



Synthesis and characterization of bioplastic film from banana (*Musa Cavendish* species) peel starch blending with banana pseudo-stem cellulosic fiber

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Abstract

The exponential growth of the human population has led to the accumulation of non-degradable waste materials across our planet. Bioplastics synthesized from only starch have less mechanical strength and thermal stability and can be improved by the addition of fillers. In this study, banana peel starch and banana pseudo-stem fiber was used for the synthesis of bioplastic film. The optimum process variables for starch yield were 27.66 min and 37.92 °C temperature, which gives a 12.06% maximum yield. The extracted starch was characterized as pH (6.31), moisture (7.2%), loss on drying (11.2%), amylose (19.3%), amylopectin (80.7%), and ash content (0.8%). For fiber density (1.43 g/cm³), yield (8%), moisture content (61.3%), water absorptions (4.6%), and cellulose (55–60%) have been investigated. The cellulosic fiber was employed as reinforcing material to improve the produced film physicochemical and mechanical characteristics. The film was synthesized by conducting 20 experiments and analyzed by design expert 11 by considering three factors: dry oven temperature(35–65 °C), glycerol concentration (20–50%)w/v, and fiber concentration (5–20%) w/w of starch (5 g) basis. Three responses were tensile strength (TS), water absorption (WA), and elongation at the break (EA) of the synthesized bioplastic film. The result obtained in ranges were 0.2–7.25 Mpa TS, 4.9–31.01% EA, and 18.6–35.3% WA. The obtained results at the optimal point were 7.23 Mpa TS, 5.46% EA, and 18.60% WA at a combination of 56.5 °C dry oven temperature, 28.6% glycerol, and 18.26% fiber concentrations. The film is also characterized by TGA, DSC, and FTIR. This study indicates the incorporation of cellulosic fiber as a filler enhances the tensile strength, lowers water absorbent, and improves the thermal stability of starch-based bioplastic film.

Keywords Banana peel starch · Banana pseudo-stem cellulosic fiber · Bioplastic film · Bio polymers · DSC · TGA · FTIR

Abbreviations

ANOVA	Analysis of variance
BIO-PE	Bio-polyethylene
BIO-PVC	Bio-polyvinyl chloride
C ₆ H ₁₀ O ₅	Starch
C ₆ H ₁₂ O ₆	Glucose
CCD	Central composite design
CV	Coefficient of variation

D.oven tem	Dry oven temperature
DSC	Digital scanning calorimetry
EA	Elongation at the break
FB	Fiber
FTIR	Fourier-transformed infrared ray spectroscopy
GHG	Greenhouse gasses
Gly. coc	Glycerol concentration
JIT	Jimma Institute of Technology
NaOH	Sodium hydroxide
PRESS	Predicted residual error sum of squares
RSM	Response surface method
SNNP	Southern nation, nationalities, and peoples
Tc	Crystal temperature
Td	Decomposition temperature
Tg	Glass transition temperature
TGA	Thermogravimetric analysis
Tm	Melting temperature

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TS Tensile strength
WA Water absorption

1 Introduction

1.1 Background

Polymers are compounds with large molecular mass, built of interlinking, basic building blocks called monomers. When those polymers bunch together, they form plastics. Plastics are relatively low cost, durable, and versatile materials. Plastics have brought some importance to society in terms of economic activity, jobs, and quality of life. However, plastic wastes also impose negative environmental impacts. It is usually non-biodegradable and, therefore, can remain as waste disposed of in the environment for a very long time; it may pose risks to human health as well as the environment, and it can be difficult to reuse and/or recycle in practice [1]. Most polymers are originated from renewable resources which are biomass [2]. Biomass has the potential to reduce greenhouse gas (GHG) emissions by replacing fossil fuels.

As plastics are dumped into the environment, degradation starts with photodegradation, followed by hydrolysis and thermo-oxidation process. These processes lead to plastic waste breakage into low molecular weight (MW) compounds, which can subsequently be metabolized by microbial activity. A small amount of carbon dioxide is released into the atmosphere during the degradation of biomass-based bioplastics; on the other hand, some petroleum-based conventional plastics emit a large amount of carbon dioxide and methane into the atmosphere during degradation after a long period. Conventional plastics are made from raw materials like natural gas and petroleum oils. Bio-based plastics consist of biodegradable and recycled. Some properties of biomass-based polymers are similar to conventional polymers those derived from fossil; for example, bio-polyvinyl chloride (Bio-PVC) and bio-polyethylene (Bio-PE) originated from sugarcane are almost similar to petroleum and natural gas-based plastics [3]. Biodegradation for biopolymers is breaking into smaller particles with the help of microbial activity and converted into biomass, water, and carbon dioxide, but some conventional plastics are degraded after a long period into methane and carbon dioxide [4–7]. Cellulose, lignin, and starch are commonly available in nature. Cellulose is abundant in most plants, although some plants produce more than others [8]. Wood contains lignin, and starch is commonly found in plants such as corn, potatoes, banana peels, and wheat [9]. Starch is a natural biodegradable polymer that has the potential to substitute synthetic polymers used for limited-time applications [6, 8, 9].

Fiber from the banana plant is comparably good in physical strength and cellulose content relative to fiber obtained from other fibrous commodity by-products, extracted from their fruit stalk, pseudo-stem, and leaves as indicated in the “Result and discussion” section [18, 32]. Fiber obtained from the banana pseudo-stem is highly strong and stable and most of the time used as a rope and other activities in Ethiopia. When compared to other wastes, banana pseudo-stem is highly fibrous and mostly available in Ethiopia. Fiber from bananas is environmentally friendly and an alternative to producing food packaging plastics, dry goods packaging plastics, or other reinforcing fiber utilized in engineering composites [11, 17]. The latest studies have indicated that banana fiber possesses a lot of importance in chemical and physical properties which can be used as a very good raw material for the textile and packaging industry [15]. Approximately 90 million tons of banana fruit are produced, mainly in tropical areas [19]. In Ethiopia, bananas occupy nearly 60% of the harvested area of fruit crops in 2017/18. Ethiopia produces 494,000 metric tons of bananas [20]. It is produced in the Southern Nations, Nationalities, and Peoples (SNNP) and the Oromiya regional states. In the Arba Minch, about 2500 hectares of bananas are produced. Another second production area in the southwestern part of the country is found around Jimma, Sheka, Kaffa, and Bench Maji zones in Southern and Oromiya regional states. In Amhara, Tigray, Benishangul-Gumuz, and Gambella are mainly for domestic production. However, due to irrigation systems, it is produced in Afar and Somali [39]. We have such resources, but our country imports plastic raw materials 100%. Banana pseudo-stem cellulosic fiber also produced from the waste banana stem. Therefore, it is possible to obtain a banana sufficiently, and the banana peel is rich in starch to use for the synthesis of biodegradable bioplastic films. Both banana peel and cellulosic fibers were the main ingredients that are thrown as waste; this study gives a new insight to utilize waste biomass to change into some usable products. This study was very unique by synthesizing a comparable dry good packaging bioplastic film from waste biomass. The physical, mechanical, and thermal property of the produced bioplastic film was improved by the addition of cellulosic fiber as filler. The main objective of this paper is to give a new clue on synthesizing and characterizing bioplastic film from banana peel starch by blending with banana pseudo-stem cellulosic fiber. Both peel and pseudo-stems are thrown as a waste; instead, it is possible to utilize the wastes and convert it into usable products for the purpose of dry goods packaging. To achieve this, the following sub-activities were done.

(1) Determine the proximate composition of the banana peel (to know the availability of starch since starch is the complex form of carbohydrate), extract and characterize starch, and determine the optimum parameters which give

a maximum yield. (2) Extract and characterize fiber from banana pseudo-stem. (3) Optimize the tensile strength, elongation at the break, and water absorption of the synthesized bioplastic film. (4) To make a clear comparison of the bioplastic film with and without cellulosic fiber, the physico-chemical properties like solubility, transparency, thickness, and density for the synthesized plastic film were determined, and the best film was investigated by FTIR, DSC, and TGA.

2 Material and method

2.1 Materials

2.1.1 Raw materials

Banana plant (*Musa Cavendish*) species for both banana pseudo-stem and banana peel were collected randomly from Jimma town. The banana pseudo-stem was used as the raw material to extract the fiber. Distilled water is used to dissolve the fiber and starch. For this article's work, false banana stem cellulosic fiber was used as reinforcement. Starch was extracted from the banana peel. Starch and fiber were the main raw materials to produce bioplastic film.

2.1.2 Chemicals

The major chemicals that were used in this experiment were sodium hydroxide solution (0.3 M NaOH) for the treatment of raw fiber to remove non-cellulosic components of raw fiber; sodium bisulfate used as an antioxidant/preservative; (glycerol) Propan-1, 2, 3-triol as a plasticizer; hydrogen peroxide for bleaching; and NaOH, HCl, H₃BO₄ (boric acid), H₂SO₄ (98%), catalyst tablet VST (cod. A00000277) used for protein determination. For lipid determination, oilseed and solvent (petroleum ether, bp 40–60 °C) were used for fiber determination.

2.2 Processing methods

2.2.1 Proximate analysis of banana peel

Banana (*Musa Cavendish*) fruit peels were washed and allowed to dry at room temperature. Then, fresh peels were dried at 50 °C and then ground to obtain a fine powder [19]. The proximate analysis was carried out using the standard methods of analysis of the association of official analytical chemists. The parameters determined were moisture content, crude protein, crude fat, crude fiber, total ash, and nitrogen-free extract (carbohydrate). A triplicate was done for each parameter. Proximate analysis was done to predict the availability of carbohydrates since it is the main constituent of starch.

Starch is a complex form of carbohydrates. Proximate analysis was done following the standard methods mentioned by AOAC 1990 [22].

Moisture content determination Two grams of fresh samples were placed in a pre-dried and weighed crucible. Oven combustion was used to dry the samples at a temperature of 105 °C until a constant weight was obtained. After removing the samples from the oven, they were allowed to cool for 60 min in a desiccator and then re-weighed. The moisture content was calculated using Eq. (1) as a loss in the weight of the original sample and expressed as a percentage.

$$\text{Moisture content(\%)} = \frac{w_1 - (w_2 - w_0)}{w_1} * 100 \quad (1)$$

where w_0 = container constraint weight; w_1 = fresh sample weight; and w_2 = container and dry sample weight.

Crude protein determination To determine the crude protein content, we used a Kjeltex analyzer to measure the total nitrogen content in the sample. This was then converted to crude protein by using factor 6.25 based on the assumption that the average protein contains about 16% nitrogen. The principle of this analytical technique is similar to the conventional micro-Kjeldahl method and can be divided into three stages: (i) digestion, (ii) distillation, and (iii) titration; 1.0 g of sample was taken for each trial. The protein content (%) of the samples was calculated using the following equation:

$$\text{Nitrogen in sample (\%)} = \frac{w_2 * \text{normally of acid} * 14 / w_1}{w_1} * 100 \quad (2)$$

where w_1 = sample weight measured by milligram and w_2 = volume of HCl.

A converse factor of 6.25 was used to convert total nitrogen to protein for all the waste substrates as follow, Crude protein (%) = Nitrogen (%) × 6.25.

