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Analyze Infrared Heating Characteristics of Potato Starch and Measure its Functional Film Properties

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Abstract

Objectives: To analyze the effect of potato starch infrared heating (IR) and the functional characteristics of the starch film. Methods: Potato starch was heated by IR heater at different time intervals (45 min, 65 min and 85 min) and starch was used to develop polymer films by the addition of glycerol as a plasticizing agent. The surface temperature of starch at different bed thickness was measured using a thermal imaging camera. The temperature of the starch varied from 72.77°C to 80.83°C. The functional properties of developed films were analyzed by their thickness, solubility, contact angle, puncture strength and coefficient of friction with different surfaces. Findings: The results indicate that the puncture strength of IR treated starch film varied from 2.5 to 3.79N and the value was found less in control recorded as 1.87N. The modified starch film thickness and opacity were increased from 0.11 to 0.128mm and 1.15 to 1.22 percent respectively. The solubility percent was found less in IR heated starch than in control. SEM and FTIR analysis results show that IR heat treatment has significant effect on morphology and functional characteristics of the starch film. Novelty: Infrared heating modified the structural characteristics of potato starch and improved the functional properties of the starch film.

Keywords: Starch film; Infrared heating; Functional; Physical; Mechanical

1 Introduction

The utilization of petrochemical-based plastic materials in packaging applications has increased significantly worldwide due to its low cost, lightweight, availability, transparency, and good mechanical properties ⁽¹⁾. Globally, the production of plastic has increased continuously and reached 360 million tons in the year 2019, most of which were used as disposable products. The specific characteristics of plastic material include hydrophobic nature, high molecular weight, and low degradability, make it is difficult for microorganisms to digest and degrade. Thus, the improper disposal of plastics causes damage to the environment.

In the past few decades, the increase in consumer concern on the impacts of non-biodegradable packaging materials towards the environment and interest in eco-friendly, bio-based, biodegradable packaging has resulted in the introduction of synthetic organic polymers or natural polymers obtained from natural sources⁽²⁾. Biopolymers are referred to as polymers that are derived from various living sources that are available in nature. The degradation of this type of polymer occurs naturally by a microorganism that produces specific enzymes that consume organic compounds present in biopolymers as a nutrient source through biochemical mechanisms under specific environmental conditions such as temperature, humidity and pressure. The most commonly used raw materials for developing biodegradable films are polysaccharides (starch, cellulose, and pectin), protein and lipids. Biopolymers are used to develop degradable films because of low cost, availability, compatibility, non-toxic, transparent and continuous film matrix⁽³⁾.

Starch is one of the most abundant and important polysaccharides to prepare polymer films. These films have excellent solubility, transparency and mechanical strength. Among the various starch sources, the corn and potato-based starch have high amylose content, which forms a good film that is stable during aging ⁽⁴⁾. In starch, the presence of amylose and amylopectin plays a vital role in film formation. Potato starch contains 20-25% of amylose, which is essential for film formation. The potato starch particle has low gelatinization temperature, high viscosity and transparency ⁽⁵⁾. The native starch contains 80% of large macromolecule of amylopectin causes limitation in moisture sensitivity and poor mechanical properties ⁽⁶⁾. Therefore, to improve their functional properties, various physical treatments such as microwave heating, conventional heating, and ultrasound have been utilized to modify starch.

Infrared heating is a novel technique used in many processing industries such as electronics, plastic blow molding, drying, dehydration, sterilization and pathogen inactivation. Infrared heating is highly energy efficient and environmentally friendly technology compared with conventional heating. When the starch is heated beyond the gelatinization temperature, granules swell upto many times their original size, collapse and release amylose. The starch which has high amylose is used to form strong and flexible polymer films ⁽⁷⁾. In order to develop a starch films with good functional properties and workability plasticizers such as glycerol and sorbitol are added up to 40% in to the native starch under heat and shear to form thermoplastic starch. The rheological and thermal properties of polymer solution vary with their internal factors and operating conditions ⁽⁸⁾. Thus, considering the advantages of IR heating and to improve the functional characteristics of biopolymer films the present study aimed to analyze the heating characteristics of potato starch and measure the functional properties of films made from IR heated potato starch with plasticizer.

2 Methodology

2.1 Raw materials

The major raw materials used in the study to develop biopolymer film are potato starch procured from M/s Angel Starch and Food Pvt. Ltd., Erode, India and plasticizer (glycerol 99% purity) analytical grade was obtained from M/s Sigma-Aldrich (Mumbai, India). Also, distilled water is used make polymer solution of starch.

