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Research Article

Optimization of some physical and mechanical properties of film developed from *Tacca* starch and plasticizer blends

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Abstract

Tacca (*L. Kunze*) starch films (TSF), that are biodegradable were developed from the blending of plasticizers with *tacca* starch for possible use in food packaging. The effects of ingredient proportions (Starch level: 5-15g, Sorbitol plasticizer: 0-4.5g, Glycerol plasticizer: 0-4.5g) and Temperature: (75-95^{0}C) on some physical (thickness, density, moisture content) and mechanical (Tensile strength and elongation) properties of the biodegradable films were investigated using Box-Behnken's experimental design in Response Surface Methodology (RSM). Linear regression models (thickness, density, moisture content and elongation) and quadratic models (tensile strength) were developed. Analysis showed that starch level significantly ($p < 0.05$) affected all the responses at 5% level of significance except for tensile strength ($p > 0.05$). Results also showed that plasticizer blends (both sorbitol and glycerol) had significant ($p<0.05$) effects on moisture content and the tensile strength of the biodegradable films. Also, there were interactive effects of the mixture ingredients and process temperature on the tensile strength of the biodegradable films. The plasticizers in combination, exerted significant ($p<0.05$) effect on the tensile strength of the biodegradable films at a 5% level of significance. The optimized values indicated that TSF prepared with 13.728g of starch, 4.5g of sorbitol, 4.5g of glycerol and 75^{0}C of process temperature had improved and satisfactory response variable with a desirability value of 0.609. The Coefficient of determination ($R^2 > 0.60$) was obtained for each response variable and the plots showed a good correlation between experimental and predicted values, revealing the adequacy and fitness of the model. The optimal values obtained for the quality indices were 2.50mm thickness, 1.236g/m^3 density, 10.196% moisture content, 9.117MPa tensile strength and 29.047% elongation. The blending of different plasticizers with *tacca* starch helped to overcome the problem of brittleness associated with the use of *tacca* starch for films and made it attractive for the optimization of the films.

1. Introduction

Recent studies have shown that biodegradable films and films made from renewable and natural polymers such as starch, cellulose derivatives, gelatin, protein and lipids are receiving attention [1]. Biodegradable films are not meant to completely replace synthetic packaging films but they possess the ability to replace conventional packaging in some cases. Biodegradable films have the advantage of being completely degraded by microorganisms without emission of toxic gases [1].

Starch is one of the most studied natural polymers for the development of biodegradable films because of its low-cost carbohydrate polymer, easy to obtain and good ability to form films [2]. Thus, it is a potential

packaging material in agriculture, medicine, and packaging industries [3].

Tacca leontopetaloides also known as Polynesian arrowroot starch is a wild perennial herb belonging to the *Dioscoreaceae* species [4]. This plant is naturally distributed from Western Africa through Southern Asia to Northern Australia [5]. This starch serves as an important food source for many Pacific Island cultures and is additionally used to stiffen fabrics in some of the islands. Application of this starch was reported as a treatment of stomach ailments. It is also believed to treat diarrhea and dysentery [4].

The process of biodegradable film production involves the use of native and slightly modified starches whose advantages are that they are cheap, abundant and renewable. However, the use of starch itself in bio-plastic manufacturing is unsuitable because of various disadvantages [6]. These include the brittleness of the material in the absence of suitable plasticizers and the hydrophilic nature of starch. As a result, the mechanical properties deteriorate upon exposure to environmental conditions like humidity. Thus, starch needs to be blended with other synthetic polymers or plasticizers to eliminate these disadvantages.

Varieties of plasticizers to improve the processing properties and product performance of biodegradable films have been evaluated. Studies show that plasticizers used in promoting the plasticization of starch are glycerol, glycol, xylitol, sorbitol, sugars, and amides, such as urea, formamide and ethylenebisformamide [7].

Despite the considerable contribution of petroleum-based plastics to the global economy exceeding billions of dollars, their non-degradability represents a huge challenge for the ecosystem, leading to many environmental crises [8]. Concentrated efforts toward the development of biodegradable materials with properties that ensure food safety and security while minimizing the environmental impact of their uses have been the focus of different studies. Most of these biodegradable polymers exhibit excellent properties comparable to those of petroleum-based plastics but their applications in food packaging are limited by certain poor barrier and mechanical properties [9].

Studies have been carried out using common starches such as corn, potato, cassava and wheat [10]. However, these starches have important roles in the human food hierarchy system and large industrial production of starch has been focused on these crops. Therefore, there is a need to find alternative sources of starch among the underutilized crops and to apply novel technologies in extending their applications in more special or severe circumstances.

The advantages of starch for bio-plastic production include its renewability, good oxygen barrier in the dry state, abundance, low cost and biodegradability. *Tacca* starch in which the percentage of amylose to amylopectin is quite similar to corn and cassava starch among other properties, promises to provide elastic properties and become an alternative to common starch in the production of biodegradable film. Adversely, native starch-based materials are reported in many investigations to be very brittle with many surface cracks and are difficult to handle. However, these drawbacks can be resolved by the addition of plasticizers to pure starch to improve its workability and suppress film brittleness [11]. Thus, the blending of different plasticizers with tacca starch seems attractive for the optimization of the film in different aspects [12]. This study investigated the use of *tacca* starch for use in the production of biodegradable films which can be utilized for food packaging; starch was extracted from *tacca* crop and biodegradable films were developed in combination with different blends of plasticizers (Glycerol and Sorbitol) using different process temperatures. The physical (thickness, density and moisture content) and mechanical properties (tensile strength and elongation) of the film were determined. The individual and interactive effects of the process parameters on the films were determined; the plasticizers and process temperatures were also optimized.

