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Development of sweet potato starch-based thin films to produce biodegradable packaging materials

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Abstract - **Starch-based bioplastics are widely used due to their renewability, sustainability, and cost-effectiveness. On the other hand, native starch films have some barriers to creating good and effective products because of their excessive water absorption and weak mechanical characteristics. In this study it was developed an acid hydrolyzed, sweet potato starch-based thin film as a biodegradable packaging material, and the physiochemical properties of the acid hydrolyzed thin films were also investigated. The sweet potato starch was extracted, and acid hydrolyzed by HCl for 30, 60, and 90 minutes. The results of Fourier Transform Infrared spectrometry (FTIR) show that increasing the acid reaction time considerably reduces water absorption and that the best acid reaction time is resolved at 60 minutes. Films were prepared through gelatinization, using glycerin as a plasticizer. The thin films were characterized through FTIR, X-ray diffraction (XRD), and thermogravimetric analysis (TGA) tests. The FTIR test results indicate that in acid reaction, moisture absorbance decreases. The water absorption test results showed that after 24 hours, the acid hydrolyzed sweet potato starch films have lower water absorption than the native sweet potato starch thin films, with 26. 39% and 46.88%, respectively. The acid hydrolyzed film will increase the strength to 5.7 MPa and reduce elongation. It is conclusive that acid hydrolyzed starch-based thin films have increased tensile strength and durability due to their lower water absorption. Further, these films are introduced as non-toxic, low-cost, and biodegradable materials for packaging applications.**

Keywords - Sweet potato starch, acid hydrolysis, biodegradable packaging, bioplastic, water absorption

I. INTRODUCTION

Petroleum-based packaging applications are one of the biggest challenges to the environment. Therefore, as an alternative to this, starch-based bioplastics have been widely used because of their renewability, sustainability, and cost-effectiveness. Native starches have some barriers to making good efficient products because of their poor mechanical properties and high-water consumption [1]. As a result, starch modification is important for producing efficient films. Acid modification has a significant impact on the physicochemical properties of starch [2]. Sakkara et al. discovered that acidic pH reduces the moisture content and the water solubility of maize starch films [3]. Acid hydrolyzed pea starch was prepared which enhanced tensile strength while reducing water vapor permeability [5]. Previous research has revealed the positive results of the acid hydrolysis process on water absorption in various starch-based thin films and their thermal and mechanical properties. Studies have done those acid hydrolysis processes with changes in different acid concentrations, acid hydrolysis temperatures, and different stirring times. However, none of these investigations focused on the effect of acid hydrolysis on the thermomechanical characteristics and water absorption of sweet potato starch thin films. Therefore, in this research acid hydrolysis process was followed for sweet potato starch-based thin films, and chemical properties, thermal and mechanical properties were investigated. This research aims to discover the effective acid hydrolysis time and use the best hydrolysis sample to develop the sweet potato starch-based thin films and discover the thin film's physiochemical properties for use in packaging applications.

II. MATERIALS AND METHOD

The following processes were done when developing the sweet potato starch-based thin films.

A. Starch extraction

Sweet potatoes were peeled off and cut into 2-3 mm thickness and blended with water and using Nylon cloth the starch slurries were filtered. After that, before removing the upper layer of water, filtered samples were allowed for 6 hours to sediment. The wet starch was allowed to dry for 24 hours at room temperature.

B. Acid hydrolysis process

5 g sweet potato starch and 50 ml HCl (2.2 M) were mixed. After that solution was stirred for 30 minutes, 60 minutes, and 90 minutes in different periods at 200 rpm at 45°C. Also using 1 M NaOH solution the acid-treated solution was neutralized. Finally, acid-treated starch was washed with distilled water using a vacuum filter, starch was filtered out.

C. Preparation of sweet potato starch-based thin films

Initially, 9.5 g of native starch and best acid-modified starch was mixed with 60 ml of distilled water separately. After that 10 ml of 5% acetic acid was mixed and 5 ml of glycerin was added. Finally, both solutions were stirred at 25°C, at 200 rpm for 10 minutes. Then both samples were stirred at 70°C, at 500 rpm for 45 minutes to complete the gelatinization process. After that, solutions were poured out into the Petri dishes, and samples were kept at room temperature to clear out the air bubbles. After that, samples were kept for six hours at 65ºC in an air-circulating oven. Finally, for moisture absorption, the samples were placed in a desiccator, and the sweet potato starch-based films from the Petri dish were removed.

Fig. 1. Prepared acid hydrolyzed thin film

D. Characterization test and Mechanical test

1) FT-IR spectroscopy Test

Characteristic peaks were identified in the native and modified starches and films using FTIR Spectrometer (PerkinElmer, USA). The device was configured with a 2.0 cm^{-1} resolution and a 500 – 4500 cm^{-1} scanning range.

2) X-ray diffraction (XRD) Test

The X-ray diffraction test for sweet potato starch was done using an X-ray diffractometer (Rigaku, Japan), with a scan speed of 2°/min.

