full of plastic is dumped into the world's oceans, rivers, and lakes, contributing to an annual total of 19 to 23 million tons of plastic waste ("Plastic Pollution", n.d.). It is estimated that by 2050, the accumulation of plastic—a man-made material—in the oceans and seas will outweigh all the fish in the sea (Fava, 2022).

For these reasons, since plastics cannot be easily decomposed by natural processes, researchers and scientists are currently focusing on the use of natural resources to create an alternative that can be essential as much as plastic but can also offer several significant benefits to the environment at the same time.

Biodegradable plastic is a type of material designed to break down naturally in the environment, primarily through the action of microorganisms, into simpler substances such as water, carbon dioxide, and biomass. These can be made from plant-based materials, synthetic materials, or blends that are engineered to degrade relatively quickly, reducing their long-term environmental impact. (Song et al., 2009)

One of the most common renewable sources used as a key ingredient in the production of bioplastics is starch that can be found in various crops, including corn, potatoes, cassava, and wheat. (Gadhave et al., 2018)

Starch is a polysaccharide that tends to be found in seeds, bulbs, and tubers. It is made up of many glucose monomers that are bound together by glycosidic linkages. Starch is valuable because, in addition to being inexpensive and widely available, its amylose content is responsible for the gelatinization and retrogradation, which are required during film formation. ("Starch", n.d.)

Starch-based bioplastics also provide several advantages such as better tensile strength, biodegradability and ease of manufacturing. They can be processed using conventional plastic processing techniques, such as extrusion, injection molding, and film blowing. This versatility allows for the production of a wide range of biodegradable products, including packaging materials, disposable cutlery, and agricultural films. (do val Siqueira et al., 2020)

On the other hand, Cyperus rotondus Linn, commonly known as nut grass is considered one of the world's worst weeds, infesting a wide range of crops and growing in a variety of soil types by 20 to 90 percent yield. (Peerzada, 2017)

It grows quickly, produces subterranean tubers that can regenerate plants, and is herbicide tolerant. The starch granules of its tubers are reported to have a significant amylose concentration of 26.73 percent and yield a percentage of 24.1 on a dry weight basis (Umerie et al., n.d.).

Hence, the primary aim of this study was to characterize and develop a biodegradable plastic using starch extracted from its tubers. Consequently, it endeavored to institute a sustainable methodology for weed control, thereby introducing a resource of considerable value to agriculture.

MATERIALS AND METHODS

Experimental Design

The study was laid out in Completely Randomized Design (CRD) with five (5) treatments and three (3) replications as shown in the table below:

Table 1. Experimental Design

Treatments	Replicates		
	1	2	3
1	T1R1	T1R2	T1R3
2	T2R1	T2R2	T2R3
3	T3R1	T3R2	T3R3
4	T4R1	T4R2	T4R3
5	T5R1	T5R2	T5R3

Furthermore, the following are the different ratios of nut grass and corn starch per treatment:

Table 2. Different Treatments of Bioplastic

Treatment	
T1	10 g Corn Starch
T2	10 g Nut Grass Starch
T3	7.5 g Nut Grass Starch + 2.5 g Corn Starch
T4	5 g Nut Grass Starch + 5 g Corn Starch
T5	2.5g Nut Grass Starch + 7.5g Corn Starch

The value from the tensile properties, density, thermogravimetric analysis, water absorption, and biodegradability were the responsible variables.

Materials

The following materials, glassware and instruments were used in the study: beaker, graduated cylinder, spatula, stirring rod, magnetic stirrer, mortar and pestle, cheesecloth, plastic container, blender, analytical balance, and hot plate.

Moreover, citric acid, bovine gelatin, distilled water, glycerol, and corn starch were the chemicals and reagents used in conducting the study.

Collection of the Nut Grass (Cyperus rotondus) Tubers

The nut grass (Cyperus rotondus L.) weeds were collected at Purok 5, Brgy. Mauanan, Rizal, Cagayan. The tubers were separated from the stem, cleaned, and peeled for the extraction of starch.

Extraction of Starch

A total of 200 g of nut grass tubers were processed using the method outlined by Gonzales and Reyes (2020). The tubers were ground using an electric blender with 200 mL of distilled water. The mixture was filtered through cheesecloth, and this procedure was repeated three times to extract more starch.

The filtrate was then mixed with distilled water and allowed to settle in a beaker for one hour. The supernatant was discarded, and distilled water was repeatedly added to the filtrate until the water became clear, indicating that the starch was clean.

Once cleaned, the starch was sun-dried for 30-45 minutes to achieve the desired texture and dryness. Finally, it was ground into a fine powder using a mortar and pestle.

Preparation of Bioplastics Film

The starch from nut grass, cornstarch, glycerol, gelatin, and citric acid were added to 100 mL distilled water in various ratios as shown below. These formulations are adapted from the study of Marichelvam et al. (2019).