Crude lipid determination Exhaustive Soxhlet extraction was used to determine the crude lipid by petroleum ether on a Soxhlet extraction system. A total amount of 1.0 g of the sample was put into pre-weighed thimbles and covered with a wad of cotton. A Soxhlet extraction cup was weighed, and 100 mL of petroleum ether was added to each sample. Then the sample was boiled for 20 min, rinsed for 35 min, recovered for 10 min, and finally dried for 5.0 min. The extraction cup was removed and placed in an oven for drying at 105 °C. The extraction cup was removed from the oven after 60 min and allowed to cool in desiccators. The lipid was measured by re-weighing the extraction cup, and the crude lipid content was determined using Eq. (3).

$$\text{Crude lipid content (\%)} = \frac{(w_3 - w_1)}{w_2} * 100 \quad (3)$$

where w_1 = container weight; w_2 = sample weight; and w_3 = lipid and container weight.

Total ash determination The porcelain crucible was dried in the oven at 105 °C for 60 min and then weighed after being cooled in the desiccator. As much as 1.0 g of the sample was weighed and placed overnight in a muffle furnace which was set at 550 °C. After washing, the sample was removed from the furnace. The crucible was cooled in a desiccator and then re-weighed. The total ash content was calculated using the following equation:

$$\text{Ash content (\%)} = \frac{(w_2 - w_0)}{w_1} * 100 \quad (4)$$

where w_0 = crucible weight; w_1 = sample weight; and w_2 = crucible and ash sample weight.

Crude fiber determination Triplicate fat-free dried samples of 1.0 g were weighed in a clean pre-weighed filter crucible. The crucible, together with the sample, was transferred to the hot-extraction unit, and the sample was left to digest for 30 min with 150 ml of the solution containing 12.5% sulfuric acid and 0.25 mL of octanol. The condenser was switched off after 30 min and allowed to cool. The acid solution was filtered and washed with hot distilled water by suction. Then, the sample was digested for 30 min with 150 mL alkali solution (12.5% sodium hydroxide) and 0.25 mL of octanol to dissolve alkali-soluble matter from the sample. The porcelain crucibles, together with the final residue, were dried at 105 °C in an oven for 60 min, cooled in a desiccator, and then weighed. The ashing of the residue was ignited in a pre-heated muffle furnace at 550 °C for 180 min. The percent of crude fiber content was calculated using Eq. (5).

$$\text{Crude fiber * (\%)} = \frac{w_2 - w_3}{w_1} * 100 \quad (5)$$

where w_1 = sample weight; w_2 = crucible with residue weight; and w_3 = empty crucible with ash residue weight.

Determination of nitrogen-free extract Nitrogen-free extract (carbohydrate) was determined according to the equation below:

$$\text{NFE} = (100 - (\text{WC\%} + \text{CP\%} + \text{CL\%} + \text{CF\%} + \text{ash\%})) \quad (6)$$

where NFE = nitrogen-free extract; WC = water content; CP: crude protein; CL = crude lipid; and CF = crude fiber

2.3 Methods for the extraction of starch from banana peel

The preparation method of banana peel starch used in this study was a modification of the preparation method of durian seeds starch [23]. The banana peel was weighed and cleaned, cut into small pieces using a knife, and homogenized with distilled water containing sodium bisulfate solution (0.25 g/L) at a proportion of banana peel:distilled water (1:2 w/v). This was done by hand blender machine; 77 g/144 mL (peel: distilled water) was taken for each trial. The mixing was taken out with the aid of a mechanical stirrer for 15–30 min at 25–45 °C temperature. After that, the slurry was filtered through a cotton cloth, and the waste solution was cleaned. The starch suspension was gone away overnight in refrigeration at 6 °C, yielding complete starch sediment, and centrifuged for 15 min at 3000 RPM. The white sediment was then dried at 40 °C in a convective oven for up to 40 h. Then, starch in dried form was triturated with the supporter of a motor pestle to obtain the starch in powder form. The dried starch was grounded and screened through a 0.125-mm mesh sieve. Twelve experiments were done to observe the effect of the main factors (by varying extraction time and temperature by fixing antioxidant concentration). The yield of starch present in the peel of Musa Cavendish banana peel was calculated by using Eq. (7). Twelve experiments were done, and the D-optimal response surface method was used to analyze the response (yield) [23]. A D-optimal design is generated by an iterative search algorithm and seeks to minimize the covariance of the parameter estimates for a specified model. D-optimal designs are straight optimizations based on a chosen optimality criterion and the model that will fit. This model is used to determine the best optimal values. D-optimal designs are described as model-specific designs that address the limitations of traditional designs. It is generated by an iterative search algorithm and used to minimize the covariance of the parameter estimates for a specified model [23].

$$\text{Yield (\%)} = \frac{\text{Mass of starch extracted (g)}}{\text{Mass of peeled (g)}} * 100 \quad (7)$$

2.4 Extraction of banana pseudo-stem fiber

The outermost covers of the banana plant stem were peeled, and brown-green skin was thrown away, retaining the cleaner or white portion, which can process into knotted fibers. Then, the fiber is extracted manually by scraping away the plant tissue using an iron ribbon until the fibers were fully separated. To remove the non-cellulosic component, 5 g of sample was taken and treated with a concentration of 0.3 M

of 200 ml sodium hydroxide at 90 °C treatment temperature and a duration of 1 h. It was washed individually with distilled water and allowed to dry in the atmosphere for 48 h [13]. The yield of fiber present in the stem was calculated by measuring one segment of the stem before extracting fiber and measuring of extracted fiber, and then the two masses were compared [13].

$$\text{Yield (\%)} = \frac{\text{Mass of fiber extracted (g)}}{\text{Mass of stem bundle used (g)}} * 100 \quad (8)$$

2.5 Characterization of the extracted starch from banana peel

The banana peel starch was evaluated qualitatively for its properties including pH, moisture content, ash content, density, amylose content, loss on drying, and amylose content [24].

The presence of starch was identified The appearance of bluish violet color upon the addition of iodine was observed by adding iodine [24].

pH measurement Five grams of starch was mixed with 25 ml of CO₂-free distilled water for 1 min and kept for 15 min. The pH value was subsequently measured using a pH meter. In general, the pH of starch varies between 4 and 7 [24].

Moisture content starch powder Three grams of starch was weighed into a crucible and placed in an oven with a temperature of 105 °C and dried for 24 h to constant weight. Moisture content in the dried starch was determined by keeping the weighed quantity of sample in a thermostat-controlled oven at 105 °C for 24 h. The dry weight of each sample was taken on a weighing balance. The moisture content of starch should be approximately less than 20%, as indicated in the “Result and discussion” section [24].

$$\text{Moisture content(\%)} = \frac{(\text{Initial weight} - \text{Final weight})}{\text{Initial weight}} * 100 \quad (9)$$

Determination of loss on drying One gram of starch was weighed and put into a tarred pre-heated (105 °C for 30 min) weighing bottle with its lid, placed into an oven (105 °C), and dried until a constant weight is achieved [24].

$$\text{Loss on drying(\%)} = \frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} * 100 \quad (10)$$

Determination of ash content The sample was heated at the temperature of 550 °C as such that the organic compound and its derivatives were destroyed and evaporated, yielding mineral elements and organic compound residue. The ash

content should be less than 1%. The ash content was calculated using the following formula [25]:

$$\text{Ash content (\%)} = \frac{(w_2 - w_0)}{w_1} * 100 \quad (11)$$

where w_0 = crucible weight; w_1 = starch weight; and w_2 = crucible and ash sample weight.

Determination of amylose content One hundred milligrams of amylose was dispersed using 1 mL absolute ethanol, and 9 mL 1 M NaOH was added to the solution. The solution was positioned in a boiling water bath for 15 min with frequent shaking to obtain a clear solution; then, the entire volume was adjusted to 100 mL using distilled water to obtain the stock solution.

To generate absorption spectra, a 4 mL amylose stock solution or a 2.6 mL amylopectin stock solution was added to 30 mL distilled water, and the pH was adjusted to 3.6 using 0.5 M HCl. Then, 0.5-mL potassium iodide–iodine (I₂-KI) solution (2 g/L I₂, 20 g/L KI) was added, and the total volume was adjusted to 50 mL using distilled water. This reaction solution was mixed and placed at room temperature for 30 min, after which it was scanned using a spectrophotometer at 600 nm wavelengths. To generate the amylose standard curve, the amylose stock solution was diluted to obtain reaction solutions with final concentrations of 10 µg/mL, 20 µg/mL, 30 µg/mL, 40 µg/mL, 50 µg/mL, 60 µg/mL, and 70 µg/mL, respectively [25]. Based on this amylose standard calibration curve, the maximum value of the amylose that is found in banana peel starch was determined using the following formula [25].

$$\text{Amylose content (\%)} = 3.06 * \text{Absorbance} * 20 \quad (12)$$

$$\text{Amylopectin content (\%)} = 100 - \% \text{Amylose content} \quad (13)$$

The amount of amylose and amylopectin present in starch affects the mechanical property of the starch polymer. High amylose starch presents higher stiffness and hardness than high amylopectin starch due to the comparatively less branching structures [25].

2.6 Characterizations of Banana Pseudo-stem Fiber

The following parameters were determined using the method mentioned in the literature [26].

Moisture content The moisture content of the fiber was measured with the principle of the mass difference between the mass of the fiber before and after drying [26].

$$\text{Moisture content(\%)} = \frac{(\text{Initial weight of fiber} - \text{Final weight of fiber})}{\text{Initial weight of fiber}} * 100 \quad (14)$$

Cellulose and non-cellulose content The cellulose content of raw banana fiber was analyzed by soaking the raw fiber with 0.3 M of 7.5% sodium hydroxide solution for 1 h at a temperature of 90 °C. After the process, soluble components were removed, and the fiber was washed with distilled water to a neutral condition that happened and dried. The mass difference of raw fiber before and after soaking was taken to calculate the cellulose and non-cellulose content of the fiber by gravimetric analysis [26].

$$\text{Cellulose(\%)} = \frac{(\text{weight before soaking} - \text{final weight after soaking})}{\text{weight before soaking}} * 100 \quad (15)$$

Water absorption of banana pseudo-stem fiber The water absorption test was done by soaking grams of dried fiber in water for 24 h, and finally, the soaked fiber was measured. After immersion, the excess water on the surface of the fiber was removed using a filter cloth, and the final weights of the specimens were then taken. The increase in the weight of the fiber was calculated using the following equation [26].

$$\text{Fiber water absorption (\%)} = \frac{\text{Final weight} - \text{Initial weight}}{\text{Initial weight}} * 100 \quad (16)$$

The density of extracted fiber Density was measured using the Archimedes principle by using water as liquid. The mass of extracted fiber was measured and immersed in a measured volume of water, and density was calculated with a mass-over-volume formula [26].

Thermo gravimetric analysis (TGA) The thermal stability of fiber was tested by measuring the mass loss during a heating ramp rate by the thermo gravimetric analyzer (TGA instrument) using temperature programming from 25 to 600 °C at the heating rate of 20 °C/min to which the sample was heated. The thermal degradation onset temperature and the thermal degradation weight loss of the fiber were recorded and analyzed [33].