3) Thermogravimetric analysis (TGA) Test

The film's moisture and thermogravimetric temperature were evaluated using thermogravimetric analysis. In a nitrogen atmosphere, the TGA (TA Instruments, USA) was used. With 100 µl platinum crucibles, a heating amount of 10 °C/min and heats ranging from 25ºC to 650ºC were used. (2008, Stawski).

4) Mechanical Test

The tensile strength test was carried out using a Universal Testing Machine (Instron Ltd, UK) following ASTM D 882-02 test standards. 25 mm per minute was the crosshead speed setting. Five samples were evaluated.

5) Water absorption test

The ASTM D570-98 standard was used to calculate the water absorption of sweet potato films. First, the sweet potato starch-based film samples were separated into 2×2 cm squares and dried for 24 hours in a 50°C oven before being weighted. After soaking the native and modified film samples in distilled water weight was measured after 24 hours. The film's water absorption was calculated using the equations below.

$$\text{Water absorp.}(\%) \text{ at } 24\text{hrs} = \frac{M_1 - M_0}{M_0} \times 100 \quad (1)$$

Where M_0 represents the initial weight, M_1 represent the final weight after 24 hours.

6) Biodegradability test

The acid hydrolysis and native films were cut into 2×2 cm pieces. After that, for 15 days, the samples were submerged in the soil at 3 cm depth at a temperature of 25ºC. Finally, the reduction of weight was calculated using the following equations:

$$\text{Weight loss}(\%) = \frac{M_1 - M_0}{M_0} \times 100 \quad (2)$$

Where w_0 represents the initial weight and w_1 represent the final weight of the film after-biodegradability test.

III. RESULTS AND DISCUSSION

A. *Fourier transforms infrared spectroscopy (FTIR) Test for starches*

The FTIR spectra of acid hydrolyzed starch and native sweet potato starch were compared, as shown in figure 2. The band at 1648 cm^{-1} indicates the flexion of the O-H of the water which indicates that starch is hygroscopic. This spectrum shows that with an increment of acid hydrolyzed time, the amplitude of the 1648 cm^{-1} bands decreased. Based on the above facts, it is conclusive that acid hydrolysis causes a decrease in water absorption.

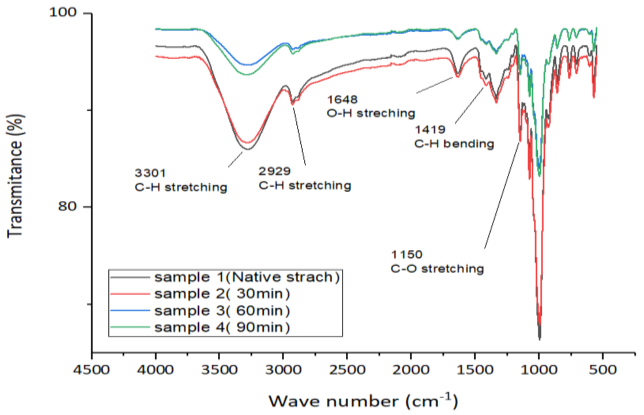

Fig. 2. The FT-IR spectrum for acid modified and native starches in different time

B. *Fourier transforms infrared spectroscopy (FTIR) Test for film*

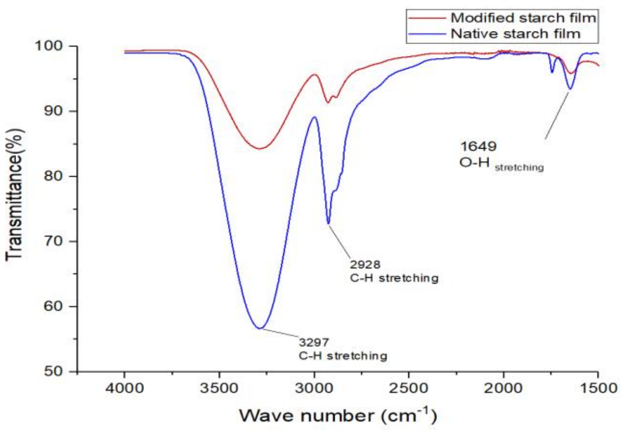

Fig 3: The FT-IR spectrum for acid-modified and native starches-based thin film

According to the FTIR test for the native and modified starch-based thin film, which is shown in figure 3, the O-H bonds were shown in the 1649 cm-1 bands. In that, the acid hydrolysis film has low amplitude as same as the acid hydrolysis starch which was mentioned in figure 2. Therefore, it was concluded that the acid hydrolysis process has decreased the water absorption of the thin film

C. *X-ray diffraction (XRD)*

XRD results are shown in figure 4. According to the results, the peak intensities of the acid-modified film were higher than those of native starch film. Significant variations between acid-modified sweet potato starch-based film and native starch-based film were seen in the X-ray diffraction patterns, peak intensities, and 2θ values [4]. According to the XRD test, the results showed that the acid hydrolysis process boosted the sweet potato starch's crystallinity [5].