2. Materials and methods

2.1 Materials

The material used for starch production was *Tacca* tubers obtained from a local market in Shendam Local Government Area of Plateau State, Nigeria. Other materials for the starch extraction were knife, bowl, grinder, sieves, filter clothes and distilled water. Sorbitol and glycerol were purchased from SIM BEST Scientific and Chemicals Minna, Niger State.

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2.2 Methods

2.2.1 Starch preparation

The *Tacca* tubers were peeled and washed with potable water. This was followed by mechanical grating of the tuber. The grated *Tacca* was mixed with water (3 times the volume of the grated *tacca*). The mixture was sieved and filtered using coarse sieve and filter clothes respectively. Thereafter, the filtrate was allowed to settle for twelve hours. This process is known as starch washing. At the end of the twelfth hour, it was decanted. The wet starch was dewatered manually and oven-dried using Mqeco-4t4030 oven. It was dried at a temperature of 105°C for twelve hours. This was to make sure that the starch was in the barest minimum moisture content of 12% [13]. The flowchart for the starch preparation is shown in Fig. 1.

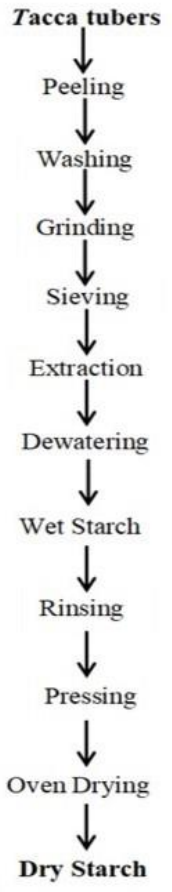

Figure 1. Flow Chart for *Tacca* starch preparation [13]

2.2.2 Film preparation and experimental design

The method described by [14] was adopted with slight modifications. The *Tacca* starch films were produced by conventional solution-casting techniques at the Chemical Engineering Laboratory facility of the Ahmadu Bello University (ABU), Zaria. The formulations were done based on the experimental design (Table 1).

Table 1. Box-Behnken's experimental design for formulation

Runs	A Starch w/w	B Sorbitol w/v	C Glycerol w/v	D Temp ^{0}C
1	10	4.5	2.25	95
2	5	2.25	4.5	85
3	10	2.25	4.5	95
4	5	4.5	2.25	85
5	10	2.25	2.25	85
6	10	0	2.25	75
7	15	2.25	0	85
8	5	2.25	0	85
9	10	2.25	0	95
10	10	0	2.25	95
11	15	2.25	2.25	95
12	10	4.5	4.5	95
13	15	0	2.25	85
14	5	2.25	2.25	85
15	10	0	0	75
16	15	2.25	4.5	85
17	10	2.25	2.25	85
18	10	4.5	2.25	75
19	15	4.5	2.25	85
20	10	2.25	2.25	85
21	10	2.25	4.5	75
22	5	0	2.25	85
23	10	2.25	2.25	85
24	10	4.5	0	85
25	10	0	4.5	85
26	15	2.25	2.25	75
27	10	2.25	0	75
28	5	2.25	2.25	95
29	10	2.25	2.25	75

The experiments were conducted using Box Behnken experimental design for investigating the individual and interactive effects of ingredient proportions (tacca starch, glycerol and sorbitol concentration) and process temperature on the film thickness, density, moisture content, tensile strength and elongation of Tacca starch-based film in which 29 different trials were conducted. Box-Behnken is a spherical, revolving response surface methodology design that provides an efficient solution compared with a three-level full-factorial design and reduces the number of required experiments, which becomes more significant as the number of factors increases [15].

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5-15g (w/w) of Tacca starch was dissolved in 100ml distilled water. This was followed by heating the film forming solution at a temperature range of 75°C to 95°C (75°C, 85°C and 95°C) for 15 minutes under constant stirring in a water bath. Temperature control was achieved by putting off the heat when the thermometer indicated the required temperature measurement. This step helped to provide homogeneous dispersion by disintegrating the starch granules. Thereafter, the different plasticizers were added into the dispersions at 0 - 4.5g (w/v) of each plasticizer (Glycerol and Sorbitol) representing 0-30% (w/w starch basis). The heating process for each was continued for an additional 15 minutes for each temperature for proper gelatinization. The gelatinized film solutions were cooled down, prior to their casting in glass petri-dishes. The glass petri-dishes served as casting surfaces, enabling the film to have a smooth and flat surface. The fresh casted films were placed in an oven (45°C) to allow evaporation. After 36 hours of drying and a constant weight, films were peeled from the casting surfaces and stored in desiccators with 53% relative humidity (RH). The selection of the above levels of the filmogenic components were based on preliminary studies and reviewed literature [16, 17]. The film preparation process is shown in Fig. 2. The formulation, heating and casting of the films are shown in Plate I.

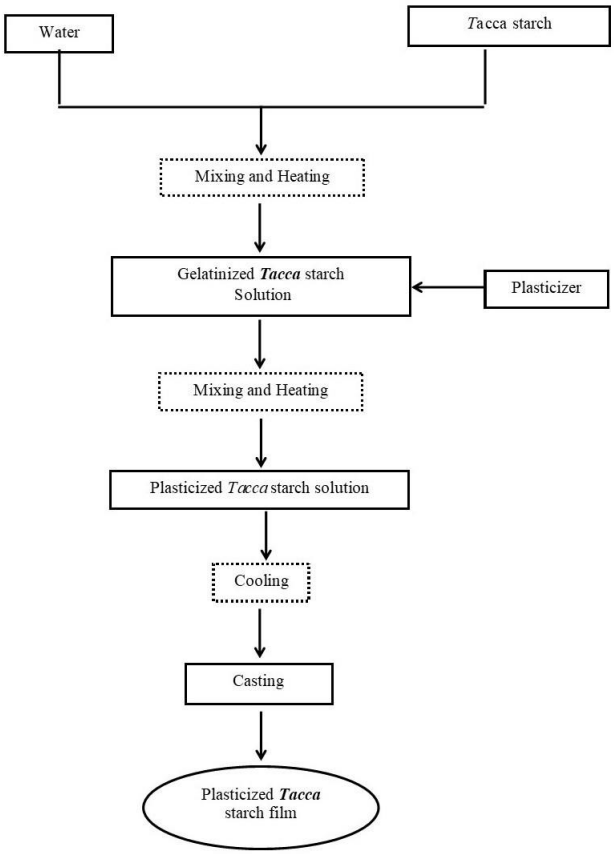

Figure 2. Flow chart for film preparation [14]