Synthesis of bioplastic from the extracted starch via fiber The sample was prepared by the procedure adapted from the method described in the literature [39, 40]. A film-forming dispersion was prepared by mixing the starch (5 g) extracted from banana peel and distilled water (100 ml). The dispersion was stirred manually on the shaker water bath set at 75 °C for 20 min while stirring at the same rate until it becomes gelatinized. Then glycerol was added at different concentrations (20%, 35%, and 50%) w/v starch and 100-ml distilled water basis. Cellulosic fiber extracted from the banana pseudo-stem was then added at 5%, 12.5%, and 20% w/w based on a starch weight basis. Each mixture was stirred for homogeneity to make the gelatin very strong and then allowed to cool to 50 °C before being cast on a Petri dish.

Film-forming solutions (40 mL) still in hot were transferred into casting in glass Petri dishes having 15 cm diameter. The dishes were placed in an oven set at 65 °C, 50 °C, and 35 °C until the film was dry. Subsequently, the dishes were removed from the oven and the films were peeled off and then stored at room temperature in polyethylene bags for further analysis [39, 40]. The bioplastic production process is described in Fig. 1, as shown below.

2.7 Characterization method of physical properties of plastic film

For every physical test, before starting any test, the sample was conditioned for 48 h in a specified temperature and humidity. For this purpose, we use the standard test method (ISO 2418:2005 and ISO2419:2005, Sampling, and Conditioning) [39, 40].

Water solubility test The aim of investigating water solubility is to know the rate of solubility of the sample in water. This parameter is used to know how the bioplastic film sample is dissolved, dispersed, or swollen in water and consequently changes the physical properties of aqueous systems undergoing gelation or thickening [27].

The film's solubility in water was determined according to the method reported in the literature [27]. Disks of the film (2 cm × 2 cm) were cut, weighed (M_1), and immersed in a beaker containing 50 ml of distilled water. After 24 h of immersion at 25 °C with a slow agitation, the pieces of the sample were taken out and dried to constant weight (M_2) in an air-circulated oven set at 105 °C for 24 h [27, 40].

$$\text{Water solubility(\%)} = \frac{M_1 - M_2}{M_1} * 100 \quad (18)$$

where M_1 = the initials mass and M_2 = the final mass of the sa

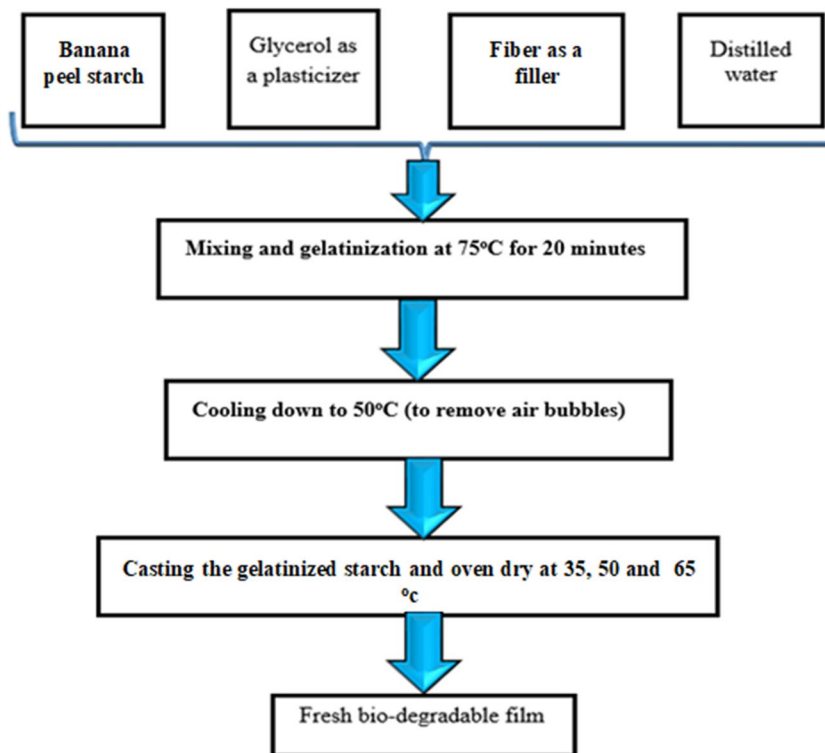
Water absorption test Water uptake was investigated by cutting film with a size of approximately 2 × 2 cm and then weighing the mass. The film was put into a container filled with distilled water for 24 h. After water immersion, the film was removed from the water and weighed to measure the wet weight. Water uptake was calculated as follows [27].

$$\text{Water absorption (\%)} = \frac{W_w - D_w}{W_w} * 100 \quad (19)$$

where W_w = wet weight and D_w = dry weight

Transparency of produced bioplastic The transparencies of the films were determined using a spectrophotometer

Fig. 1 Bioplastic synthesis process flow diagram



(UV 7804C). The transmittance of films was determined at 600 nm, as described in the literature [27].

$$\text{Transparency}(\%T) = \frac{-\log T_{600nm}}{X} \tag{20}$$

where T600 is the transmittance at 600 nm and X is the film thickness (mm).

Moisture content Before determining film properties, samples were conditioned at 25 °C and 53% relative humidity (RH) for 48 h. Film moisture content was determined through the weight loss through which the film went after a 24-h oven drying at 90 °C. Preliminary experiments showed us that 24 h was enough to dry up samples. The temperature was chosen to avoid the loss of the plasticizer [27].

$$\text{Moisture content}(\%) = \frac{\text{Final weight} - \text{initial weight}}{\text{Final weight}} * 100 \tag{21}$$

The thickness of the films The film thickness was measured using digital micrometers, and measurements were made in at least three random locations [27].

Tensile strength and elongation at break Tensile strength test specimens were conditioned to conform to standard requirements of thin plastic sheeting following ASTM D882 equipped with a 1 KN load cell at a crosshead rate of 50 mm/

min. From the developed plastic sheet, film strips of uniform width and thickness (10 mm × 50 mm) were cut [27]. T

$$\text{Tensile strength at yield (MPa)} = \frac{\text{Force}(N)}{\text{Area}(\text{width} * \text{thickness})\text{mm}^2} \tag{22}$$

$$\% \text{Elongation} = \frac{\text{Increase in the length}(\text{mm})}{\text{Original length}(\text{mm})} * 100 \tag{23}$$

FTIR analysis of the plastic film The functional groups and chemical bonds of produced bioplastic were identified by Fourier transform infrared spectrometry (FTIR). Then, the FTIR spectrum was allowed to pass through the prepared sample, and the spectrum responses were recorded [39].

Thermogravimetric analysis The thermal stability of the film was studied by using TGA and was performed on the Shimadzu TGA-50 equipment under a nitrogen atmosphere, with a flow rate of 50 mL/min, a heating rate of 20 °C/min, and a temperature range of 20 to 450 °C [33].

2.8 Experimental design and statistical analysis

Randomization of the experimental runs as well as appropriate analysis technique was ensured through proper utilization of software Design Expert 11 versions. By using the design expert of the response surface, the central composite design

Table 1 Factors and ranges of the CCD design quadratic model

Factors/independent variables	Range and levels	
	Ranges	Levels
Fiber conc	(5–20%) w/w	5, 12.5, and 20
Oven dry temperature	(35–65) °C	35, 50, and 65
Glycerol conc	(20–50%) w/v	20, 35, and 50

(CCD) of a quadratic model of the total experiment to be conducted was 20 for the synthesized film. Three independent variables were as follows: (A) dry oven temperature (°C) ranged from 35 to 65, (B) glycerol concentration (%) ranged from 20 to 50, and (C) fiber concentration (%); the dependent variables were tensile strength, water absorption, and elongation of the film. A second-order polynomial model was selected by taking into account the benefit of order and hierarchy. The factors and levels were available in Table 1.

$$\text{Tensile } Y_i = b_0 + \sum_{i=1}^n b_i X_i + \sum_{i=1}^n b_{ii} X_i^2 + \sum_{i=1}^n \sum_{j=i+1}^{n-1} b_{ij} X_i X_j \quad (24)$$

where $Y_i = b_0$ is the predicted response, b_0 the offset term, b_i the linear effect, b_{ii} the square effect, and b_{ij} is the interaction effect.

The following second-order polynomial models were used analogically to describe the relationship between the three independent variables and the three response variables:

$$\begin{aligned} TS = & a_0 + a_1 A + a_2 B + a_3 C \\ & + a_{12} AB + a_{13} AC + a_{23} BC \\ & + a_{11} A^2 + a_{22} B^2 + a_{33} C^2 \end{aligned} \quad (25)$$

$$\begin{aligned} ES = & b_0 + b_1 A + b_2 B + b_3 C \\ & + b_{12} AB + b_{13} AC + b_{23} BC \\ & + b_{11} A^2 + b_{22} B^2 + b_{33} C^2 \end{aligned} \quad (26)$$

$$\begin{aligned} WA = & c_0 + c_1 A + c_2 B + c_3 C \\ & + c_{12} AB + c_{13} AC + c_{23} BC \\ & + c_{11} A^2 + c_{22} B^2 + c_{33} C^2 \end{aligned} \quad (27)$$

where TS, EA, and WA are the predicted response which are tensile strength, elongation, and water absorption, respectively; a_0 , b_0 , and c_0 are intercepts; $[a_1, a_2, a_3]$, $[b_1, b_2, b_3]$, and $[c_1, c_2, c_3]$ were the estimated linear effects; $[a_{11}, a_{22}, a_{33}]$, $[b_{11}, b_{22}, b_{33}]$, and $[c_{11}, c_{22}, c_{33}]$ were quadratic effects; and $[a_{12}, a_{13}, a_{23}]$, $[b_{12}, b_{13}, b_{23}]$, and $[c_{12}, c_{13}, c_{23}]$ were interaction effects of independent variables which were “A” (dry oven temperature), “B” (glycerol concentration), and “C” (fiber concentration). The following second-order polynomial model is also used to describe the relationship between extraction time, extraction temperature, and the response variable (yield of extracted starch from the peel):

$$Y_i = e_0 + e_1 X_1 + e_2 X_2 + e_{12} X_1 X_2 + e_{11} X_1^2 + e_{22} X_2^2 \quad (18)$$

where Y_i is the yield of starch; e_0 is an intercept; e_1 and e_2 are the estimated coefficients of time (X_1) and temperature (X_2), respectively; e_{11} and e_{22} are quadratic effects; and e_{12} is the interaction effect of independent variables. Levels and factors are represented in Table 1.

3 Result and discussion

The result on proximate composition of raw banana peel

The main reason why the proximate analysis was done during this research is to predict the percentage of carbohydrates. Glucose is the simplest form of carbohydrate, while starch is a complex form of carbohydrate. So, estimating carbohydrates means indirectly determining the amount of starch. The result is illustrated in Table 2 based on the official method of analysis and association of official analytical chemists AOAC 1990 [22].

The moisture content The result shows small when compared to the value obtained [19, 29] but relatively higher than the value obtained [29]. This variation may be due to handling problems and the level of ripping. And high moisture content causes food items to enhance microbial spoilage and short shelf life, which can lead to its deterioration. As much as possible, the moisture content should be small, and the above result was somewhat good and acceptable.