2.2 Preparation of starch film

Potato starch was exposed to infrared heating in a prototype setup shown in Figure 2. Starch was taken in the teflon plate at different bed thickness such as 5, 7 and 9mm and heated for 45, 65 and 85min respectively by the IR light placed at 350mm above the product. Thin layer drying methods of starch (0-10mm) increase the drying rate and minimize the drying time $^{(9)}$. The surface temperature of the starch was measured at the end of treatment time using a Thermal Imaging Camera (Model: FLIR T530). About 5g of heat treated potato starch was taken into 250ml beaker then added glycerol 40% (w/w) with 100ml of distilled water. The solution was mixed uniformly and heated at 90°C for 30 min with gentle stirring in a heating mantle until obtain complete gelatinization. The starch solution was transferred to glass trays $(225 \times 150 \times 25 \text{mm})$ to develop biopolymer film using casting method. The samples were placed in dark atmospheric temperature $(32\pm2^{\circ}\text{C})$ until the solution dried completely $^{(10)}$. After 24hrs the developed films were taken from the tray and stored in ambient conditions. The developed starch films are shown in Figure 3.

2.3 Physical properties of film

2.3.1 Thickness

A digital thickness gauge (Model: Insize 2871-10) with an accuracy of ± 0.02 mm was used to determine the thickness of potato starch film. The values were measured at five different positions in each film and the average was recorded. The mean value of thickness was used for further calculation (11).

2.3.2 Opacity

The film opacity was observed using a UV spectrophotometer (Model: UV-1800 Shimadzu, Japan) at 600 nm. Film strips (10×40 mm) were selected and their thickness was measured. Then the film and was placed inside the transparent side of the quartz tubes and the film absorption was noted. The opacity of the film was calculated from equation (1), where A600 is the absorbance value at 600nm and X is the average thickness of the film (12).

$$Opacity = \frac{A600}{x} ...(1)$$

2.3.3 Moisture

The film moisture content was determined with $(10\text{mm}\times30\text{mm})$ samples following the procedure mentioned by $^{(11)}$. Three samples from each film were cut into the desired size and the weight of the initial film (m_o) was noted. Three films of each sample were placed in a hot air oven (Model: Remi RDHO50) at 105° C for 24 h and then the final weight was recorded (m_1) . The moisture content was calculated by using the following equation (2).

Moisture content (%) =
$$\frac{m_0 - m_1}{m_0} \times 100 \dots (2)$$

2.3.4 Solubility

The solubility of the film was measured by using the previously dried films as initial weight (W_{ini}). The dried starch films were soaked in 50 ml of distilled water in a beaker sealed and kept in ambient condition for 24h. Then, the residues were filtered from the beaker, dried at 105°C for 24 h and reweighted (W_{fin})⁽¹⁰⁾. The solubility of the film was calculated by using the following equation (3)

Water solubility (%) =
$$\frac{W_{initial} - W_{final}}{W_{initial}} \times 100$$
 ...(3)

2.4 Functional properties of film

2.4.1 SEM analysis

The microstructure of developed films was analyzed using a scanning electron microscope, SEM images (JEOL, model no.JSM-IT200) using an accelerating voltage of 20 kV. Sample was mounted on aluminum stubs, coated with the thin gold plasma layer. High resolution images were taken on the surface of all the films (11)

2.4.2 Fourier transform infrared spectroscopy (FTIR analysis

The FTIR test was performed by the spectrometer (Model: Nicolet iS50 FTIR Spectrometer). The preparation of the specimen was done by KBr-disk technique. The spectrum was read by an average of 32 scans in $4000-500 \text{ cm}^{-1}$ (13). The test analyzed the structural links and functional groups of developed films

2.4.3 Contact angle

The contact angle of water on the surface of the film was measured using the sessile drop technique where the solid/liquid/air phases in contact. A droplet of distilled water (10μ l) was placed on the developed film surface by using a micropipette. Then, the image of droplet size on the film was captured using a camera (Model DSC-HX400) and the angle was calculated using the ImageJ software. The angle measurement was done between the baseline of the drop and the tangent at the drop boundary. The contact angle on both air sides of the droplet was recorded and averaged (14).

2.4.4 Puncture load

The puncture load of potato starch films was measured using an S430 puncture probe in a single column universal testing machine (Model: Tinius Olsen L-series-H5KL) equipped with a 1KN load cell. The film preparation for the study was carried out as reported by $^{(11)}$. The filmstrip (100mm×100mm) (LxW) were fixed in the sample holding setup using two flat clamps with a grip separation height of 300mm. The peak force was calculated by running the instrument, and the puncture property of each film was calculated using the average thickness value of the sample.