Plate I. (a) Film formulation and heating (b) Casting and oven dried film samples

2.3 Determination of film properties

The following properties of the Tacca starch film were investigated: Physical (thickness, density, and moisture content) and Mechanical (tensile strength and elongation). Film thickness, density and moisture content were determined according to the method described by [18] with slight modifications. Tensile strength and elongation were determined using a standard method of [19].

2.4 Statistical analysis

Statistical Analysis of variance (ANOVA) was done to study the effects of the different independent parameters on all dependent variables by Response Surface Methodology (RSM) using statistical design software (Design Expert 11 version 27. 3D).

2.5. Optimization and verification of models

Numerical optimization based on the desirability function using Design expert software was used to analyze the responses for obtaining optimum process parameters. Model equations were developed for each response to calculate the optimum condition for both dependent and independent variables.

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Table 2. Physical and mechanical properties of the Tacca starch-based films

Runs	A (w/w)	B (w/v)	C (w/v)	D (⁰C)	T (mm)	F (g/cm)	Mc %	Ts (MPa)	E (%)
1	10	4.5	2.25	95	1.624	1.05	5	3.051	11.735
2	5	2.25	4.5	85	1.28	1	5	2.699	50.415
3	10	2.25	4.5	95	1.834	1.05	10	6.666	28.578
4	5	4.5	2.25	85	0.85	1	5	1.418	52.35
5	10	2.25	2.25	85	1.648	1.05	10	6.086	17.514
6	10	0	2.25	75	1.42	1.05	10	5.67	3.91
7	15	2.25	0	85	2.504	1.2	10	8.35	5.38
8	5	2.25	0	85	0.892	1.01	5	7.95	6.42
9	10	2.25	0	95	1.284	1.05	10	9.13	2.57
10	10	0	2.25	95	1.092	1.05	10	6.34	2.41
11	15	2.25	2.25	95	3.386	1.1	15	6.649	14.514
12	10	4.5	4.5	95	1.532	1.04	10	2.948	8.943
13	15	0	2.25	85	2.784	1.2	10	8.37	2.68
14	5	2.25	2.25	85	1.45	1	5	2.225	53.174
15	10	0	0	75	1.43	1.04	10	16.15	2.041
16	15	2.25	4.5	85	1.406	1.2	10	1.151	9.683
17	10	2.25	2.25	85	1.676	1.05	10	1.006	20.14
18	10	4.5	2.25	75	1.772	1.04	10	3.501	24.251
19	15	4.5	2.25	85	3.366	1.09	10	4.455	25.152
20	10	2.25	2.25	85	2.136	1.11	10	1.071	21.14
21	10	2.25	4.5	75	2.042	1.1	10	8.657	29.523
22	5	0	2.25	85	1.284	1	5	1.365	20.51
23	10	2.25	2.25	85	2.104	1.05	10	1.199	12.945
24	10	4.5	0	85	2.564	1.11	10	1.52	9.886
25	10	0	4.5	85	2.648	1.05	10	2.124	46.62
26	15	2.25	2.25	75	2.578	1.15	10	6.086	17.514
27	10	2.25	0	75	0.59	1.04	10	8.35	16.36
28	5	2.25	2.25	95	1.244	1	10	4.33	46.143
29	10	2.25	2.25	75	1.53	1.05	10	1.006	20.14

Where: A = starch, B = sorbitol, C = glycerol, D = temperature, T = thickness, F = density, Mc = moisture content, Ts = tensile strength, E = elongation.

3. Results

The mean results of the film thickness, density, moisture content, tensile strength and elongation properties of Tacca starch-based film are presented in Table 2. These parameters were chosen among other quality indices due to their important roles in determining the handling, workability as well as the shelf-life of biodegradable films.

4. Discussion

The influence of the concentration of Tacca Starch (A), Sorbitol (B), Glycerol (C) and Temperature (D) over the physical (film thickness, density and moisture content) and mechanical (tensile strength and elongation) properties of Tacca Starch biodegradable films were analyzed through the Response Surface Methodology (RSM) using the Box-Behnken Design (BBD). The different formulations of the Box-Behnken's experimental design and the response variables are obtained for each combination as shown in Table 2. The developed models were investigated for adequacy and fitness between process variables and response variables for the Tacca starch biodegradable film as shown in Tables 3 to 8.

4.1 Thickness (mm)

Table 3 showed that only the mass concentration of tacca starch (A) significantly ($P < 0.05$) affected the film thickness. Plasticizer (Sorbitol and Glycerol) blends and process temperature had no significant ($p > 0.05$) effects on the film thickness. The kind and concentration of plasticisers play a significant part

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Table 3. Summary Statistics of the Thickness of TSF

Source	F-value	p-value	R^2	Adjusted R^2	Predicted R^2	Remarks
Thickness (mm)						
Model	6	0.0017	0.72	0.6867	0.6077	Significant
A-starch	22.97	<0.0001				
B-sorbitol	0.311	0.5822				
C-glycerol	0.6162	0.4401				
D-temperature	0.1057	0.748				

Table 4. Summary Statistics of the Density of TSF

Source	F-value	p-value	R2	Adjusted R2	Predicted R2	Remarks
Density (g/cm-3)						
Model	17.54	< 0.0001	0.7451	0.7027	0.6157	Significant
A-starch	69.36	< 0.0001				
B-sorbitol	0.2887	0.596				
C-glycerol	0.008	0.9294				
D-temperature	0.5133	0.4806				

towards attaining successful plasticization [20].

