



Preparation and Characterization Studies of *Amorphophallus paeonifolius* and *Manihot esculenta* as a Bioplastic using Glycerol and Agar-Agar as Plasticizer

J. MORRIS PRINCEY^{1*}, A. NANDHINI² and E. ABINAYA³

PG and Research Department of Chemistry, Holy Cross College (Autonomous),
Tiruchirapalli-620002, Tamil Nadu, India.

*Corresponding author E-mail: princeymorris@gmail.com

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ABSTRACT

Bio-plastics are biodegradable materials which can be obtained from the renewable sources such as corn starch, straw, vegetable fats and oils, wood-chips, recycled food waste, saw dust etc. They can be used as an alternative to the commercial plastics in the market which contaminate our environment. Now-a-days bio-plastics are worldwide popular due to its concern towards the environment, limited fossil fuel resources and the climatic change. The purpose of this study is to assess the properties of the starch based bio-plastics prepared from *Amorphophallus paeonifolius* and *Manihot esculenta* using glycerol and agar-agar as the plasticizers. The synthesized bio-plastic was characterized with the help of UV-Visible spectrophotometer, FT-IR spectroscopy and SEM Analysis.

Keywords: *Amorphophallus paeonifolius*, *Manihot esculenta*, Starch isolation, Physico-chemical parameters, FT-IR and UV-spectroscopic Studies, SEM analysis.

INTRODUCTION

The environmental impact of plastic wastes is one of the current concerns due to the accumulation of the non-biodegradable plastics on earth. Plastics play a vital role in packaging industry and in the recent years over 67 million tons¹ of packaging waste results in a negative environmental impact. Therefore the synthesis of bio-polymers has generated a great interest amongst the researchers as they bring a significant contribution to the sustainable development with a wider range of disposal options. Bio-plastics are

biodegradable plastics, derived from biological substances like corn and sugarcane rather than petroleum. According to the European Bio-plastics Organization, bio-plastics are defined as "Plastics based on renewable or bio-based resources or plastics which are biodegradable or compostable". They are 100% degradable, resistant and versatile and they are used to develop medical materials used in packaging, cosmetics, food additives and clothing. A polymer categorized to be bio-plastic must be biodegradable, it should not be a potential threat to the growth of the plants and it must disintegrate² within a time frame of two months. Bio-plastics are water



insoluble, resistant, optically pure and impermeable to oxygen. Many scientists work on biodegradable polymers as a substitute for petrochemical based polymers, focusing on environmental, economic and safety challenges. These bio-degradable polymers or bio-plastics show considerable eco-friendly surroundings than the conventional plastics. Cassava and elephant foot yam are important food³ and cash crops in south Asia. The present study aims in the preparation and characterization of bio-plastics from cassava (*Manihot esculenta*) and elephant foot yam starch (*Amorphophallus paeonifolius*) using glycerol and agar-agar as plasticizers. Glycerol is widely used as a plasticizer⁴ in fabricating the bioplastics. The bio-plastics obtained were further studied for their physico-chemical⁵ and spectroscopic properties.

MATERIALS AND METHODS

Starch Extraction

The fresh tubers were peeled off and washed carefully in water to remove soil and other particles. After the manual peeling it was cut into small pieces and dried. After drying, the tuber was grinded into a fine powder after which the powder was submerged in distilled water for about 24 hours. After 24 h, filtration was done using a muslin cloth. The starch was dewatered and dried at sunlight. Finally, the dried starch was collected in container and used for further studies⁶.

$$SC(\%) = \frac{W_2}{W_1} \times 100 \quad (1)$$

Where, SC is Starch isolation, W2 is dry weight of the sample and W1 is wet weight of the sample.

Test for moisture content of starch

The petri dishes with lids were washed and dried in an oven at 105°C and cooled to room temperature⁷ in a desiccator. Approximately 2 g of starch samples were weighed accurately in the petri dishes. The samples were dried for 8 h at 120°C, cooled in desiccator and weighed. The moisture content was calculated using equation 2.

$$MC(\%) = 100 - \frac{W_2}{W_1} \times 100 \quad (2)$$

Where, MC is Moisture content, W2 is dry weight of the sample, W1 is wet weight of the sample.