Table 2 Result on proximate composition of the raw banana peel on a mass basis

Ash (%)	Moisture (%)	Crude protein (%)	Crude lipid (%)	Crude fiber (%)	Organic matter (%)	Carbohydrate (%)	Reference
8.5 ± 0.5	10.5 ± 0.03	2.17 ± 0.81	6.66 ± 0.1	34.37 ± 0.23	81 ± 0.33	37.8 ± 0.11	Current study
8.8	14.5	5.3	1.6	29.2	-	40.6	[19]
9.83	13.5	5.53	24	14.83	-	32.4	[28]
8.5	6.7	0.9	1.7	31	-	59	[29]

Total ash content The lower ash content may suggest low mineral content. On the other hand, the high values of the ash were indicative of high mineral content. The above result shows almost similar to the literature [19, 29].

The crude lipid content It was low and may be a good source of carbohydrates and can also contribute significantly to the energy content of the feeds that can be prepared with the wastes. The obtained result shows higher compared to the literature [19, 29]. This variation may be a result due to differences in varieties of bananas.

The crude fiber The values of crude fiber (34.37%) were reasonably higher than the first two studies but similar [28]. The high fiber content in diets has been reported to result in increased removal of potential mutagens, steroids, and Xenobiotics by binding or absorbing to dietary fiber components and thereby aiding digestion. Fibers are an indigestible portion of carbohydrates (cellulose, lignin, and hemicellulose).

Protein It is an essential component of the diet needed for the survival of animals and human beings; its basic function in nutrition is to supply an adequate amount of required amino acids. The crude protein content of the samples was very low when compared to other studies but higher than the result obtained [28].

Organic matter It is the total composition of the above constitute excluding the moisture content and ash contents. It comparatively depended on the amount of nitrogen-free extract.

Carbohydrate content The samples were very high in carbohydrate content and can be good sources of energy for the animals. The obtained result of carbohydrate ($37.8 \pm 0.11\%$) was almost higher than the value determined [28] and lower when compared with the two other studies [19, 29]. Since starch is a complex form or the polymer of carbohydrates, the result implies banana peel is rich in starch.

3.1 Starch extraction and yield results

Extraction time and extraction temperatures were the two factors and starch yield was the response. D-optimal RSM was taken to analyze the variance for 12 experiments.

The maximum yield was obtained at 35 °C and 22.5 min, which is 11.8 wt%. As the temperature rises beyond the range, the starch becomes jelly like, and it might be gelatinized and almost at a difficult stage to re-crystal. So, care should be given during operating the process. As extraction time increases, the yield, to some extent, slightly increases, but it is much more significantly affected by extraction temperature. The temperature very

significantly affects the yield. As temperature increases up to 35 °C, the yield increases (maximum) and then it decreases. But, starch gelatinization, which might start to occur at high temperatures (> 40 °C), may also increase the viscosity. Considering the efficiency of starch separation, the medium temperature should be used. Medium extraction temperature is mostly advisable because it reduces the viscosity of the starch suspension and makes it easy for centrifugation. The optimum conditions obtained were 27.66 min of extraction time and 37.92 °C extraction temperature with a yield of 12.06%. The optimization was supported by maximum desirability of 1. The maximum starch recovery had been achieved at 40 °C, and the minimum starch recovery had been achieved at 22 °C. This result almost agrees with the author which is 13% [29].

PH determination Banana peel starch powder was mixed with distilled water yielding a suspension. The pH of the mixture was found to be 6.31, which falls within the range of 4–7 [30]. In general, a starch suspension in water has a pH of 4–7 [30].

Moisture content The moisture content of starch powder may determine its quality and stability. Starch powder with high moisture content is vulnerable to bacterial growth compared with that with low moisture content. The moisture content of starch should be less than 20% [30]. The moisture content of banana peel starch powder was found to be 7.2%.

Loss on drying Testing of loss on drying is performed to measure the loss of water upon heating. The result showed that the loss on drying of banana peel starch was 11.20%, meeting the required specifications (Table 2). In general, the loss on drying of starch is less than 15%. As shown above, loss on drying is greater than moisture content, even if both parameters follow up the same procedure but differ slightly as a loss on drying is an analytical method to determine all volatile impurities like alcohol; conversely, the moisture content is related to water content.

Ash content It was below 1% which is 0.8% which satisfies the above specification. The color of the resulting banana peel starch was brownish white by visual inspection. It is also possible to change into pure white by using hydrogen peroxide as a bleaching agent [8]. The characteristics of banana starch were compared with the literature value and presented in Table 3.

Amylose determination Amylose was about 19.3%, and amylopectin was at 80.7%. This result agrees with the researcher which determines that banana peel starch consists of 20% amylose and 80% amylopectin [24]. Since the specification criteria of banana peel starch completely met

the above criteria, the extracted starch was efficient during plastic film production.

3.2 Chemical and physical properties of banana pseudo-stem fiber

The density of extracted fiber The density of the fiber after treatment was 1.4 gm/cm³. The results described the density as increased as the fiber was treated more because more polymerization gives higher molecular arrangements. Lignin and hemicellulose were removed, and the denser component (cellulose) probably described the content of the treated fiber. Before treatment, density was determined as 1.37 gm/cm³.

Yield for banana pseudo-stem fiber The average percentage yield of fiber in the pseudo steam is around 8%. Vigneswaran et al. [31] describe that the yield of banana stem fiber from bundle stem was around 10% which is almost approximated with the experimental result of 8%, and the reason for the decrement in yield was probably influenced by external disturbance variables [32].

The moisture content of the extracted fiber The average percentage of moisture content obtained was around 61.3 wt%, which is less moisture content than the work of Ebisike et al. [26]. Does this show the moisture content of fresh banana fiber was around 90% which is higher than the value of 61.3%? This may probably cause by the removal of moisture during transportation and the extraction process.

Water absorption of the extracted fiber The water absorption capacity of fiber was measured. The absorption capacity of fiber was around 4.6%. It is possible to minimize this number by treating it with soda; during this, the hydroxyl group is substituted by sodium during the treating process.

Table 3 The result on characteristics of banana peel starch comparison with the literature value

Types of test	Present study result	[30]
Qualitative test	Bluish violet color	Bluish violet color
Form	Powder	Powder
Color	Brownish white	White
Oder	Odorless	Odorless
Test	Tasteless	Tasteless
PH	6.31	4–7
Moisture content	7.2%	<20%
Loss on drying	11.20%	<15%
Ash content	0.8%	<1%
Amylose	19.278%	17–25%

Cellulose and non-cellulose content of the extracted fiber The percentage cellulose content was around 55–60% which matches and describes that the cellulose content of banana pseudo-stem fiber was around 50–60% [18]. And the other authors have also quantified the cellulose content of banana stem fiber between 80 and 85% [32]. This variation may be observed due to the variety difference as well as due to the difference in the procedure used. The cellulose content of the raw fiber is higher than the other wood and non-wood source of fiber, as already mentioned in the literature [32]. As indicated by this researcher [32], banana fiber consists of 82–85% of cellulose, but other particulars like rice straw consist 28–48%, 32–480% (sugarcane), 45–50% (jute), and 29–50% (wheat straw) of cellulose content [32]. This implies that the banana stem is highly fibrous. Based on the literature [32], banana fiber contains a higher amount of cellulose, which implies banana pseudo-stem is best for this work since banana cellulosic fiber was used as a filler reinforced with starch. Banana pseudo-stem cellulosic fiber is chosen in this research as it is highly available and due to its efficiency toward improving the physicochemical properties of the film as investigated throughout the document.

Thermal characterization (TGA) for the extracted fiber The TGA analyses of fiber are shown in Fig. 2. The fiber had three main weight loss regions. The initial small weight loss, which was caused by the loss of free water adsorbed in the fibers, occurred in the range of 45 to 135 °C. The second weight loss in the temperature ranges 260 to 360 °C. The weight loss percentage was about 15 to 60%. And it mainly contributed to the depolymerization of hemicelluloses and the thermal decomposition of α -cellulose. The third weight loss region was due to the degradation of non-cellulosic substances such as lignin. Given that lignin is the most difficult constituent to decompose, its decomposition usually covers the whole temperature range of 200 to 600 °C. The above result obtained was almost similar to the literature done by Teli and Terega [33].

3.3 Analysis of bioplastics on tensile strength, elongation, and water absorption

The experimental values of the tensile strength (TS), elongation at the break (EA), and water absorption (WA) obtained under different conditions are presented in Table 4.

3.3.1 Statistical analysis of factors affecting the response variables

The results were analyzed by using ANOVA (analysis of variance) appropriate for the experimental design used and shown in Table 5. The ANOVA of the quadratic regression

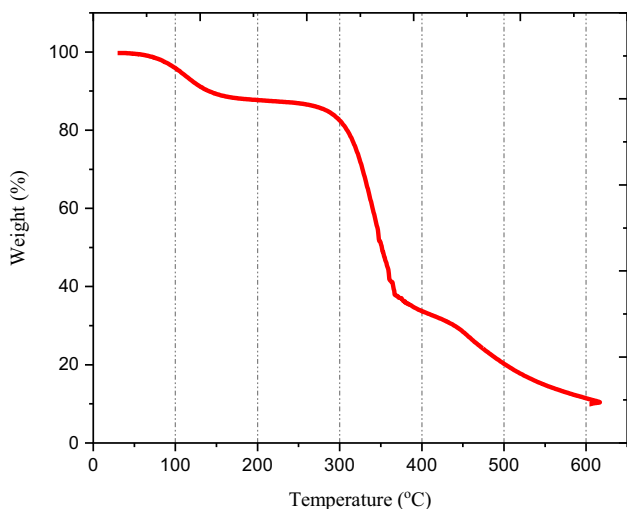


Fig. 2 Thermograph (TGA) of banana pseudo-stem fiber

model indicates that the model is significant. The model *F*-value of 439.37, 182.1, and 159.97 implied the model to be signed for tensile strength, elongation at the break, and water absorption, respectively. The values of *P* less than 0.0500 indicate the model terms to be significant. For tensile strength, the coefficients estimate and the corresponding *P* values suggest that, among the test variables used in the study, in this case, A, B, C, A², B², C², AB, and AC are

significant model terms, as shown in Table 5. For elongation, at the break, the coefficients estimate and the corresponding *P* values suggest that, among the test variables used in the study, in this case, A, B, C, AB, BC, A², and B² are significant model terms, and for the water absorption, the coefficients estimate and the corresponding *P* values suggest that, among the test variables used in the study, in this case, A, B, C, AC, A², B², and C² are significant model terms. The “lack of fit *F*-value” of 0.7923, 1.94, and 0.4192 implies the lack of fit is not significant relative to the pure error for tensile strength, elongation at the break, and water absorption, respectively.

3.3.2 Adequacy check for the developed response surface quadratic models

The predicted *R*² of 0.9932, 0.9529, and 0.9789 is in reasonable agreement with the adjusted *R*² of 0.9952, 0.9885, and 0.9869 for tensile strength, elongation at the break, and water absorption, respectively. The fit of the model was also expressed by the coefficient of regression *R*², which was found to be 0.9975, 0.9939, and 0.9931, indicating that 99.75%, 99.39%, and 99.31% of the variability in the response could be explained by the model for tensile strength, elongation at the break, and water absorption, respectively.