Fig. 3a shows that film thickness is well augmented with the higher level of starch and this effect is attributed to the formation of intermolecular hydrogen bonds between starch and dry matter content as well as the interaction between polysaccharides. This inference is drawn on the basis that plasticizer blends did not influence the film thickness since there was negligible significant difference in the film thickness variation in plasticizer concentration and process temperature. This real effect on the film thickness might be due to the large amount of the mass of starch with a larger surface area, which improves when interacted with plasticizers [21].

A linear model was obtained for the film thickness as follows:

$$\textit{Thickness (T)} = 0.356787 + 0.150400A + 0.038889B + 0.054741C + 0.005100D \quad (1)$$

From the above model equation, the regression coefficient depicts a rise in the film thickness as the starch level (A) increased by 0.1504 (codded value). From Table 4, the coefficient of determination (R^2) of 0.7200 designates a moderate correlation between the experimental and predicted values. Fig 1b also attests to the correlation between the predicted and actual values. The predicted R^2 value of 0.6867 is in reasonable agreement with the adjusted R^2 value of 0.6077. This implies that the model can be used to navigate the design space.

Furthermore, the model F-value of 6.00 and a corresponding p-value of <0.0017 implies the model is significant indicating its suitability for packaging. According to the Indian Government Packaging Standard, packaging films must be at least 0.05 mm thick [22]. The film thickness is an important parameter which influences both the mechanical properties and WVP of the biodegradable film [23]. Fig 3 is the 3-D response plot for film thickness and the comparison between predicted and experimental values for TSF for the quality index.

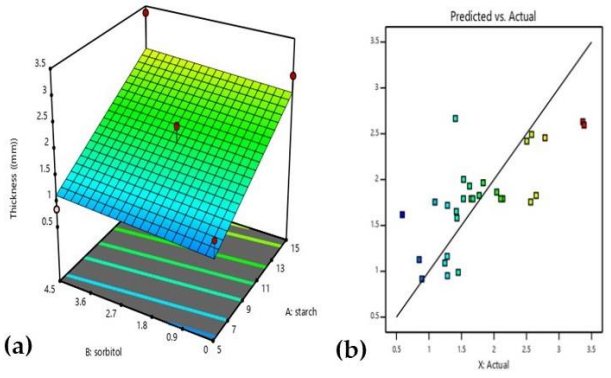

Figure 3. (a) Response surface plot (3-D) for film thickness and (b) comparison between predicted and experimental value for film thickness

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Table 5. Summary Statistics of the Moisture content of TSF

Source	F-value	p-value	R^2	Adjusted R^2	Predicted R^2	Remarks
Moisture content (%)						
Model	6.39	0.0012	0.6159	0.5352	0.5134	Significant
A-starch	24.23	< 0.0001				
B-sorbitol	0.6731	0.02				
C-glycerol	0	0.041				
D-temperature	0.6731	0.42				

4.2 Density g/cm³

Table 4 reveals that the concentration of plasticizer (B and C) blends and process Temperature (D) showed no significant ($p > 0.05$) effects on the film density.

Only the mass concentration of tacca starch (A) had significant effect on the film density at 5% level of significance. This is similar to a report by [24] that no significant effect was observed in density by adding glycerol plasticizer in corn starch film. This can be ascribed to change in film formulation that caused a simultaneous rise in volume, which increased the thickness of the film, hence, no significant difference in film's density. The empirical relationship between the observed experimental results and input effects was expressed by a linear equation fitted according to the experimental design used in this study as follows:

$$\textbf{\textit{Density}} = 0.974052 + 0.015500A - 0.002222B - 0.000370C - 0.000667D \quad (2)$$

The regression coefficients as depicted by the above model equation showed a rise in density as starch level (A) increased at constant plasticizer blends and process temperature. On the other hand, a decrease in density results from increase in plasticizer concentration and process temperature at a constant mass concentration of tacca starch.

From Table 4, the R^2 of 0.7451 and the plots in Fig. 4 showed a good correlation between the experimental and predicted values, revealing the adequacy and fitness of the model. Also, there was a reasonable agreement between the adjusted R^2 (0.7027) and the predicted R^2 (0.6157) for the film density. The model F-value of 17.54 and a corresponding p-value of <0.0001 implies that the model is significant and can be used to navigate the design space.

4.3 Moisture content (%)

The influence of mixture components and process temperature as indicated by ANOVA (Table 5)

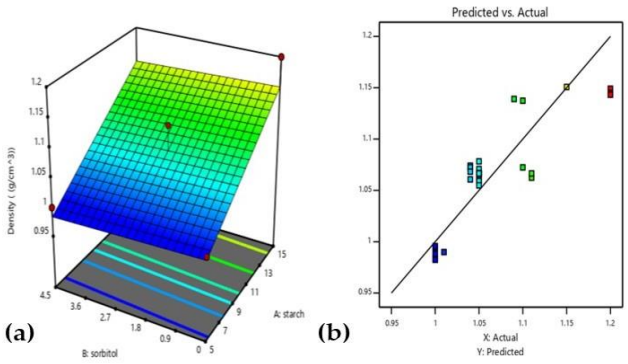

Figure 4. (a) Response surface plot (3-D) for film density and (b) comparison between predicted and experimental value for film density.

showed that the mass concentration of Tacca starch (A) and plasticizer (B and C) blends had significant ($p<0.05$) effects on the film moisture contents while process temperature (D) had no significant ($p>0.05$) effect on the film moisture content. This implies that both starch and plasticizers blends have a real or true effect on the film moisture content in the research study while the effects exerted by the process temperature and solely by chance or some random factors.