Test for ash content

The crucibles were cleaned, heated for 30 min at 200°C, cooled to room temperature in a desiccator. Approximately 2 g of the starch sample (W1) were weighed accurately in the crucible and incinerated on a Bunsen burner until the carbonization of the sample was complete. Then the incineration was done at 200°C for about 2 hours. The incinerated samples were cooled in a desiccator to room temperature and weighed (W₂).

$$AC(\%) = \frac{W_2}{W_1} \times 100 \quad (3)$$

Where, AC is the Ash Content, W2 is dry weight and W1 is wet Weight

pH of the starch

About 20% W/V dispersion of the sample was shaken in water for 5 min the starch was allowed to settle and the pH of the water phase was determined using a calibrated pH meter⁸.

Titrateable acidity

2 gram of the starch sample was suspended in 20 mL distilled water and titrated against 0.1 M NaOH using phenolphthalein as the indicator.

Amylose content

About 0.1 g of the starch sample was taken in a test tube along with 1 mL of 95% ethanol and 9 mL of 0.1 M NaOH. The test tube was covered with an aluminum foil and thoroughly mixed and heated for 10 min in a water bath to gelatinize the starch. The suspension was cooled and then diluted to 10 times its volume. About 0.5 mL of the extract was used in the analysis to which 0.1 mL acetic acid followed by 0.2 mL of iodine in CCl₄ was added. The solution was made up to upto 10 mL with water and kept for 20 min for the color development. The absorbance of the solution was read at 620nm⁹. The amylose content determination was carried out using a colorimetric iodine affinity procedure.

Bio-plastic preparation

The bio-plastics were made using the

starch of cassava and elephant foot yam. About 2 g of the cassava starch was taken in a beaker with 10 mL of the distilled water and stirred well to which 1.5 mL acetic acid and 0.5 mL glycerol¹⁰ was added. The mixture was heated in a Bunsen burner with continuous stirring until the formation of a white colloidal gel. The gel was poured as a flat sheet dried at room temperature for about 48 h which later developed as a white film. The bio-plastic from elephant foot yam was also developed in the similar method using agar-agar and glycerol as the plasticizers.

Water Absorption Capacity

The samples were dried in the oven for about 24 h at 50°C, cooled in a desiccator and weighed. The water absorption test was done for one hour of immersion after which the samples were reweighed¹¹. The water uptake capacity of the film was calculated using Equation 4.

$$\text{Water Uptake} = \frac{\text{Wet weight} - \text{Dry weight}}{\text{Wet weight}} \times 100 \quad (4)$$

Biodegradability test

The biodegradable plastics produced were cut into 1×1 cm and buried in a small rectangular hole in a compost soil at a depth of 12 cm for a period of two weeks. The degradation of the film was monitored at regular intervals. The weight of the samples before (W_0) and after burial (W_1) in the compost soil were noted and the weight loss of the samples were calculated¹² using Equation 5.

$$\% \text{ Weight loss} = \frac{W_0 - W_1}{W_0} \times 100 \quad (5)$$

Application of bio-plastics as packaging for liquid

The application test was conducted using groundnut oil and distilled water¹³ as the test samples. The bio-plastics were cut into 1cm x1cm, initial weight (W_1) was taken and immersed with the test sample in a beaker. The beaker was covered and kept at room temperature for 120 minutes. After the incubation time was complete the sample was cleansed and weighed to obtain the final weight (W_2). The percentage weight change of the bio-plastics was calculated as described in equation 6. If the weight change of bio-plastic after being immersed in the test food products is less than 10% (W_2/W_1), then

the bio-plastics can be considered to be compatible with those test foods.

$$\% \Delta W = \frac{W_2 - W_1}{W_1} \times 100 \quad (6)$$

Characterization of the Bio-Plastics

The characterization of the bio-plastics was done by different spectroscopic and microscopic techniques .

UV-Visible Spectroscopic study

The bio-plastic was cut into 1cm× 3cm and the wavelength used was in the range of 300-800nm for the characterization¹⁴ of the synthesized bio-plastics. The UV-Vis spectral analysis was done by UV-VIS spectrophotometer.