2.5 Statistical analysis

Each experiment was conducted in triplicates and the average was taken for data analysis (mean \pm standard deviation). SPSS software version 20.0 was used for One-way ANOVA using Turkey's range test.

3 Results and discussion

3.1 Heating characteristics of starch

The effect of infrared heating on the rise in temperature of potato starch at different time intervals and bed thickness is given in Figure 1. The maximum, minimum and average temperature on the surface of starch has been measured using the thermal image camera. From the table it is observed that the temperature of the potato starch increases with increasing the heating time and bed thicknesses. The maximum temperature of potato starch at 5, 7, and 9mm bed thickness heated at 85 min exposure were measured as 72.77, 77.47 and 80.83°C respectively. The increase of heating time from 10min to 45min increase the smoothness and depolymerisation of starch biopolymers (15). The drying temperature and mass of the materials is interfering the functional and technological characteristics of starch. The thermal image pictures show that heat dissipation on the starch surfaces. Infrared heating occurs due to IR waves action that penetrates the solid particles and changes in molecular vibrational create heating. The highest surface temperature was observed in 7 and 9mm bed thickness is due to high absorption of heat content, mass and moisture content (8). The moisture content of the IR heated starch is varied from 4.34 to 7.7 percent. In general, the radiation effects highly influenced by the source and environment. The higher mass of the materials emits high radiation energy. The bed thickness and heating time have (p<0.05) significant changes in the temperature of potato starch. The emissivity of a material is the relative ability of its surface to emit energy by radiation. In general, the black material has an emissivity closer to 1.0.

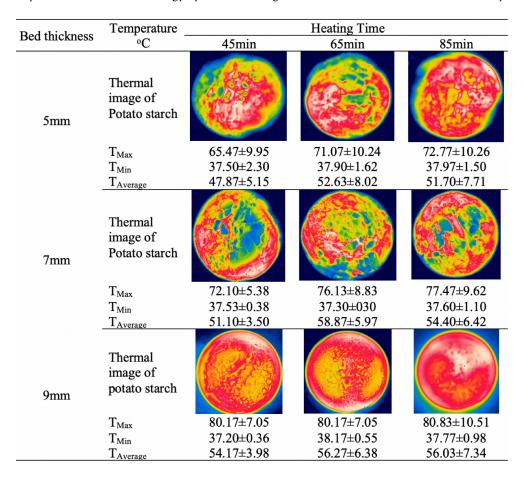


Fig 1. Heating characteristics and thermal image of IR heated potato starch

3.2 Physical properties of starch film

The physical properties of the developed films are depicted in Table 1. Film thickness impacts the film properties such as density, opacity, tensile and tearing strength. The thickness of the control and IR heated (45, 65 and 85min) starch-based films was measured as 0.104, 0.11, 0.126 and 0.142mm, respectively. The results showed that the control film had the lowest thickness

value compared to other films. ANOVA results showed that the films prepared from IR treated (85 min) starch had a significant (p<0.05) effect on the thickness of the film as compared to control. The film's thickness depends on the internal characteristics of film forming solution and formation condition. The increase in viscosity increases the thickness of the film $^{(4)}$.

The opacity of the film is an essential criterion in selecting packaging materials. The average value of the film opacity is given in Table 1. The opacity of the films was found in between 1.11 to 1.22 and the higher value observed for modified starch based film treated for 65 min. The values show decrease in the opacity of films. However, the ANOVA result showed that modified starches opacity was not significantly different (p>0.05) than the control film. Starch granule in potato starch upon heating may break down and form complexes with glycerol and makes the paste less clear and the film developed from this shows less transparency (16).

Table 1. Physical and functional characteristics of potato starch film

Sample	Film thickness (mm)	Moisture (%)	Solubility (%)	Opacity (%)	Contact angle (°)
Control	$0.104{\pm}0.015^a$	26.34±0.25 ^a	34.31 ± 0.05^{a}	1.11±0.03 ^a	36.41±1.27 ^a
IR treated 45 min	0.11 ± 0.019^a	$25.19{\pm}0.17^{a}$	$32.56{\pm}0.17^a$	$1.15{\pm}0.2^{a}$	$48.60{\pm}1.87^{\mathrm{b}}$
IR treated 65 min	$0.126{\pm}0.23^{ab}$	$24.77{\pm}0.09^a$	$31.65{\pm}0.05^{ab}$	$1.22{\pm}0.04^a$	$60.05{\pm}2.41^{\mathrm{b}}$
IR treated 85 min	$0.128{\pm}0.02^{\mathrm{b}}$	$24.53{\pm}0.11^a$	$30.44{\pm}0.23^{b}$	$1.22{\pm}0.04^a$	72.01 ± 4.21^{b}