Similar results were reported by [25] and [26] for glycerol and Sorbitol as plasticizers. Also, a high concentration of plasticizers favors the adsorption of water molecules due to their hydrophilic nature. This may be due to the hydrophilic nature of both starch and the plasticizers which assists in the formation of hydrogen bonding with free OH groups [27]. Water acts as a solvent and causes textural degradation, chemical and enzymatic reactions. Also, the water activity of food is an important parameter in relation to the shelf life of the food. Therefore, in low moisture food, a low level of water activity must be maintained to minimize the deterioration, chemical and

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Table 6. Summary Statistics of the Tensile Strength of TSF

Source	F-value	p-value	R^2	Adjusted R^2	Predicted R^2	Remarks
Tensile strength						
Model	3.62	0.011	0.7836	0.6671	0.6073	Significant
A-starch	3.47	0.0837				
B-sorbitol	8.16	0.0127				
C-glycerol	11.29	0.0047				
D-temperature	0.0429	0.8389				
AB	0.7206	0.4102				
AC	0.1737	0.6832				
AD	0.1088	0.7464				
BC	10.93	0.0052				
BD	0.0574	0.8141				
CD	0.3514	0.5628				
A^2	0.2543	0.6219				
B^2	0.5748	0.4609				
C^2	11	0.0051				
D^2	6.92	0.0198				

enzymatic reactions and prevent textural degradation. The empirical relationship between the observed experimental results and input effects was expressed by a linear equation fitted according to the experimental design used in this study as follows:

$$\textit{Moisture Content} = +1.01293 + 0.500000A - 0.185185B - 1.99244C + 0.041667D \quad (3)$$

The model coefficients reveal a decrease in moisture content as a result of increase in sorbitol (B) and glycerol (C) as depicted by -0.18518 and -1.99244 (coded) respectively when other variables are kept constant. Conversely, the model coefficients of 0.500 and 0.041 for starch level (A) and process temperature (D) reveal an increase in moisture content due to their increase, when other variables remain constant. The model R^2 value of 0.6159 and the plot in Fig 5 showed the correlation between the experimental and predicted values. Also, the adjusted R^2 and predicted R^2 of 0.5352 and 0.5124 respectively, implies a good fit for the model. The model F-value of 6.39 and a corresponding p-value of 0.012 implies the model is significant.

4.4 Tensile strength (MPa)

Table 6 revealed that plasticizer (B and C) blends, and their interaction (BC) exerted a significant ($p < 0.05$) effect on the tensile strength of the biodegradable film while starch level (A) and process temperature (D) had no significant effect ($p > 0.05$) on the tensile

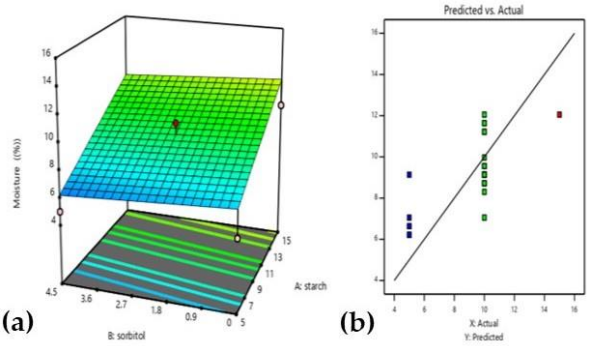

Figure 5. (a) Response surface plots (3-D) for film moisture content and (b) comparison between predicted and experimental value for film moisture content.

strength of the Tacca starch biodegradable film at 5% level of significance. On the other hand, the quadratic terms of glycerol (C^2) and temperature (D^2) had significant effects ($P < 0.05$) on the tensile strength of the biodegradable film. The possible reason for the real effect of plasticizer concentration on the tensile strength of the biodegradable film is the domination of strong hydrogen bonds produced by starch–starch intermolecular interaction over starch–plasticizer attraction. This phenomenon can be explained through the role of plasticizers in diminishing the strong intra-molecular attraction between the starch chains and promoting the formation of hydrogen bonds between plasticizers and starch molecules. [28] reported similar observation that glycerol plasticizer

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induces great effect on the tensile strength starch based biodegradable films.

Tensile strength was found to have a quadratic relationship with the process variables as per the following equation:

$$\textit{Tensile Strength} = +168.62540 + 0.832190A - 1.25251B - 2.38020C - 3.91480D - 0.088178AB - 0.043289AC - 0.007710AD - 0.763160BC - 0.012444BD - 0.030789CD + 0.018510A^2 + 0.137430B^2 + 0.601208C^2 + 0.024136D^2 \quad (4)$$

From the empirical model, the quantitative effects of the individual mixture components and process parameter (temperature) as obtained from the above regression coefficients infer that an increase in either plasticizer (B and C) blends, process temperature (D), the interaction between starch level and sorbitol (AB), the interaction between starch level and glycerol (AC), the interaction between starch level and temperature (AD), the interaction between the plasticizers (BC), interaction between sorbitol and temperature (BD) or the interaction between glycerol and temperature (CD) would lead to a decrease in the tensile strength of the biodegradable film. On the other hand, the model coefficients also imply an increase in the tensile strength for any increase in starch level (A), and the quadratic terms of starch (A^2), sorbitol (B^2), glycerol (C^2) or temperature (D^2) when other variables were kept constant.

The model F-value of 3.62 and a corresponding p-value of 0.0110 implies the model is significant. Also, the coefficient of determination R^2 value of 0.7836 implies a good correlation between predicted and experimental values as depicted by the plot in Fig. 6. The adjusted R^2 of 0.6671 and the predicted R^2 of 0.6073 showed an agreement within the values and good fit for the model.