Table 3. Formulation of Different Treatments

T	NS	CS	G	CA	GL	DW
	Weight (in Grams)					
T1	0	10	3	1	2	100

T2	10	0	3	1	2	100
T3	7.5	2.5	3	1	2	100
T4	5	5	3	1	2	100
T5	2.5	7.5	3	1	2	100

Legend: T - Treatment; NS - Nut Grass Starch; CS - Corn Starch; G - Glycerol; CA- Citric Acid; GL - Gelatin; DW - Distilled Water

The mixture was stirred at a rate of 180 rpm for 10 minutes. Then the mixture was heated on a hot plate at 100°C, and manual stirring was done for 70 minutes, continuously. Then it was poured onto Teflon-coated tray and spread uniformly with a thickness of less than 1 mm. It took three to four days for the mixture to dry out and the cast film was removed.

Characterization of Bioplastic Films

Tensile Properties

Tensile properties were measured using the standard ASTM D882 (Adapted) at DOST - ITDI laboratory. Specimens need to be between 250 mm long and 15 mm wide, and the thickness should be less than 1 mm. In this procedure, the specimen extension can be measured by grip separation, extension indicators, or displacement of gauge marks. (" Standard Method for Tensile Properties of Thin Plastic Sheeting",n.d.)

Moreover, tensile strength (nominal) was calculated by dividing the maximum load by the original minimum cross-sectional area of the specimen. The result was expressed suing megapascal (mPa). The calculation for tensile strength at break was calculated in the same way except that the load at break was used in place of maximum load. ("Standard Method for Tensile Properties of Thin Plastic Sheeting", n.d.)

Percent elongation at break, on the other hand, was calculated by dividing the extension at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. The result was expressed in percent. ("Standard Method for Tensile Properties of Thin Plastic Sheeting", n.d.)

Density

The method used for the test of density was adapted from the study of Darni et al. (2017). Each three (3) grams bioplastic sample was placed into a 10 mL beaker filled with five (5) mL distilled water for fifteen minutes. Subsequently, the new volume of water was calculated by determining the difference between the final and initial volumes of water.

Using the new volume, the density of bioplastic was measured using the equation below:

$$\textit{Density} = \frac{\textit{mass}}{\textit{volume}}$$

Water Absorption

The standard method ASTM D570 (Adapted) was used to determine the water absorption of the plastics. The conditioned samples were placed in a container of distilled water, maintained at a temperature of 23 ± 1°C, and positioned on their edges while completely submerged ("Standard Test Method for Absorption of Plastics", n.d.).

After precisely 24 hours, with a tolerance of ± 0.5 hours, the specimens were individually removed from the water. Any surface moisture was wiped away using a dry cloth, and the samples were promptly weighed ("Standard Test Method for Absorption of Plastics", n.d.).

Thermogravimetric analysis

Thermogravimetric analysis was performed using the Hitachi STA200RV machine at NASAT Labs. The samples were cut into small pieces using disinfected scissors with mass ranging from 8.9 mg to 9.31 mg and were placed into the aluminum pan. The temperature range for the analysis was 35°C to 500°C with a heating rate of 10°C per minute. Additionally, the flow rate of N2 gas used was 100 mL per minute.

Biodegradability Test

The soil burial test method adapted from Nissa et al. (2019) was used to test the biodegradability of the bioplastic. Fifteen (15) seedling bags were filled with compost soil.

Moreover, the bioplastic was cut into 20 x 40 mm pieces and buried in the compost soil at a depth of 7.5 cm, then incubated at room temperature for fifteen (15) days.

Sampling occurred every three (3) days, during which each buried sample was cleaned and dried before weighing. The weight loss of the bioplastic was calculated using the equation below:

$$\% \textbf{ Weight Loss} = \frac{(W_0 - W_f)}{W_0} \times 100$$

where W_0 = initial mass; W_f = degradation mass at each designated day.

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR - Attenuated Total Reflectance (ATR) Material Analysis (Spectrum) was conducted at DOST - PTRI laboratory using FTIR Shimadzu IR Spirit machine.

Analysis of the Data/ Statistical treatment

Significance of the results and statistical differences were carried out using Least Significant Difference (LSD) Test and One-way ANOVA to identify significant differences among treatments ($p < 0.05$).

CONCLUSIONS

This study focused on characterization and development of nut grass (Cyperus rotondus L.) bioplastic. Based on the results and findings in the study, the following conclusions were reached:

1. A percentage yield of 22.64% starch was obtained from nut grass tubers.
2. The five treatments exhibit significant differences in terms of tensile properties, water absorption, density, and biodegradability at a significance level of 0.05.
3. A maximum of 35.6580% is achieved in terms of elongation at break, 41.875% in water absorption, and 84.45266% in weight loss for biodegradability. Additionally, the highest density value of 1.41193 g/mL and a tensile strength of 4.76504 MPa are obtained. Furthermore, all treatments experience significant weight loss between 260°C and 350°C.
4. The FTIR spectra reveal the presence of C-H stretches at 2926.03/cm, 2857.05/cm, 1455.83/cm, 1409.83/cm, 1365.55/cm, 1238.82/cm, 925.52/cm, 860.85/cm, 822.05/cm, 810.55/cm, and 761.69/cm, respectively, representing the starch structure present in all bioplastic samples. Moreover, strong absorption is evident in 3286.75/cm absorption peaks which corresponds to hydroxyl group.
5. Statistical treatment using Analysis of Variance and LSD test showed that the differences among treatments were statistically significant, which strongly suggests that nut grass is an effective source of starch to make bioplastic ($p>0.05$).

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