Table 4 Values of the three response variables associated with the three factors

STD	Run	Factor1 A: dry. temp (°C)	Factor2 B: glycerol conc. % (w/v)	Factor 3 C: fiber conc. % (w/w)	Response1 TS (Mpa)	Response 2 EA (%)	Response 3 WA (%)
1	16	35	20	5	1.05	15.2	30
2	6	65	20	5	3.8	5.9	22.36
3	19	35	50	5	0.2	31.01	35.3
4	12	65	50	5	2.02	18.8	27.54
5	5	35	20	20	1.52	13.5	25.3
6	3	65	20	20	5.01	4.9	20.69
7	4	35	50	20	0.32	26.1	30.5
8	17	65	50	20	2.86	12	25.7
9	7	35	35	12.5	3.02	20.1	24
10	15	65	35	12.5	5.7	7.8	18.6
11	13	50	20	12.5	5.55	6.5	20
12	10	50	50	12.5	3.9	17	25
13	18	50	35	5	6.3	13.3	23
14	8	50	35	20	6.9	8.5	20.8
15	20	50	35	12.5	7.25	10	19.2
16	9	50	35	12.5	6.9	9.6	19.22
17	11	50	35	12.5	6.8	11	20.5
18	14	50	35	12.5	7.01	9.4	18.9
19	1	50	35	12.5	7.18	9.5	19.2
20	2	50	35	12.5	7.21	9.6	20.1

Table 5 Analysis of variance [partial sum of squares] for tensile strength

Source	Sum of squares	Mean squ. value	F-value	Prob > f	
Model	119.55	13.28	439.37	<0.0001	Significant
A-oven T	17.64	17.64	583.33	<0.0001	
B-gly. conc	5.82	5.82	192.56	<0.0001	
C-FB	1.05	1.05	34.72	0.0002	
AB	0.4418	0.4418	14.61	0.0034	
AC	0.2665	0.2665	8.81	0.0141	
BC	0.0648	0.0648	2.14	0.1739	
A ²	17.27	17.27	571.19	<0.0001	
B ²	12.60	12.60	416.91	<0.0001	
C ²	0.1944	0.1944	6.43	0.0296	
Residual	0.3023	0.0302			
Lack of Fit	0.1336	0.0267	0.7923	0.5977	Not significant
Pure Error	0.1687	0.0337			
Cor Total	119.85				

Figure 3 represents the line of a perfect fit with points corresponding to zero error between predicted values and actual values and demonstrates that the regression model equation provided an accurate description of the experimental data for all responses, in which all the points are close to the line of a perfect fit. Normal probability plots of (A) tensile strength, (B) elongation, and (C) water absorption also fit with the line, as indicated in Fig. 4.

3.4 Effects of individual process variables

The effect of oven-dry temperature, glycerol, and fiber concentration on tensile strength, elongation, and water absorption was studied and evaluated for the best bioplastic film synthesis conditions.

3.4.1 Effects of dry oven temperature on tensile strength, elongation, and water absorption

The dry oven temperature varies from 35 to 65 °C, and both fiber and glycerol concentrations were kept constant at 12.5% and 35%, respectively. The increase in dry oven temperature improves the tensile strength and provides better quality for the produced film. But, as the temperature is beyond the range or rises, it was difficult to peel off the film at the Petri dish. The minimum tensile strength was obtained at 35 °C which is 0.2 Mpa, and the maximum tensile strength was 7.25 Mpa at 50 °C. Increasing the heating temperature can cause depolymerization in the amylose chain; the straight-chain amylose falters and becomes shorter, thus decreasing amylose content [34]. Generally, as the drying temperature increased, tensile strength increased up to a center point (50 °C), then tensile strength decreased, and finally, it becomes parabolic; this implies the center point favors the tensile strength of the film.

The increase in the dry oven temperature decreased elongation at the break from 31.01 to 4.9% and water absorption of the film from 35.3 to 18.6%. The percent elongation at the break was 20.1% at 35 °C when the temperature increased to 50 °C %EA reduced from 20.1 to 10%, and finally, when the drying temperature reaches 65 °C, %EA decreased to 7.8%. This was due to increasing the tensile strength of bioplastic as well as increasing drying temperature from the lower level up to a center point. The result of elongation at break decreases with increasing temperature. This is because the heat that is given causes an increase in the kinetic energy of the molecules in which the molecules vibrate and create a free volume to allow larger molecular chain rotation [35].

The effect of drying temperature on water absorption of bioplastic was shown in Fig. 4C. The lowest water absorption was observed at a higher level or at 65 °C which is 18.6%. At minimum dry temperature (35 °C), water absorption (WA) is 24%; when drying increases to 50 °C, WA decreases to 19.7%, and then it decreases to 18.6% when the temperature is kept at a high level (65 °C). This tells us the higher drying temperature gives the lower water uptake of the film.

3.4.2 Effects of glycerol concentration on tensile strength, elongation, and water absorption

At a minimum glycerol concentration (20%), the tensile strength of the film was 5.55 Mpa; when glycerol concentration increased from 20 to 35%, the tensile strength of the film increased from 5.55 to 6.92 Mpa, and finally, when glycerol concentration raises into 50%, the tensile strength of the film declines into 3.9 Mpa. As the glycerol concentration is beyond the range or rises more, it was difficult to peel off the film and the film becomes highly flexible. As the glycerol concentration reaches

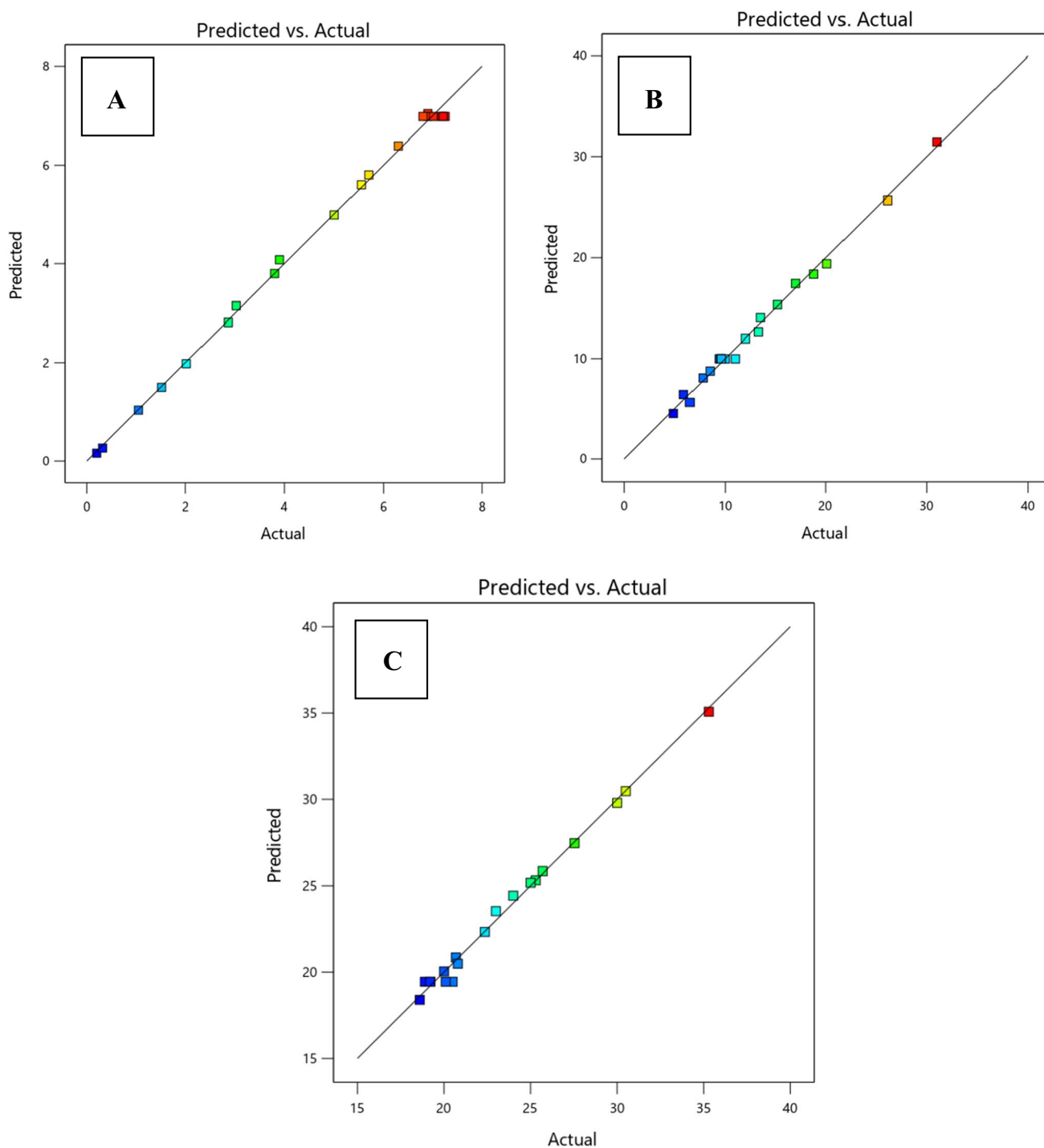


Fig. 3 Predicted versus actual plots of (A) tensile strength, (B) elongation, and (C) water absorption

beyond the saturation/increases, tensile strength declines [36]. Generally, the medium glycerol concentration (35%) favors getting the maximum tensile strength (6.92 Mpa) of the film.

The increase in glycerol concentration from 20 to 50% resulted in an increase in both elongations at the break

from 4.9 to 31.01% and water absorption of the film from 18.6 to 35.3%.

The possible reason for the high tensile strength at low plasticizer concentration is the domination of strong hydrogen bonds produced by a starch-starch and starch-fiber intermolecular interaction over starch-plasticizer attraction.