A good tensile strength is very essential as poor tensile strength can lead to premature cracking during production, handling, storage or use [29]. Tensile strength measures the maximum weight or pressure the film can withstand before breaking. The tensile strength of starch based novel biodegradable films to range between 1.5 – 2.3kg/cm^3 [29].

4.5 Elongation at break (%)

Analysis of variance (ANOVA) showed that the elongation of the TSF was significantly ($p < 0.05$)

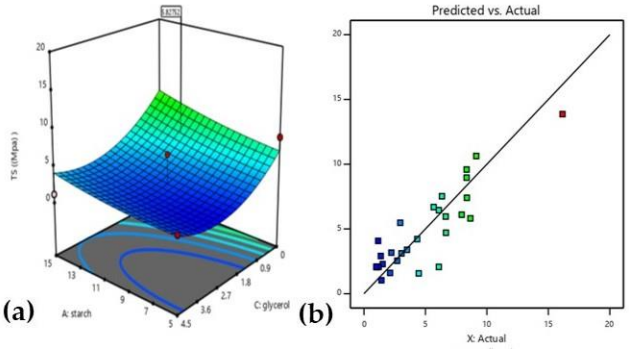

Figure 6. (a) Response surface plot 3-D for TS of TSF and (b) plot of predicted and experimental values for film tensile strength.

affected by the mass concentration of starch (A) and glycerol plasticizer (C). The concentration of sorbitol plasticizer (B) and process temperature (D) had no significant effects ($p > 0.05$) on the film elongation at 5% level of significance. Similar results were obtained by [30] for the effect of sorbitol and glycerol on sweet potato starch film. Glycerol plasticized film showed the highest value in elongation percentages. Glycerol has a smaller effective hydrodynamic radius (0.31nm) than sorbitol (0.39nm) and therefore, the distance between starch molecules in glycerol plasticized film would be the farthest. The higher values in the elongation percentage for glycerol-plasticized film can be associated with the fact that it has a smaller molecular chain which can enter the polymer network more easily than sorbitol [31]. The desired elongation or flexibility of the biodegradable film depends on their intended applications and subsequent transportation, handling and storage of packaged foods.

The empirical relationship between the observed experimental result and input effect was expressed by the simple linear equation fitted according to the experimental design. The model equation for the film elongation is:

$$\textit{Elongation} = +57.80557 - 2.56815A + 2.00541B + 4.85574C - 0.32318D \quad (5)$$

From the model coefficients of regression, the quantitative terms of the effects of starch level (A), sorbitol (B), glycerol (C) and process temperature (D) were – 2.5681, +2.0054, +4.8557 and -0.3231 (coded) respectively. The coefficients infer that an increase in either starch level (A) or process temperature (D) tends to a corresponding decrease in the elongation of

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Table 7. Summary Statistics of the Elongation of TSF

Source	F-Value	P-Value	R^2	Adjusted R^2	Predicted R^2	Remarks
Elongation						
Model	6.89	0.0008	0.7346	0.657	0.65748	Significant
A-starch	14.43	0.0009				
B-sorbitol	1.78	0.1945				
C-glycerol	10.44	0.0036				
D-temperature	0.9139	0.3486				

Table 8. Optimization constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A: starch	is in range	5	15	1	1	3
B: sorbitol	is in range	0	4.5	1	1	3
C: glycerol	is in range	0	4.5	1	1	3
D: temperature	is in range	75	95	1	1	3
Thickness	maximize	0.59	3.386	1	1	3
Density	maximize	1	1.2	1	1	3
Moisture	minimize	5	15	1	1	3
TS	maximize	1.006	16.15	1	1	3
Elongation	maximize	2.041	53.174	1	1	3

TSF while an increase in plasticizers (B and C) tends to increase TSF elongation.

The coefficient of correlation R^2 value of 0.7346 and the plot (figure implies a good correlation between the predicted and experimental values. The adjusted R^2 value of 0.6570 and the predicted R^2 value of 0.6578 implies a good fit. The model F-value of 6.89 and a corresponding p-value of 0.0008 implies the model is significant (Table 7).

The higher the elongation at break of a biodegradable film, the better the film quality with good tensile strength. Films with less than 5% elongation are said to be brittle. However, all the films showed elongation at the break above 5%, showing a flexible nature.

Optimization was carried out on the basis of the following objectives; Minimize moisture content, Maximize Tensile strength, maximize elongation, maximize density and Maximize thickness as shown in Table 8. The film is intended to be used for food packaging applications. The optimization solution for the desirability of TSF is presented in Table 9.

The optimal value was selected based on the set goals (Table 9) and the best solution that met these goals. The optimal levels of process parameters were obtained to be 13.782g of starch, 4.5g of glycerol, 4.5g of sorbitol and 75^{0}C process temperature resulting in a thickness of 2.5mm, density of 1.126gcm^{-3}, moisture

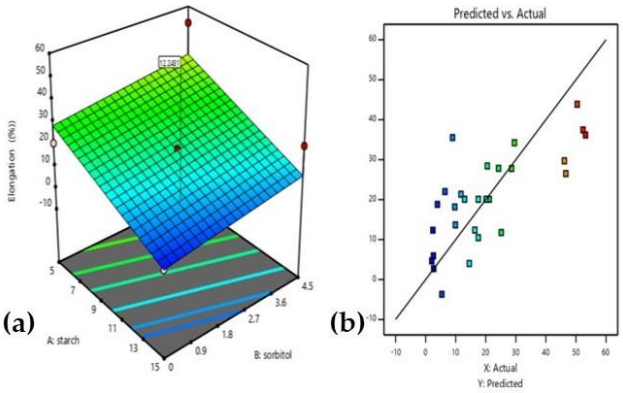

Figure 7. (a) Response surface plot of tacca starch biodegradable films and (b) comparison between predicted and experimental value for film Elongation.

content of 10.196%, 9.117MPa tensile strength and 29.047% elongation obtained with a desired value of 0.609 as obtained from 100 optimization solutions (Table 9).