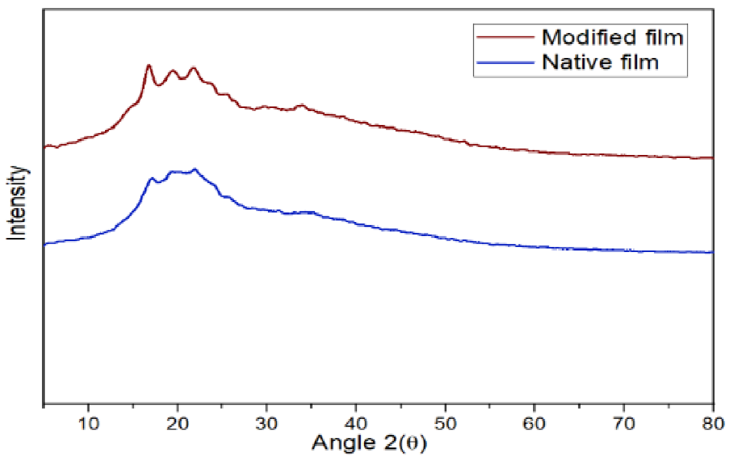

Fig 4: Native and acid-modified sweet potato starch-based thin film X-ray diffraction patterns

D. *Thermogravimetric analysis (TGA) Test*

According to the TGA test result shown in figure 5, the thermal decomposition temperature of the thin film is at around 300°C and mass loss was 39.2 %.

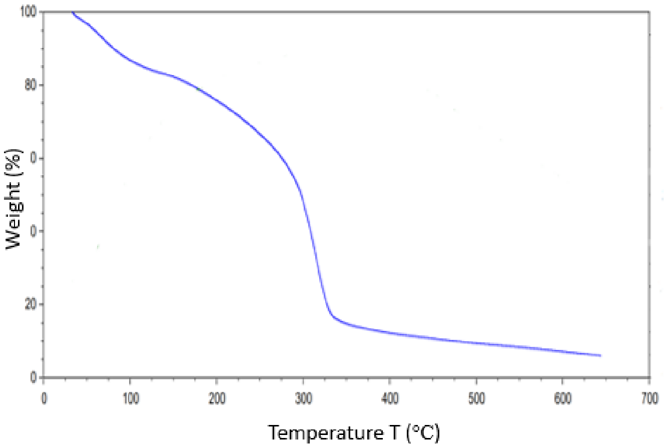

Fig. 4. Thermogravimetric analysis for acid hydrolyzed films

E. Mechanical Test

The tensile strength (TS) was increased in the acid hydrolyzed film than in the native thin film 5.72 MPa and 4.25 MPa, respectively. Therefore, the acid hydrolysis process helps increase sweet potato starch's crystallinity. While increasing the crystallinity, the amorphous area is reduced. According to that the elongation at break will limit and tensile strength will increase [5].

F. Water absorption Test

Starch is soluble in water, and therefore, industrial applications are limited. Therefore, the product prepared from starch is critical for promoting biodegradability but for industrial applications, the starch-based products must be water refused. However, the biobased product must absorb the water to show biodegradability because most microorganisms lived in high moisture environments [6]. According to the water absorption results, the water absorption percentage of native starch thin film and acid hydrolyzed film was 46.88 % and 26.39 % respectively for 24 hours. According to the water absorption test result, it was proved that the acid hydrolyzed process reduced the water absorption level.

G. Biodegradability Test

The factors of pH value, temperature, nutrition, and moisture have a significant impact on how quickly films degrade [7]. According to the biodegradability test results, the weight loss of native thin film and acid hydrolyzed thin film were 50.00 % and 30.88 % respectively. According to those results, the native starch film samples exhibited high degradation profile due to the faster degradation of starch, but acid hydrolysis thin film has a low biodegradation rate. Furthermore, as explained previously the acid hydrolysis film has low water absorption and due to that, acid hydrolysis film samples have low biodegradation rate. However, the results proved that both films were biodegraded. Therefore, acid hydrolysis films are more useful for packaging applications due to their low degradation rates.

IV. Conclusion

The findings of this research demonstrated how acid hydrolysis altered the physiochemical characteristics of thin starch films. According to the FTIR results of native and acid hydrolyzed sweet potato starch, the water absorption decreased as the acid hydrolyzed time increased. Furthermore, water absorption tests revealed that the acid hydrolysis films absorbed less percentage of water. Acid hydrolysis increases tensile strength while decreasing elongation at breaks in the films. The biodegradability test results proved that the acid hydrolyzed film was biodegraded. Also, based on the XRD test, the results were conclusive that, because of the acid hydrolysis process the modified starch-based thin film has increased crystallinity. According to all the outcomes of this research, it was proved that the acid hydrolyzed process can develop physicochemical properties and mechanical properties of sweet potato starch based thin films. According to all the results, the modified films

lead to a lot of industrial applications such as biodegradable film preparation for packaging materials and due to the use of sweet potato starch as raw material can be stated that this film is low-cost, environmentally friendly thin films. Therefore, it will make a significant decline in the pollution caused by non-biodegradable packaging products

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