FT-IR Analysis

The infra-red spectroscopy determines the structure of the molecular components and it is mainly used to find out the functional groups in it. The spectral wavelength range is usually around 4000-400 cm^{-1} . The spectra was obtained by mixing the sample with potassium bromide pellets.

Scanning Electron Microscopy

Scanning electron microscopy is a versatile technique used to collect the information about the topography, morphology, composition and crystallographic information of the sample. In SEM, the electrons are heated at high temperature which emits the electron from the tungsten filament and flows towards the anode. The electron beam¹⁵ used has an energy from the range of few hundred eV to keV.

RESULTS AND DISCUSSION

Starch isolation

The yield of the two starches is shown in Table 1. The yield is appreciable compared with the starches from other sources. Based on the yield, it can be used as a commercial product in packaging industry.

Table 1: Results of physico-chemical properties of *Manihot esculenta* and *Amorphophallus paeonifolius*

No	Parameters	<i>Manihot esculenta</i>	<i>Amorphophallus paeonifolius</i>
1	Appearance/colour	White	Brown
2	Odour	Odourless	Odourless
3	% Yield	82	78.5
4	pH	5.00	7.78
5	Amylose content	0.5	0.22

Table 2: Proximate analysis of *Manihot esculenta* and *Amorphophallus paeonifolius* starch

No	Parameters	<i>Manihot esculenta</i>	<i>Amorphophallus paeonifolius</i>
1	Moisture content	12.18	12.16
2	Ash content	0.11	0.109
3	Water uptake	86 %	79%

Moisture content

The moisture content value of two starches was shown in table in Table 2. The moisture content for the cassava starch and the elephant foot yam was found to be in the range 12-13%¹⁶. The moisture content helps to maintain the water content in slurry. This test used to determine whether the tuber starch can be used as a packaging material to maintain food longevity. *Manihot esculenta* and *Amorphophallus paeonifolius* starches have higher moisture content compared to other tuber starches. The high moisture of these starches is a reflection of the loose granules in them, which confirms that the granules are not tightly associated together.

Ash content

This test helps to determine the type and the amount of minerals present in the sample. The ash content of both the cassava and the elephant foot yam starches was around 0.11%. The ash content indicates the total minerals in the food¹⁷. The mineral and organic salt in the tubers is expressed as the ash content which is the food residue after the combustion process. A maximum ash content of 3% is allowed for the edible tubers.

pH of the Starches

Manihot esculenta and *Amorphophallus paeonifolius* were found to have a pH value of 5.0018 and 7.78. The paste clarity depends upon the pH value, due to the higher pH value, the paste clarity of the tuber starches was also high. The value is given in Table 1. Most of the studies show that starches with the pH range 3-9 can be used in pharmaceutical and food industries.

Amylose content

The amylose content of starch influences its properties like water binding capacity, thickening, gelling and film forming properties. The high amylose content of the starch will affect its the pasting properties¹⁹. *Manihot esculenta* and *Amorphophallus paeonifolius* were found to have a low amylose

content value of 0.5% and 0.22%²⁰ which will not affect their film forming property.

Water Absorption Capacity

The water absorbing capacity is very important to determine the water absorptivity of the sample. For the cassava and the elephant foot yam samples it was found to 86% and 79% and the high uptake of water may be due to the presence of three hydroxyl groups in the glycerol²¹ molecule. The degree of water absorption is also related to its amylose content. If the water holding capacity is high it improves the ability of the starch granules to expand in volume without collapsing.

Biodegradability test

The biodegradability test was done using soil burial method. The film of the biopolymer was monitored regularly and it was found that after two days micro-organisms were formed on the film after which it started degrading. After a week the film began to change physically where cracks were observed on the surface of the film associated with a weight loss²². This test shows that the bio-plastics synthesized were eco-friendly in nature.