Values are represented as mean \pm standard deviation; Means at the same column with different letters are significantly different (p<0.05)

The moisture content of biopolymer film is important in maintaining film characteristics and strength. Starch films are hygroscopic in nature and the moisture content of developed film are shown in Table 1. The moisture content of native starch film (control) and modified starch film (IR heated for 45, 65 and 85min) were measured as 26.34, 25.19, 24.77 and 24.53 percent respectively. It was observed that the moisture content was decreased as the exposure time of heating increased, and the IR treatment had a significant (p<0.05) effect on the moisture content of films. The changes may be due to modification of starch molecules due to heat treatment made the film structure hard and not to observe the moisture from the surroundings reported by $^{(17)}$. The moisture content wheat starch films at 33% and 50% glycerol content were measured as 64.1 and 80.8gwater/100g-1(18)

The starch based films are water sensitive, modified as water resistance to enhance their application in packaging. The films solubility favors specific applications such as encapsulation or edible coating $^{(4)}$. Table 1 shows the solubility of films developed from native and modified starches. The solubility of the control sample was measured as 34.31% and it was decreased (30.44%) as the IR heating time increased to 85min. The decrease in solubility of modified starch is mainly due to intermolecular chain of amylose content. However, several factors, such as the origin of starch, glycerol concentration, and structural modification of starches, influence the solubility of the film $^{(18)}$

3.3 Functional characteristics of starch film

3.3.1 SEM Analysis

The morphological characteristics of biopolymer film developed from control and IR heated starch with plasticizer was studied. It is observed that all the films were transparent and homogeneous. SEM analysis shows microstructure arrangements, pore distribution, homogeneity and surface smoothness ⁽¹²⁾. The micrograph of films prepared from native and IR treated (85 min) potato starch are shown in Figure 4 (a) and (b). Figure 4 a IR heated starch shows a smooth surface with some non-dissolved starch particle that reflects the morphology of potato starch. The microstructure of native starch film shown in Figure 4b indicates some starch granule on the film surface. It suggests that IR heating support homogenization of solution, starch pasting and gelatinization behavior. Starch films morphological characteristics depend on the plasticizer types used and their concentration. Accordingly, the highest proportion of plasticizers used in the starch film is about 60% ⁽¹⁹⁾

3.3.2 Contact angle

The contact angle is the quantitative measurement used to characterize potato starch film surface properties. It is an indicator for measuring the degree of hydrophilicity or hydrophobicity. The contact angle of starch films was measured by using the sessile drop method and the angles were measured by using ImageJ software and the results were mentioned in Table 1 and the droplets on the surface of the film are shown in Figure 5. It is observed that films developed from modified starch had a higher contact angle value 65.67° than native starch (control) film 36.41°. The modified starch films showed low wetting properties. If the water contact angle of polymer film more than 90° is hydrophobic and when the contact angle is less than 90° is hydrophilic (20). The

values obtained from the films developed from modified starch showed statistically significant (p<0.05) compared to control. It may be due to amylose and amylopectin content, degree of polymerization and granule size.

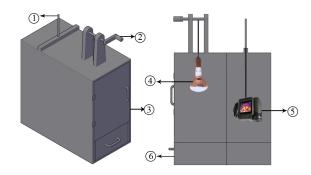


Fig 2. IR Heating Experimental setup (1.Screw lever, 2. IR lamp height control, 3. Heating chamber, 4. IR lamp 250w, 5. Thermal camera, 6. Starch holding tray)

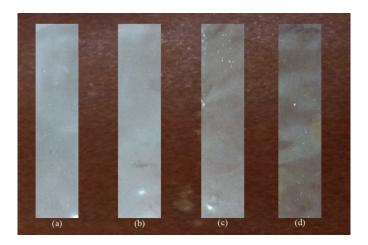


Fig 3. Biopolymer film developed from modified potato starch (a)IR 85 min, (b)IR 65min (c) IR 45min and (d) Control