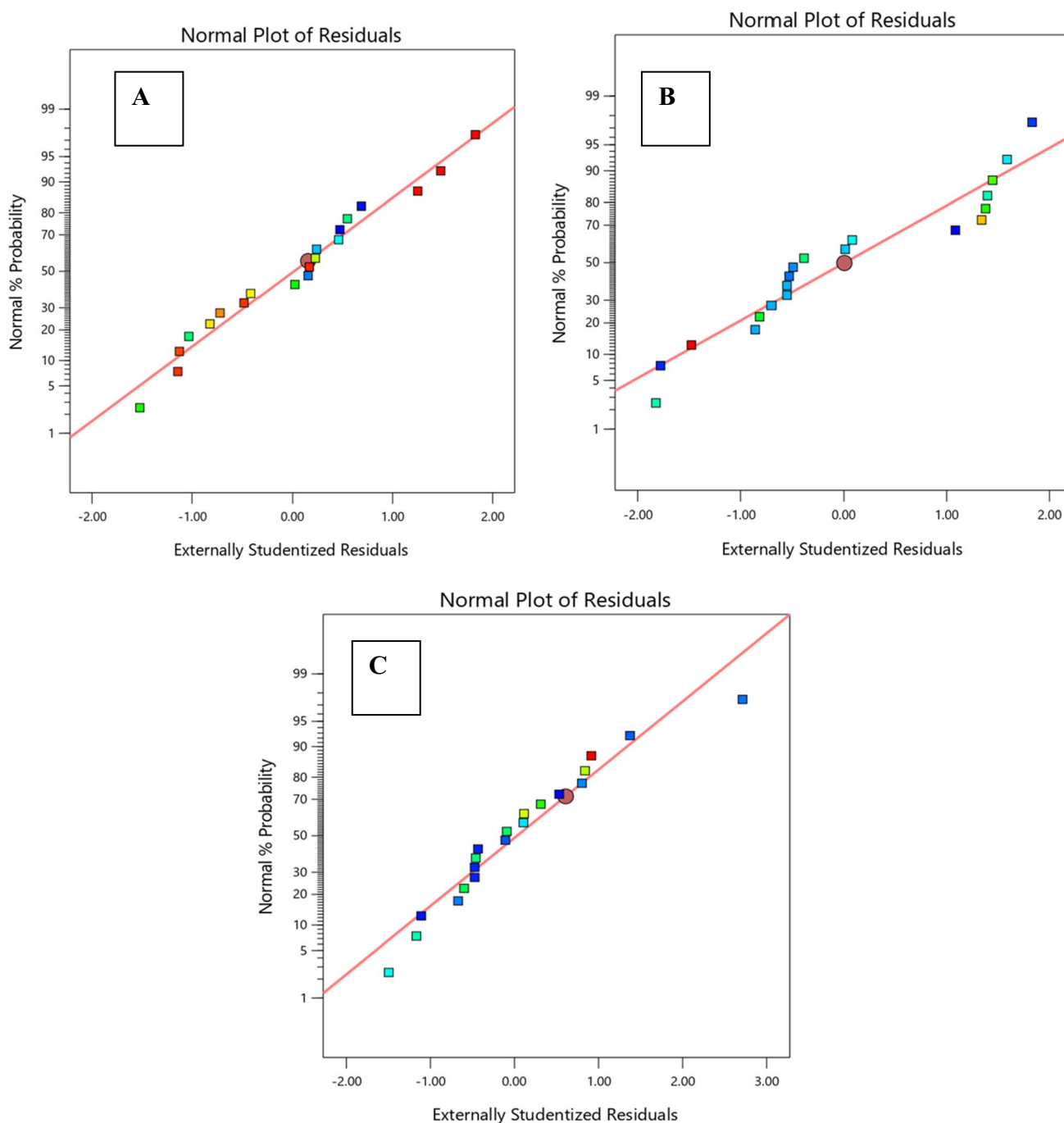


Fig. 4 Normal probability plots of (A) tensile strength, (B) elongation, and (C) water absorption

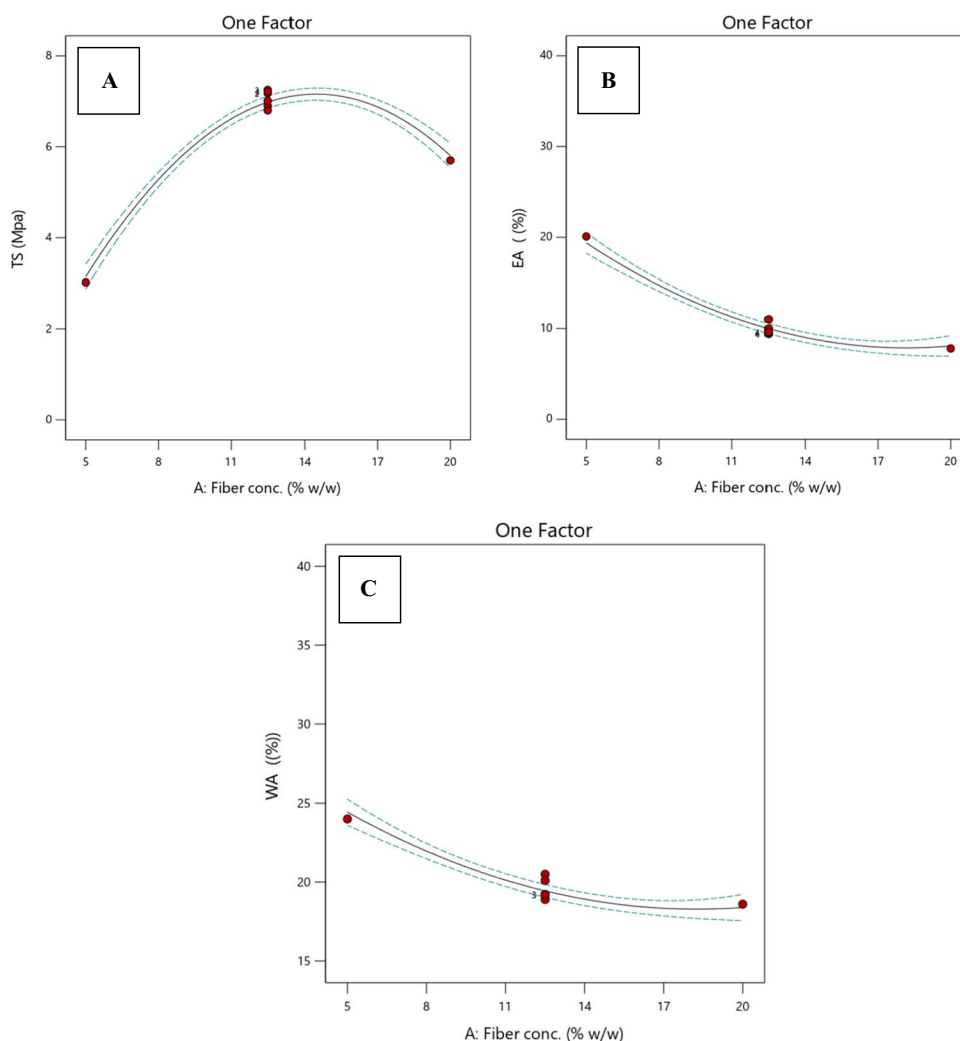
3.4.3 Effects of cellulosic fiber concentration on tensile strength, elongation, and water absorption

When fiber concentration increased in the center point (12.5%), the tensile strength of the film enhanced (7.03 Mpa); finally, as fiber concentration increased to a higher level (20%), the tensile strength decreased to 6.9 Mpa.

Generally, as fiber concentration increased up to a center point, tensile strength increases, and beyond the center point, it decreases. This increment is due to the stronger bonding between the fiber and the starch matrix. The researcher describes that at 0% matrix, tensile strength was 3 Mpa, and at 15% fiber, 9 Mpa was recorded [37]. As fiber concentration increased up to a center point (12.5%),

elongation decreased to 10%. Finally, as fiber concentration increased to a higher level (20%), elongation was reduced to 8.5%. As the matrix of synthetic or biological polymers increased, the values of the tensile strength of bioplastics increased (up to a center point) and decreased the values of elongation at fracture. This is in agreement with other literature [37]. When the fiber content was 5%, water absorption was maximum (23%), and as fiber concentration increased up to a center point (12.5%), water absorption decreased to 19.7%; finally, as fiber concentration increased to a higher level (20%), water absorption was slightly increased into 20.8%. This water absorption reduction in bio-composites can be attributed to stronger hydrogen bonds between the matrix and the reinforced material [38]. The above discussion is illustrated in Fig. 5 below. Figure 5A shows effects of cellulosic fiber concentration on tensile strength. Figure 5B shows effects of cellulosic fiber concentration on elongation at the break, and Fig. 5C shows effects of cellulosic fiber concentration on water absorption.

Fig. 5 Effects of fiber concentration on tensile strength (A), elongation (B), and water absorption (C)



3.5 The interaction effect between process variables and responses

To study the interactive effect of factors on tensile strength, elongation, and water absorption, the response surface methodology was used and the 3D surface was drawn. The significant interaction effect of the factors was discussed. There are three interaction factors analyzed by the model equation. These are AB (dry oven temperature and glycerol concentration), AC (dry oven temperature and fiber concentration), and BC (glycerol concentration and fiber concentration). Figures 6, 7, and 8 describe all about this.

3.5.1 Interaction effects of dry oven temperature and glycerol concentration on tensile strength, elongation, and water absorption

In Fig. 6, an increase in dry oven temperature and the glycerol concentration up to a center point enhance the tensile

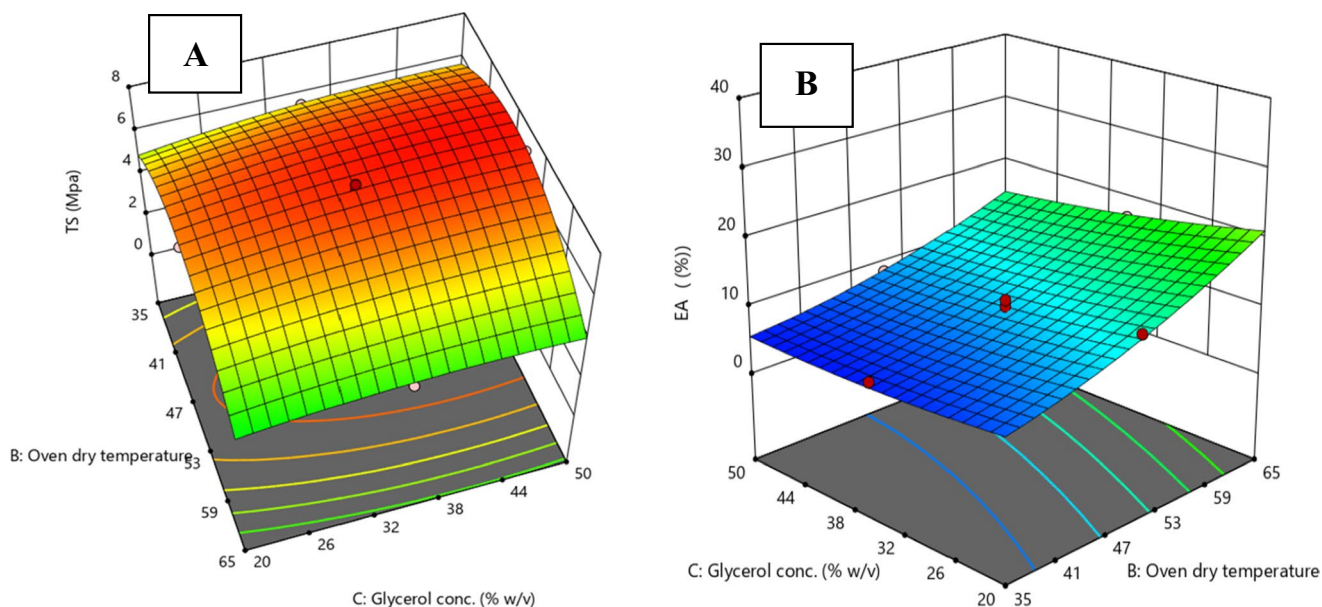


Fig. 6 Interaction effects of dry oven temperature and glycerol concentration on tensile strength (A) and elongation (B)

strength of the film. And at the lower temperature and higher glycerol concentration, the minimum tensile strength was recorded. Dry oven temperature increases, and glycerol concentration decreases the elongation and water absorption increase; on the other hand, dry oven temperature decreases, and glycerol concentration raises both elongation and water absorption increase; this is because the higher mobility of the polymer due to the increase glycerol enables the film to absorb moisture over time which is likely due to the hydrophilic nature of glycerol.

