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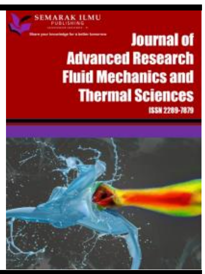

Optimization of Starch-Based Bioplastic from Sweet Potato using Box-Behnken Design with Plasticizer and Filler for Reduced Water Absorption

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ABSTRACT

Plastic is a ubiquitous material used in a wide range of applications, but its disposal has become a significant environmental concern. Bioplastics, which offer several advantages over conventional plastics, including a smaller carbon footprint, greater biodegradability, and adaptability, are being explored as a potential alternative. Among the bioplastics, starch-based polymers have gained significant attention due to their natural abundance, biocompatibility, low cost, and renewability. In this study, a starch-based bioplastic was developed using glycerol and sorbitol as plasticizers and wood dust as a filler through the casting method. Starch was extracted from sweet potato (*Ipomoea batatas*). The optimal conditions for preparing the bioplastic with minimum water adsorption were determined using a three-level Box-Behnken design. The amount of starch, plasticizer, and filler were the key parameters for this study. The percentage of water absorption was determined by immersing the prepared bioplastic in distilled water for 24 hours and weighing it. The result of the Fourier Transform Infrared (FTIR) spectroscopy of extracted starch showed a similar observation peak pattern when compared to the FTIR spectrum of commercial starch. The linear response model produced a good coefficient of determination (R^2 = 0.8237), indicating that the chosen parameters implicitly affect the percentage of water absorption. The prediction percentage of water absorption after optimization is 20.069 with a desirability value of 0.806. This study shows that the response surface approach helps in determining the optimum conditions for the minimum percentage of water adsorption from starch-based bioplastic.

1. Introduction

The production of synthetic organic polymers, known as plastics, derived from petroleum, is widely utilized in modern society and is ubiquitous in various sectors [1]. Despite their usefulness, petroleum-based plastics are notorious for their slow decomposition rate [2]. Plastic bags can take anywhere from 10 to 1000 years to break down in landfills, while disposable diapers can take up to 250 to 500 years, depending on the landfill conditions. In water, a single plastic bag may break down

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within 20 years, but it could take more than 500 years to degrade. The environmental degradation caused by plastic poses one of the most significant challenges to contemporary society and all forms of life on our planet. The problem of plastic pollution affects the whole world and is shared by both developed and developing nations, leading to harmful effects on habitats in landfills and oceans [3].

Biodegradable alternatives, such as bioplastic made from plant sources like corn starch, sweet potato starch, banana peel, and tapioca starch with plasticizer and filler, have been developed to address this issue. Biopolymers are highly versatile, resistant, and completely biodegradable, and are already widely used in agriculture, medicine, the textile industry, as well as the packaging and container market [4]. Among these plant sources, the sweet potato, or *Ipomoea batatas*, is a popular nutritional source in many diverse countries, including Peru, Japan, Tanzania, New Zealand, and Malaysia [5]. It is the only economically significant species in the Convolvulaceae family and is commonly known as a root vegetable with large, starchy, sweet-tasting tuberous roots that are similar in taste to pumpkin [5]. They are also a source of starch, which can be classified into three groups based on the rate of absorption: quickly digested starch (80%), slowly absorbed starch (9%), and resistant starch (11%), which functions like fiber and is beneficial for gut health. Allowing cooked sweet potatoes to cool can increase the amount of resistant starch [6]. The sweet potato was chosen as a starch source due to its high starch content of 60.1–71.4% [7]. The plasticizer in bioplastic enhances its extensibility, pliability, biodegradability, lifespan, durability, and other qualities, while the filler provides reinforcement [8,9].