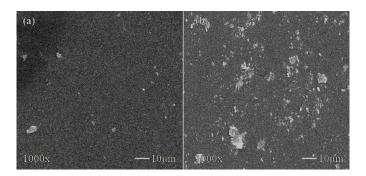


Fig 4. SEM micrograph of potato starch films (a) IR heated (85 min) (b) Control

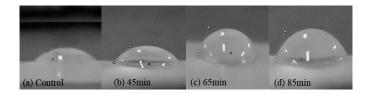


Fig 5. Contact angle of control and IR heated modified potato starch film3

3.3.3 FT-IR

The functional groups of native starch and IR-treated modified starch-based films are shown in Figure 6. The change in bands of the spectra reflected the chemical interactions when two or more components were added together. The strong and broad signal has appeared at 3265.92 to 3266.26 cm⁻¹ all film samples revealed O-H stretching and inter and intramolecular hydrogen bonds. The wave number of O-H stretching shifted from 3265.92 cm⁻¹ in control to higher wave number 3265.97, 3266.26 and 3266.26 cm⁻¹ due to IR heating. The increase in temperature increased the hydrogen bond formation between starch and the polymers but the differences were not significant. In general, all the spectra of modified starch composites film showed the same pattern of bands. From the results, it is observed that starch is neither chemically affected nor modified by the influence of glycerol but the effect of heating has a significant difference on O-H stretching. Similarly, the wavelength at 2926.90 to 2829.30 cm⁻¹ reflects asymmetric and symmetric stretching of C-H represents aliphatic groups from the hydrocarbon backbone of starch and glycerol-starch. This is because the IR heating time increases the C-H stretching. The film developed from IR treated potato starch with the addition of glycerol represented by C=O stretch shifting of the peak at 1646.68 to 1646.61 cm⁻¹ for control at 1646.78 cm⁻¹ showed water molecules adsorbed in the amorphous region and stretching vibration of compound class with weak amide $^{(21,22)}$. Finally, the large ring C-O stretching was observed at 995.60 to 996.15 cm $^{-1}$ for IR treated potato starch film and control was measured as 996.38 cm⁻¹. Similar bands between 1400 and 900 cm⁻¹ were found for native starch and thermos plastic starch material where C-O vibrations of alcohol or ether groups from glycerol and carbohydrate starch in the matrix are involved. Compared with native starch film, the IR treated starch film has a reduction in peak which shows preheating reduces the intensities of peak (23).

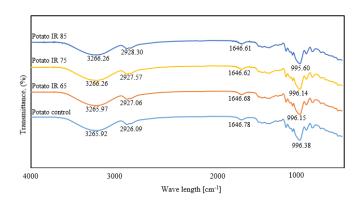


Fig 6. FTIR spectra of control and IR heated potato starch film

3.3.4 Puncture strength

The puncture load of the control and IR heated potato starch films is shown in Figure 7. The control film showed a lower puncture strength value of 1.87N than the modified starch based films, which were found to be 2.5, 2.55 and 3.79N for IR 45, IR 65 and IR 85 min respectively. Analyze the puncture strength used to measure the puncture and rupture characteristics of the polymer film. The starch films developed from IR treated potato starch had a significant (p>0.05) effect on the puncture strength of the film as compared to control. The strain was found high in IR 85min treated starch (13.7%) followed by IR 65 and IR 45 min recorded as 13.2 and 14.0% respectively. The strain of starch increased with puncture load. The heating time modified the functional characteristics of starch and increased the strain of starch film. During heating, amylose in the potato starch leaches out and forms the amylose-lipid complex with semi crystalline and lamellar structure, thus improving films tensile and puncture strength (24).

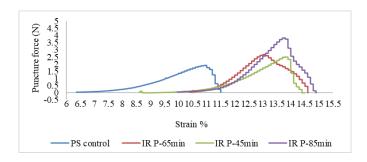


Fig 7. Puncture strength of IR heated potato starch films

4 Conclusion

This study used infrared heated potato starch to develop a biopolymer film. The opacity increased 10 percent as the heating time increased to 65min. Similarly, the thickness of the film increased to 18 percent compared with control. The force required for puncture or rupture was found high in IR heated starch 3.79N than control 2.5N. FTIR peaks showed that the pre heat treatment with IR has modified the functional group of starch molecules. Morphological structure results showed IR heated starch based film has homogeneous smooth surface and less moisture content 24.53% which is evidence of their network integrity. Thus, IR heat treatment has significant improvement in the functionality of biopolymer film compared with native starch film. Taken together, this study may offer significant insights for future research especially on optimization of IR heating levels and plasticizers interaction in development of reinforcement polymer films

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