All the inferences made from this study are only valid within the experimental variables ranges which are starch (5w/w – 15w/w), sorbitol (0w/w – 4.5w/w), glycerol (0w/w – 4.5w/w) and temperature (75^{0}C – 95^{0}C).

5. Conclusions

Results showed that the optimal formulation for the production of Tacca starch biodegradable film was 13.728g of starch, 4.5g glycerol, 4.5g sorbitol and 75^{0}C

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Table 9. Numerical optimization desirability solution for the TSF

S/N	Starch	Sorbitol	Glycerol	Temp	Thick	Den	Mc	TS	E	D
1	**13.782**	**4.5**	**4.5**	**75**	**2.52**	**1.126**	**10.196**	**9.117**	**29.047**	**0.609**
2	13.553	4.5	4.5	75	2.485	1.122	10.081	9.078	29.635	0.609
3	13.735	4.498	4.5	75	2.513	1.125	10.172	9.106	29.164	0.609
4	14.217	4.5	4.5	75	2.585	1.133	10.413	9.195	27.932	0.609
5	14.311	4.499	4.5	75	2.599	1.134	10.46	9.212	27.688	0.608
.										
98	8.706	0	1.052	95	1.495	1.045	9.324	11.507	9.854	0.404
99	8.541	0	1.05	94.816	1.469	1.043	9.234	11.349	10.329	0.404
100	8.523	0	0.937	95	1.461	1.042	9.233	11.956	9.764	0.403

process temperature. The optimal values obtained for the film properties were: 2.50mm thickness, 1.236g/m^3 density, 10.196% moisture content, 9.117MPa tensile strength, and 29.047% elongation with a desirability value of 0.609. Tacca starch level had a significant effect ($p < 0.05$) at 5% level of significance on all the response variables with the exception of tensile strength. Linear regression models were developed from the experimental data for film thickness, density and moisture content, while film solubility and tensile strength models were both quadratic models. From the regression models, there were notable interactions of mixture variables and process temperature on the tensile strength of the biodegradable films. The blending of different plasticizers with *tacca* starch helped to overcome the problem of brittleness associated with the use of *tacca* starch for films and made it attractive for the optimization of the films. Further studies should be conducted on the biodegradability of the *tacca* starch film; also, the potential use of the film for food packaging should be investigated.

Authors' contributions

Conceptualization, B.A.O.; Methodology, B.A.O.; Software, S.S.O.; Validation, S.S.O.; Formal analysis, B.A.O and S.S.O.; Investigation, S.S.O.; Resources, B.A.O and S.S.O.; Data curation, S.S.O., Data, Preparation, S.S.O.; Writing original draft, Preparation, S.S.O.; Writing-review and editing, B.A.O.; Visualization, B.A.O.; Supervision, B.A.O.; Project administration, B.A.O and S.S.O.

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Availability of data and materials

All data will be made available on request according to the journal policy.

Conflicts of interest

The authors declare no conflict of interest.

References

1. Abdou, E.S.; Sorou, M.A. Preparation and Characterization of starch/carrageen edible films. Int. Food Res. J. 2014, 21(1), 189-193.
2. Srikulkit, K.; Bangyekan, C.; Aht-Ong, D. Preparation and properties evaluation of Chitosan coated cassava starch film. Carbohyd. Pol. 2006, 63, 61-71. https://doi.org/10.1016/j.carbpol.2005.07.032.
3. Bertuzi, M.A.; Vidaure, E.F.C.; Amada, M.; Gottifredi, J.C. Water vapour permeability of edible starch-based films. J. Food Eng. 2007, 80(3), 972-978. https://doi.org/10.1016/j.jfoodeng.2006.07.016.
4. Ukpabi, U.J.; Ukenye, E.; Olojede, A.O. Raw material potentials of Nigerian wild Polynesian Arrowroot (*Taccaleontopetaloides*) tubers and starch. J. Food Technol. 2009, 7, 135-138.
5. Ubwa, S.T.; Anhawange, B.A.; Chia, J.T. Chemical analysis of Tacca leontopetaloides peels. Am. J. Food Technol. 2011, 6, 932-928. https://doi.org/10.3928/asfj. 932-938.
6. Kalambur S.; Rizvi, S.H. An overview of starch based plastic blend from reaction extrusion. J. Plastic Film.

https://doi.org/10.58985/jafsb.2024.v02i01.38

Sheet. 2006, 22(1), 39-58. https://doi.org/10.1177/8756087906062729.