3.5.2 Interaction effects of dry oven temperature and fiber concentration on the tensile strength, elongation, and water absorption

Figure 7A,B of the 3D shows the effects of dry oven temperature with fiber concentration and their mutual interaction on tensile strength and water absorption at constant glycerol concentration (35%). The increase in dry oven temperature and fiber concentration increases the tensile strength. The highest response value, 7.25 Mpa, was observed at 12.5% fiber

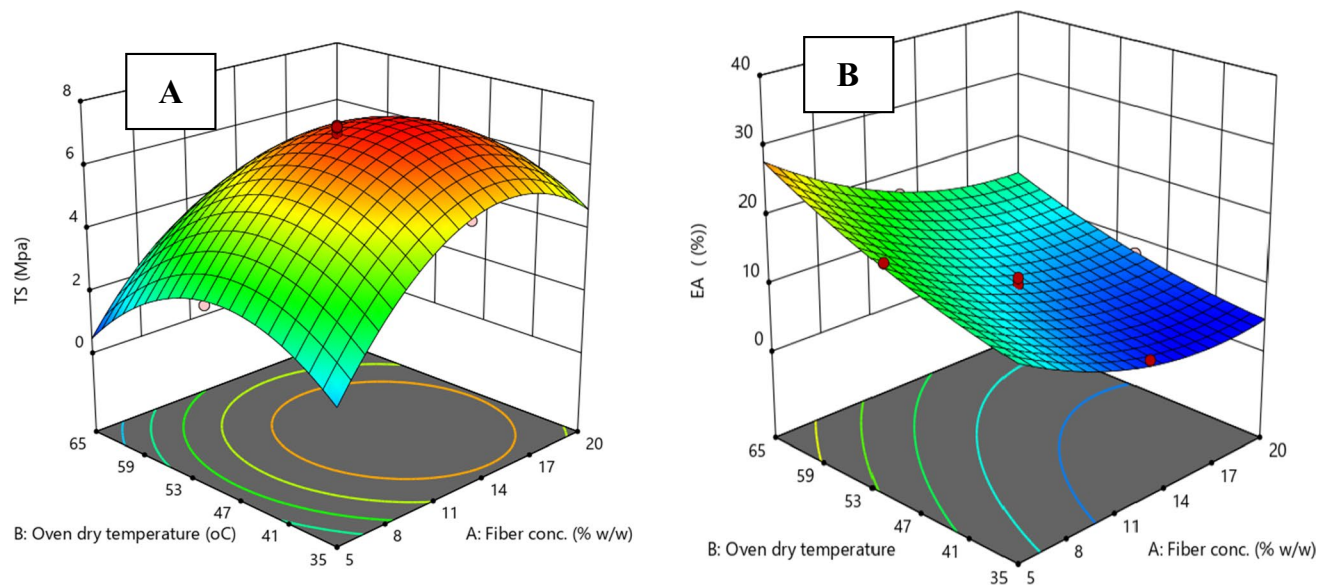


Fig. 7 Interaction effects of dry oven temperature and fiber concentration at 35% gly conc on tensile strength (A) and water absorption (B)

concentration and 35% glycerol concentration, further increasing the temperature, and fiber decreases the tensile strength due to the degradation of starches. On the other hand, both dry oven temperature and fiber concentration rise, EA and WA decrease, and as both dry oven temperature and fiber concentration decrease, EA and WA increase. As fiber concentration and dry oven temperature decreased, EA increased from 4.9 to 31.01% and WA rises from 18.6 to 35.3%.

3.5.3 Interaction effects of both fiber and glycerol concentration on the tensile strength, elongation, and water absorption

Figure 8 of the 3D represents the effects of fiber concentration with glycerol concentration and their mutual interaction on the elongation of the film. Varying fiber concentration and glycerol concentration had a significant effect on the EA value. As both fiber concentration and glycerol concentration increase, EA increases. Probably, this effect is due to an interaction of the fiber with the glycerol, increasing the molecular mobility and resulting in less rigid and highly flexible materials; the glycerol concentration dominates the fiber concentration. Interaction effects of both fiber and glycerol concentrations significantly affect EA, but the effect is insignificant for both TS and WA.

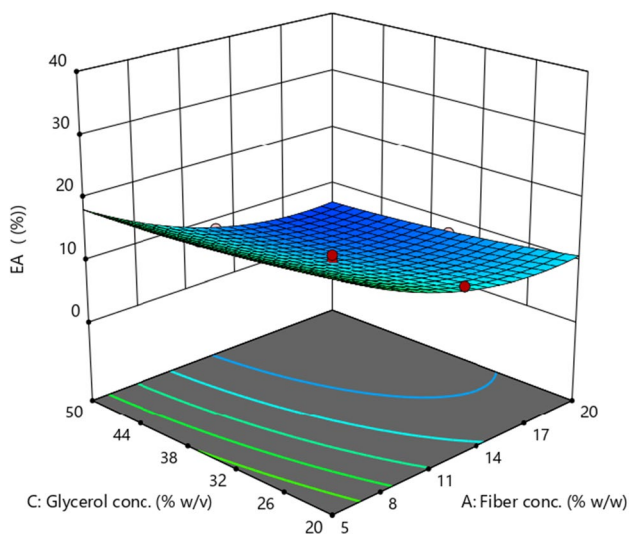


Fig. 8 Interaction effects of fiber and glycerol concentration on elongation when dry oven temperature fixed at 50 °C

Table 6 Model validation

Number	Dry oven temp	Gly. conc	FB	TS Mpa	EA%	WA%
Predicted	56.52	28.60	18.26	7.23	5.46	18.60
Experimental	56.5 ± 0.34	28.6 ± 0.72	18 ± 0.2	7.25 ± 0.01	5.49 ± 0.01	18.70 ± 0.05

3.5.4 Process factors and response variables optimization

Numerical optimization was used to optimize the process. To verify this prediction, experiments were conducted, and the results showed that 7.25 Mpa tensile strength, 5.49% elongation at the break, and 18.70% water absorption were obtained, as shown in Table 6. Therefore, the numerical optimization can be taken as an optimal value because the predicted value is close enough to the experimental. The optimum values of the process variables were 56.5 °C dry oven temperature, 28.6% glycerol concentration, and 18.23% fiber.

The tensile strength of banana peel via banana pseudo-stem fiber-based bioplastic film was found to be 7.23 Mpa, which was higher than the bioplastics produced from cassava (4.5 Mpa), corn starch (3.59 Mpa), potato starch (4.87 Mpa) [39], and banana peel (0.443–1.596 Mpa) [40]. Elongation at the break result obtained under the optimal condition was determined as 5.46%. As reported, 8.69% elongation for the antimicrobial plastic film was done before [39]. Also, this parameter was reported as 4.32% elongation for the bioplastic developed from corn starch via bagasse cellulosic fiber[7].

3.6 Physicochemical characterization of the bioplastics film

In this research, controlled film means the bioplastic film without cellulosic fiber, and reinforced film means the film with cellulosic fiber. So, the controlled film is made up of starch and glycerol without the addition of cellulosic fiber as a filler. This film is used as a reference to know the implication of the reinforced film due to the involvement of cellulosic fiber as a filler.

Water solubility test Being a hydrophilic polymer shows a high affinity toward the water. Hence, upon hydration, starch films absorb water and swell. The film with fiber had a lower swelling capacity than the films with only containing starch. Therefore, fiber-starch films showed a lower swelling capacity than only starch-based films. The result shows as the solubility of the control film or the film with starch only, excluding fiber, was 45%, and the film blends with fiber shows as 40% solubility, as indicated in Table 7.

Density test The density of the starch-based films was measured for both the control and matrix. It was observed

Table 7 Results on some physicochemical properties for both reinforced and control films of the present study

Product	Thickness (mm)	Moisture (%)	Transparency (%)	Solubility (%)	Density (g/ml)
Control	0.41 ± 0.05	9.56 ± 0.17	116 ± 0.32	45 ± 0.25	1.65 ± 0.34
Reinforced	0.42 ± 0.05	9.21 ± 0.25	103 ± 0.15	40 ± 0.08	1.72 ± 0.55

the possibility to increase the density by blending fiber. The density increment is due to the compatibility of the extracted fiber. The obtained result was 1.65 g/ml for the control film and 1.72 g/ml for the blended fiber film, as illustrated in Table 7.

Transparency of produced bioplastics The transparencies of the films are determined using a spectrophotometer (UV 7804C) for both control and the film obtained at the optimum conditions. The results showed that 116% transmittance for the control film and 103% transmittance were obtained for the blended film. Because fiber is cloudier than banana peel starch, so the transparency decreases, as shown in Table 7, from 116% for starch to 103% for matrix. The decrease in transparency values was observed due to fiber incorporation [27].

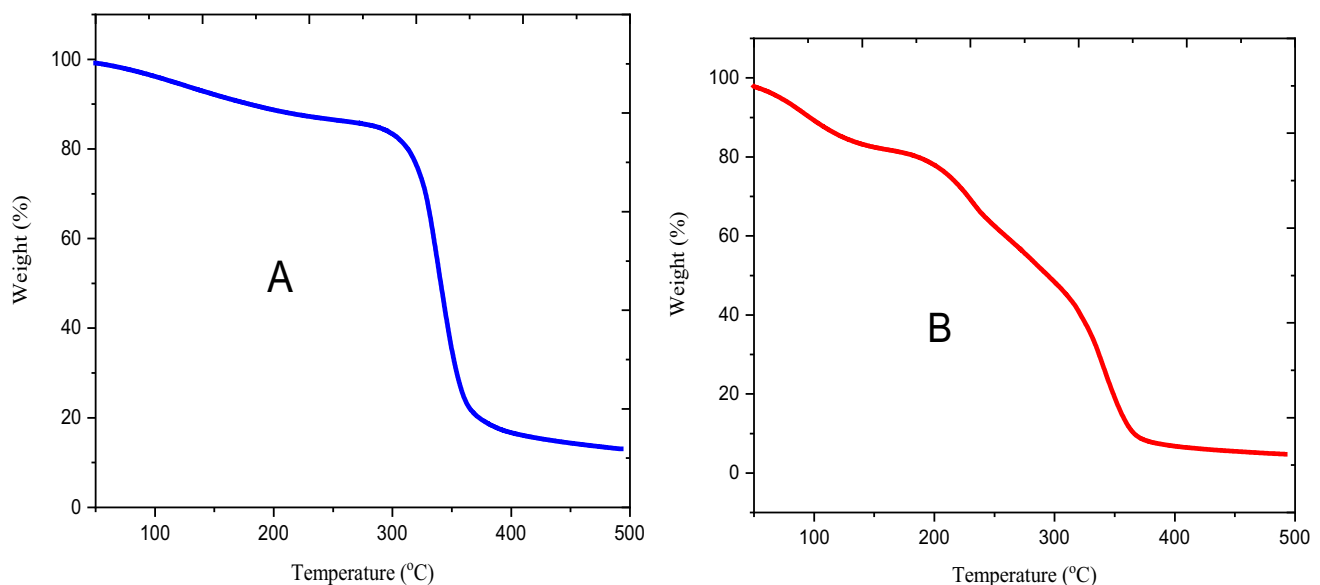
Moisture Since starch is highly hydrophilic, it absorbs more moisture than the film obtained by blending fiber. The moisture percentage for the pure starch film is higher than the blended. The obtained result was 9.56% for the control and 9.21%, as shown in Table 7, for the blended plastic film.