Application of bio-plastic as a packaging material for liquids

The presence of non-starch components impact the compatibility of bio-plastic food products by delaying the penetration of the vegetable oil into the bio-plastics. In the present study the bio-plastic is made with addition of glycerol. Increased concentration of glycerol in the bio-plastics increase the penetration of the vegetable oil to its surface. Here vegetable oil penetration is low because glycerol is used in a minimal volume. Table 3 shows that that the penetration of the vegetable oil is low compared to the penetration of water in the synthesized bio-polymers. This indicates that the bio-plastic of *Manihot esculenta* and *Amorphophallus paeonifolius* can be used as a packaging material²³ for vegetable oils.

Table 3: The weight change of the bio-plastics after being immersed in test samples for 2 hours

No	Test samples	<i>Manihot esculenta</i>	<i>Amorphophallus paeonifolius</i>
1	Distilled water	87.11	55
2	Vegetable oil	1.26	0.97

Characterization of Bio-Plastics

UV-Visible spectroscopy

The UV absorption has significant relationship with the degradation of the plastics. The plastic which does not absorb radiation is not susceptible to photo degradation²⁴. The ability of the bio-plastics to absorb UV radiation was beneficial when the bio-plastic was used in food packaging. Fig. 1 and 2 shows the UV spectrogram of the bio-plastics.

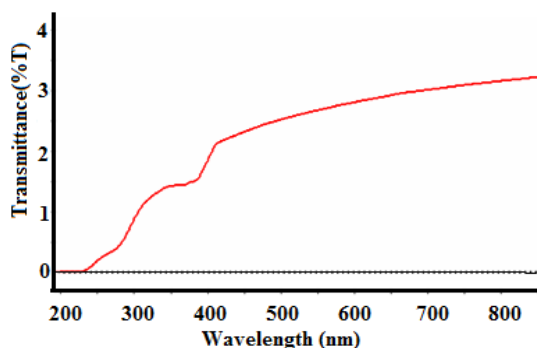


Fig. 1.

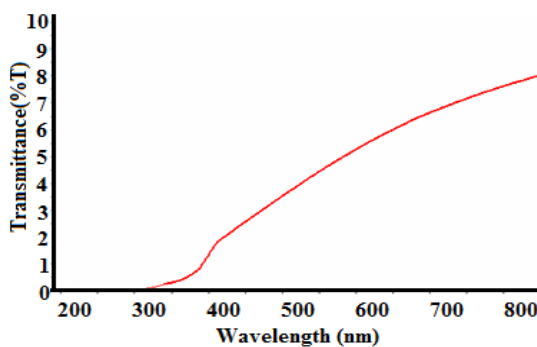


Fig. 2.

Fig.1,2. UV-Visible spectrum of the developed bio-plastic from *Manihot esculenta* and *Amorphophallus paeonifolius*

Manihot esculenta (cassava) absorbed UV with maximum absorptivity in the wavelength from 350-420 nm²⁵ which is similar to the polycarbonate, one of the common conventional plastics, whereas no such characteristic peak was obtained for *Amorphophallus paeonifolius* (elephant foot yam). The bio-plastic obtained from Cassava can protect food products from UV radiation which further prevents their photo-oxidative degradation.

FT-IR spectroscopy

The FT-IR analysis was done to determine the functional groups present in the film. The FT-IR spectrum of the film showed hydrogen bond stretching

at 3543.59 cm⁻¹ and the peak at 2928.40 cm⁻¹ corresponds to C-H stretching²⁶, the characteristic peak at 1246.09 cm⁻¹ indicates C-O bending of C-O-H group and N=C=N stretching at 2147.58 cm⁻¹ indicated the presence of the cyano group, naturally occurring *Manihot esculenta* contains cyanide content, when it is raw. If it is cooked, it will be negligible. The FT-IR spectrum of *Amorphophallus paeonifolius* film, shows the characteristic peaks at 3596.13 cm⁻¹ for the hydroxyl group), 2147.77 cm⁻¹ for strong N=C=N stretching, 1699.25 cm⁻¹ for C=C stretching²⁷ (alkene) and 1468.33 cm⁻¹ for the C-H bending.