7. Dai, H.; Chang, P.R.; Geng, F.; Yu, J.; Ma, X. Preparation and properties of starch-based film using N, N-bis (2-hydroxyethyl) formamide as a new plasticizer. Carbohyd. Pol. 2010, 79(2), 306-311. https://doi.org/10.1016/j.carbpol.2009.08.012.
8. Ilyas, R.A.; Sapuan, S.M.; Ishak, M.R.; Zainudin, E.S. Effect of delignification on the physical, thermal, chemical, and structural properties of sugar palm fibre. Bio Res. 2017, 12(4), 8734–8754. https://doi.org/10.15376/biores.12.4.8734-8754.
9. Angellier, H.; Choisnard, L.; Mollina-Boisseau, S. Optimization of the preparation of aqueous suspension of waxy maize starch nanocrystals using a response surface methodology. Biomacromolecules. 2004, 5, 1545-1551. https://doi.org/10.1021/bm049914u.
10. Hirpara, N.; Dabhi, M.; Rathod, P.J. Development of potato starch based biodegradable packaging film. J. Food Proc. Technol. 2021, 13, 529-541.
11. Dias A.B.; Muller C.M.; Laraotonda F.D.; Laurindo J.B. Biodegradable films based on rice starch and rice flour. J. Cereal Sci. 2010, 51(2), 213-219. http//doi.org/101916/jcs.2032.11.014.
12. Vieira, M.G.; Da Silva, M.A., Dos Santos, L.O. Natural-based plasticizers and biopolymer films: A review. Eur. Pol. J. 2011, 47, 254-263. http://dx.doi.org/10.1016/j.eurpolymj.2010.12.011.
13. Walid, A.; Sapuan, S.M.; Sultan, M.T.H.; Alkbir, M.F.M.; Ilyas, R.A. Development and characterization of cornstarch-based bioplastics packaging film using a combination of different plasticizers. Polymers. 2021, 13(20), 3487. http://dx.doi.org/10.3390/polym13203487.
14. Ezeoha, S.L.; Ezenwanne, J.N. Production of biodegradable plastic packaging film from cassava starch. Department of agricultural and bioresources engineering, University of Nigeria, Nsukka. 2013, p-ISSN: 2278-8719. 3(10).
15. Mohammad, H.S.; Morteza, H.; Mohsen, J.; Ghasem, N.D. Optimization of encapsulated clove oil particle size with biodegradable shell using design expert methodology. Pak. J. Biotechnol. 2015, 12(2), 149-160. https://pjbt.org/index.php/pjbt/article/view/158.
16. Dutta, P.K.; Tripathi, S.; Mehrotra, G.K.; Dutta, J. Perspectives for chitosan based anti-microbial films in food applications. Food Chem. 2009, 114, 1173-1182. https://doi.org/10.1016/j.foodchem.2008.11.047.
17. Rhim, J.W. Effect of clay contents on mechanical and water vapor barrier properties of agar-based nanocomposite films. Carbohyd. Pol. 2011, 86, 691–699. https://doi.org/10.1016/j.carbpol.2011.05.010.
18. Tarique, J.; Sapuan, S.M.; Khalina, A. Effect of glycerol plasticizer loading on the physical, mechanical, thermal, and barrier properties of arrowroot (*Maranta arundinacea*) starch biopolymers. Sci. Rep. 2021, 11(1), https://doi.org/10.1038/s41598-021-93094-y.
19. ASTM. Designation D882-02: Standard Test Method for Water tensile strength properties of thin plastic sheeting. 2002, Annual book of ASTM standards Philadephia: American Society for Testing and Materials.
20. Tummala, P.; Liu, W.; Drzal, L.T.; Mohanty, A.K.; Misra, M. Ind. Eng. Chem. Res. 2006, 45, 7491–7496.
21. White, C.D.; Willis, B.J.; Narayanan, K.; Dutton, S.P. Identifying and estimating significant geologic parameters with experimental design. SPE J. 2001, 6(03), 311–324. https://doi.org/10.2118/74140-pa.
22. Wang, L.; Xie, B.; Shi, J.; Xue, S.; Deng, Q.; Wei, Y.; Tian, B. Physicochemical properties and structure of starches from Chinese rice cultivars. Food Hydrocoll. 2010, 24(2-3), 208-216. https://doi.org/10.1016/j.foodhyd.2009. 09. 007.
23. Marichelvam M.K.; Jawaid M.; Asim M. Corn and rice starch-based bio-plastics as alternative packaging materials. Fibers. 2019, 7, 32. https:// doi.org/10.3390/fib7040032.
24. Nordin, N.; Othman, S.H.; Rashid, S.A.; Basha, R.K. Effects of glycerol and thymol on physical, mechanical, and thermal properties of corn starch films. Food Hydrocoll. 2020, 106, 105884. https://doi.org/10.1016/j.foodhyd.2020.105884.
25. Shaikh, M.; Haidar, S.; Ali, T.M.; Hasnain, A. Physical, thermal, mechanical and barrier properties of pearl millet starch films as affected by levels of acetylation and hydroxypropylation. Int. J. Biol. Macromol. 2018, 124, 209–219. https://doi.org/10.1016/j.ijbiomac.2018.11.135.
26. Abdulrahman, A.B.; Mhammed, Z.H.; Abdoulhbdi, A.; Illyas, R.A.; Sapuan, S.M. Effects of various plasticizers in different concentrations on the physical, thermal and mechanical and structural properties of wheat-starch based films. Polymer. 2023. http//doi.org/10.3390/polymer15010063.
27. Singh, T.P.; Chatli, M.K.; Sahoo, J. Development of chitosan based edible films: process optimization using response surface methodology. J. Food Sci. Technol. 2014, 52(5), 2530–2543. https://doi.org/10.1007/s13197-014-1318-6.
28. Cerqueira, M.A.; Souza, B.W.S.; Teixeira, J.A.; Vicente, A.A. Effect of glycerol and corn oil on physicochemical properties of polysaccharide films-A comparative study. Food Hydrocoll. 2012, 27(1), 175-184. https://doi.org/10.1016/j.foodhyd.2011.07.007.

29. Muscat, D.; Adhikari, B.; Adhikari, R.; Chaudhary, D.S. Comparative study of film forming behaviour of low and high amylose starches using glycerol and xylitol as plasticizers. J. Food Eng. 2012, 109(2), 189-201. https://doi.org/10.1016/j.jfoodeng.2011.10.019.
30. Kocira, A.; Kozłowicz, K.; Panasiewicz, K.; Staniak, M.; Szpunar-Krok, E.; Hortynska, P. Polysaccharides as edible films and coatings: characteristics and influence on fruit and vegetable quality—a review. Agronomy. 2021, 11, 813. https://doi.org/10.3390/agronomy 11050813.
31. Lucio, B.M., Camen, P.C.; Ricardo, A.P. Effects of glycerol and sorbitol concentration on mechanical, Optical and barrier properties of sweet potato film. NFS J. 2020. http://doi.org/10.1016/jnfs.2020.002.

https://doi.org/10.58985/jafsb.2024.v02i01.38