The thickness of the films A digital micrometer was used to measure the film thickness. The film synthesized from only

starch had a small thickness difference from with fiber. The reason is more starch and glycerol vaporize during oven drying than for the film synthesized by blending. The obtained thickness, as illustrated in Table 7, was 0.41 mm for the control and 0.42 mm for the blended film by taking the average of the triplicates.

3.7 Thermal analysis of films using TGA

As shown in Fig. 9B, the weight of the sample lost for the controlled film increases more rapidly than the reinforced bioplastic film in Fig. 9A. In Fig. 9B, 50% mass of the controlled film was lost at 291 °C, and 50% of the mass of the reinforced film was also lost at 340 °C (Fig. 9A). The final total mass residue or ash for the controlled film was 4.93%, and also for the reinforced film, the total ash was 13.4% at 450 °C. The derivative mass loss also tells that the controlled film decomposes slightly at 75.3 °C, highly decomposes at 225.16 °C, and critically decomposes at 336 °C. This film was sensitive to heat and highly affected by temperature. The reinforced film obtained by the incorporation of 18% fiber, on the other hand, is slightly affected by temperature; a small decomposition was observed at 100 °C; and a large decomposing was shown at 340 °C. Figure 9 shows the mass of the sample versus temperature for the controlled film (the bioplastic

**Fig. 9** Mass loss by using TGA for the reinforced film (A) and controlled film (B)

film without cellulosic fiber) and the reinforced film (the film with cellulosic fiber). Again, for more clarity, mass loss corresponding to temperature is clearly illustrated in Fig. 9.

Again, for more clarity, mass loss corresponding to temperature for both controlled and reinforced film is clearly illustrated in Table 8 below. A large amount of weight loss in the temperature range of 300 to 360 °C was observed for the reinforced film; this behavior is due to the nature of the fiber, as shown in Fig. 2, of cellulosic fiber investigation by TGA. Figure 2, under the fiber characterization section by TGA, states that the weight loss percentage was extremely higher, and it mainly contributed to the depolymerization of hemicelluloses and the thermal decomposition of α -cellulose above 300 °C as justified by Teli and Terega [33]. The following data were generated during characterization through TGA, as shown in Table 8.

DSC analysis of the reinforced film DSC enables us to determine the number of parameters, such as melting temperature (T_m), crystallization temperature (T_c), glass transition temperature (T_g), and degradation temperature (T_d). T_g , T_c , and T_m were taken from the onset temperatures of endothermic step change, exothermic peak, and endothermic peak, respectively. According to DSC analysis in Fig. 10, it was noted that a first endothermic peak between the temperature range of 55 °C and 58 °C represents residual water, which was used during the pre-gelatinization process. The second endothermic peak which was between a temperature range of 95 °C and 120 °C represents residual water evaporation, which was used during the process of molding gelatinized starch. The last and the third endothermic peak were between 170 °C and 175 °C representing the total loss of heat due to overheating.

Table 8 Mass loss determined by TGA for both controlled and reinforced film

Temperature (°C)	Controlled fil mass loss(%)	Reinforced film mass loss(%)
29.63	0.4	0.19
30	0.65	0.25
40	1.34	0.5
70	4.8	1.79
100	10.8	3.81
150	17.5	7.86
200	22.05	11.3
250	37.5	13.5
300	48	83
350	19.08	34.73
400	7	17

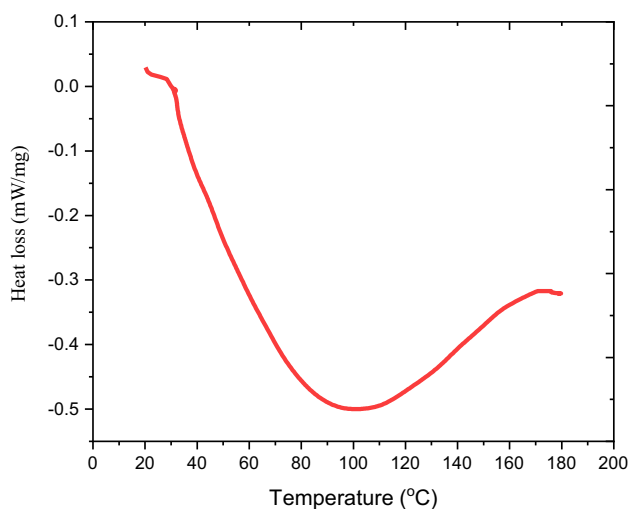


Fig. 10 Heat loss by using DSC for the reinforced films

Fourier transform infrared spectrometry analysis of the film The FTIR spectra of banana peel starch-glycerol-based films with cellulosic fiber are shown in Fig. 11. Generally, from Fig. 11, the band 1007–897 cm^{-1} C–O–H bending vibration, 1150–1005 cm^{-1} C–C stretching, 1235–1150 cm^{-1} C–O–C stretching of ester, 1734–1631 cm^{-1} bounded water, 3000–2826 cm^{-1} C–H stretches, and 3600–3000 cm^{-1} -OH stretching due to polymeric involvement of hydroxyl groups and bonded O–H stretching vibration present in starch was observed. A possible explanation for Fig. 11 is hydrogen bonding between the CH_2 -OH groups from the starch and cellulose fiber; this justification is similar as cited [39]. Having this, the fiber, starch, and glycerol

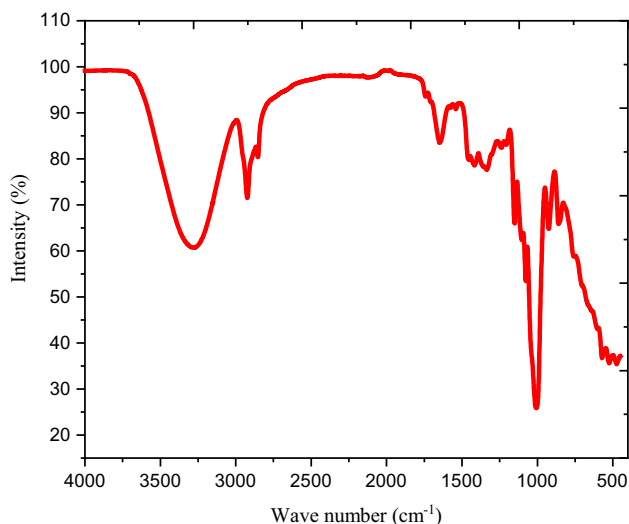


Fig. 11 FTIR result of starch-fiber, bioplastic film plot

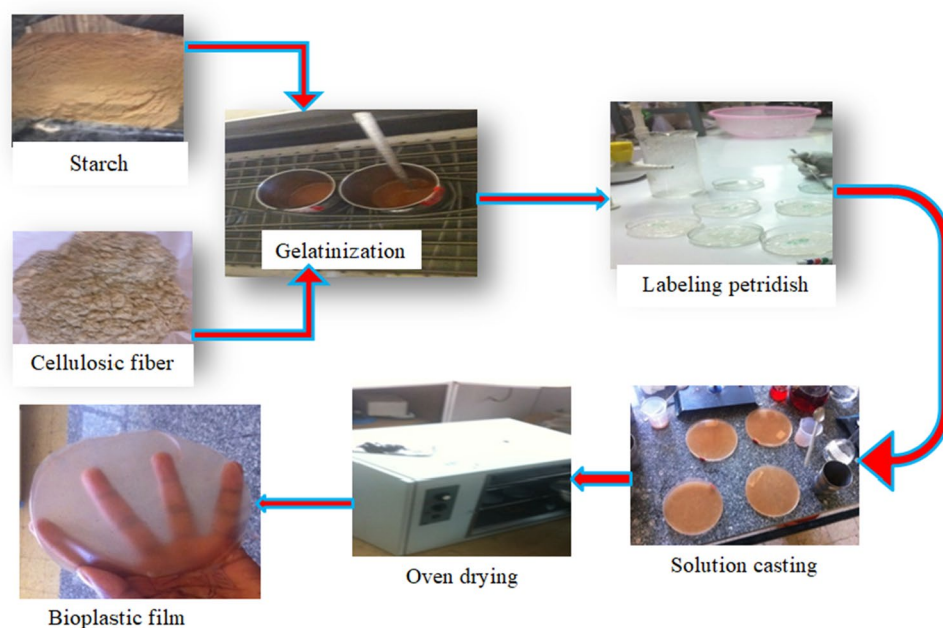
are attached chemically; this implies that fiber is favored to the starch by forming reinforced with the starch matrix [39].

The optimized conditions that have been considered had high tensile strength, low elongation, and low water absorption, which is chosen using numerical optimization as a combination of 56.5 °C dry oven temperature, 28.6% glycerol, and 18.26% fiber concentration to obtain a good quality plastic film (7.23 Mpa TS, 5.46% EA and 18.60% WA of the film). The addition of cellulosic fiber enhances both the physicochemical and thermal properties of the film. Cellulosic fiber enhances TS but lowers both EA and WA. Using the optimized plastic film, the thickness, solubility, moisture, density, and transparency were determined for both the controlled and the reinforced fiber films. The addition of 18% fiber indicates a better bioplastic film with better physicochemical properties when compared to the control film. The TGA result shows that the mass loss of the reinforced film is less than the controlled film; this is due to the incorporation of thermal-resistant cellulosic fiber. The best plastic film synthesized at the optimum operating condition was also characterized by FTIR shows as the fiber and starch well interacts chemically. Finally, the overall results showed that the banana peel starch and banana pseudo-stem fiber has a promising potential to be used in combination with glycerol plasticizer in bioplastic film synthesis for dry good packaging applications. The bioplastic production process in the laboratory is indicated in Fig. 12 below.

4 Conclusion

In this research, the development of bioplastic film from banana peel starch and banana pseudo-stem fiber was investigated. This work also intended to study the proximate composition of the banana peel, the influence of starch extraction parameters: extraction time and extraction temperature on the starch yield, and the characterization of starch properties. Based on these results, it can be concluded that banana peel should be considered suitable for bioplastic synthesis because the proximate composition shows values of 10.5% moisture, 8.5% total ash, 6.66% crude lipid, 2.17% crude protein, 34.37% crude fiber, and 37.8% nitrogen-free extract. This raw material is suitable for bioplastic synthesis mainly because of its relatively higher carbohydrate, which is the main source of starch. Banana pseudo-stem fiber was extracted, and the characterization shows high density, low water uptake capacity, higher cellulose content, and higher thermal resistance capability when analyzed by TGA. During the synthesis of bioplastic film, the effects of independent variables (dry oven temperature, glycerol, and fiber concentration) on tensile strength (TS), elongation at the break (EA), and water absorption (WA) using CCD response surface experimental design were studied. From the conducted experiment, the recorded result shows that the minimum EA was obtained at a higher drying temperature and fiber concentration but at a low level of glycerol concentrations. All in all, banana peel starch and pseudo-stem fiber are assured viable materials for bioplastic film production.

Fig. 12 Pictures of the bioplastic synthesis process during the laboratory section



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Author contribution Conceptualization, methodology, formal analysis, investigation, validation: W.G. Abera. Supervision, revising, and validation: R.C. Kasirajan. Writing the draft manuscript, re-editing, and final editing: S.L. Majamo.

Data availability The data used to support the findings of this study are included in the article.

Declarations

Ethics approval and consent to participate Not applicable.

Competing interests The authors declare no competing interests.

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