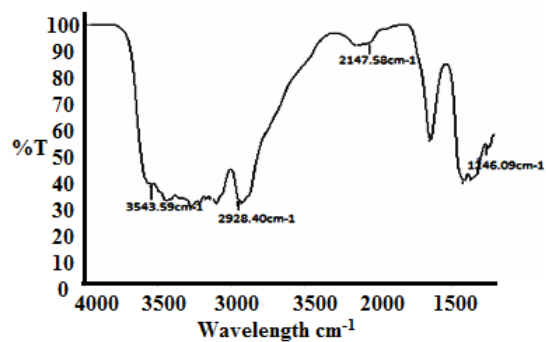


Fig. 3.

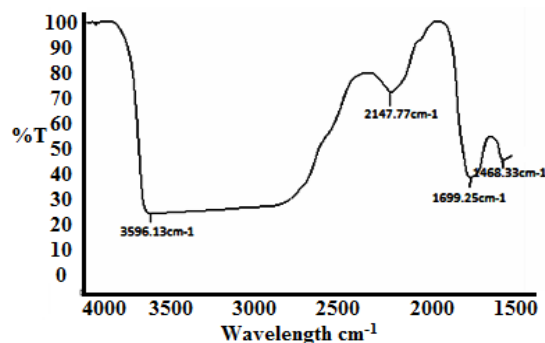


Fig. 4.

Fig. 3, 4. FT-IR spectrum of developed bio-plastic from *Manihot esculenta* and *Amorphophallus paeonifolius*

Scanning electron microscope:

SEM analysis of bio-plastic was performed using scanning electron microscope. The SEM micrograph shows the morphology of the bio-plastic and Fig. 5 and 6 reveals the homogeneous phase of the developed bio-plastic and spots portion indicates duct particles present on surface. The SEM studies revealed that *Amorphophallus paeonifolius* starch granules are round elliptical in shape with smooth surface.

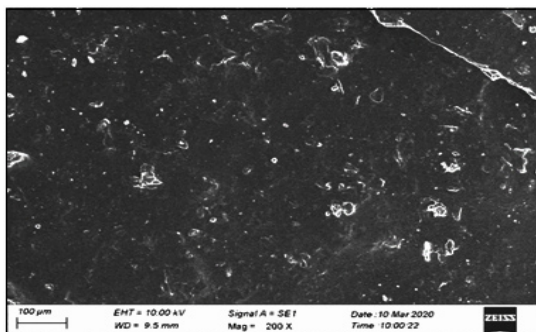


Fig. 5.

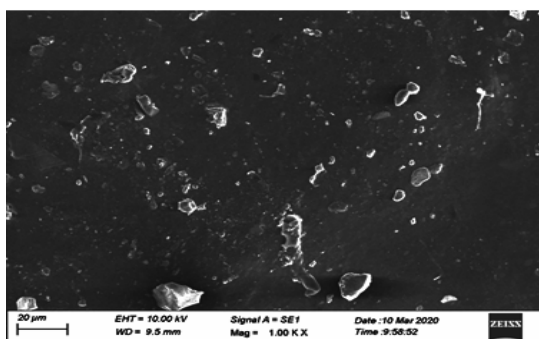


Fig. 6.

Fig. 5, 6. SEM micrograph of the developed bio-plastic *Manihot esculenta* and *Amorphophallus paeonifolius*

CONCLUSION

The future of biodegradable plastics exhibit a great potential since it is eco-friendly,

economically viable and it also reduces plastic waste accumulation. In this study starch based bio-plastics were prepared from *Manihot esculenta* and *Amorphophallus paeonifolius*. These starches had high pH, moisture content and high paste clarity than other tuber starches. Bio-plastic from *Manihot esculenta* can be used as packaging for vegetable oil. These bio-plastics are transparent enabling to view the product easily. This bio-plastic also protects the product from photo oxidative degradation.

The study of *Amorphophallus paeonifolius* starch properties contributes to a better understanding of the textural properties of the traditional yam products. The characterization studies and the physico-chemical parameters reveal that these bio-plastics can serve as a good substitute for the conventional plastics which pollute the environment for a long run.

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Conflict of Interest

The authors declare no conflict of interest.

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