Numerous techniques have been attempted in the past to remove starch from the plant, and the method for producing bioplastic has also varied. However, bioplastics made from starch still have a high percentage of water absorption under optimum conditions, which is around 44.02% as stated from previous study [10]. The Design-Expert software is employed to explore the design space and generate an optimized solution based on a desirability function. Response Surface Methodology (RSM) with the Design Expert Software (Version 8.0.6) was reported to have been utilized to effectively optimize the stability of arsenic in a solid matrix through coagulation-precipitation [11]. RSM is a statistical technique with considerable potential for enhancing process variables. RSM assists in determining the best operational parameters to maximize a system's performance [12]. RSM allows for the collection of a lot of data from a small number of experiments, which is one of its main advantages. Using models and graphical representations, the primary effects of factors and their interactions on the response can be examined. It discovers the factor levels that elicit the best response and the optimal conditions resulting from numerous responses. By utilizing Box-Behnken designs (BBD), which require fewer runs than a standard factorial technique, higher-order response surfaces are generated [13]. The study's results by previous researchers, highlight BBD able to have strong capability in detecting damage with fewer measurement points [14]. Careful experimental planning enables the estimation of the project duration, in addition to these benefits. Therefore, it is crucial to determine the optimum conditions for preparing bioplastic with a lower percentage of water absorption, and Box-Behnken Design (BBD) analysis is proposed as a suitable Design of Experiment (DOE) method to address this issue.

2. Methodology

2.1 Preparation of Filler

The wood dust was dried in the oven at 45°C for 2 hours. Then, the dried wood dust was blended in the dry blender and sieved to collect 75µm powder of wood dust.

2.2 Preparation of Starch

The sweet potato as shown in Figure 1(a) was scrubbed and sliced into manageable pieces for the blender. The sweet potato was then blended with 300 mL of distilled water into a paste. After filtering the paste to remove the solid debris, the solution could sit in the beaker for six hours. The starch was extracted from the beaker's bottom, and it was filtrated (Figure 1(b)) to separate the solution. Next, the extracted starch as shown in Figure 1(c) was left at room temperature for 24 hours. Then, the starch powder was stored in an airtight container for further usage. The functional group of prepared starch was characterized using Fourier Transform Infrared (FTIR) Spectroscopy.

(a) (b) (c)

Fig. 1. (a) Raw (b) Extracted starch (c) Wet starch of sweet potato

2.3 Preparation of Bioplastic

The amounts of starch and filler were weighted in accordance with Table 1. The sorbitol and glycerol were measured using the amounts listed in Table 1. The ratio of water to starch (1:5) was used to quantify distilled water. A beaker containing starch, filler, glycerol, sorbitol, and distilled water was cooked on a hot plate until the liquid turned gelatinous. The slurry was smeared onto the glass plate and allowed to come to room temperature for 48 hours. With caution, the dried bioplastic as depicted in Figure 2 was removed from the glass plate and placed into the sealed plastic container for storage.

Fig. 2. Dried bioplastic from sweet potato starch studies above

Table 1
Variables and experimental design level

Independent variables	Coded symbol	Coded levels in Box Behnken Design		
		-1	0	+1
Starch (g)	A	2	4	6
Plasticizer (Sorbitol: Glycerol) (mL)	B	1.6: 0	0.8: 0.8	0: 1.6
Filler (g)	C	0	0.2	0.4

The Box Behnken Design (BBD) design of the experiment (D.O.E.) was used to replicate the trial run. The design simulated a total of 17 distinct trials, each with three replicates, as can be seen in Table 2, to ascertain the extent of error resulting from chance.

Table 2
Design of experiment using BBD

Experiment	Starch (g)	Plasticizer (mL)	Filler (g)
1	-1	-1	0
2	+1	-1	0
3	-1	+1	0
4	+1	+1	0
5	-1	0	-1
6	+1	0	-1
7	-1	0	+1
8	+1	0	+1
9	0	-1	-1
10	0	+1	-1
11	0	-1	+1
12	0	+1	+1
13	0	0	0
14	0	0	0
15	0	0	0
16	0	0	0
17	0	0	0

2.4 Absorption of Water Testing

To verify that the prepared bioplastic did not include water, the 1.5 cm^2 prepared bioplastic was dried in the oven for two hours. After that, the bioplastic was left in the beaker with 50 mL of distilled water for a full day. The absorption of water test was conducted using the American Society for Testing and Materials (ASTM) D570 standard. Following a 24-hour period, water was filtered to extract the bioplastic, which was subsequently weighed to determine its ultimate weight. The dependent variable was the volume of water absorbed. Utilizing Design Expert® software (version 13, Stat-Ease, USA), the testing runs were simulated and the water absorption % was optimized. To determine the amount of random errors, a total of 17 experiments—three of which were duplicates—were included in the experimental design. Both the trial design and the statistical analysis of the data were conducted using BBD.

3. Results

3.1 Preparation of Starch

Figure 3 shows FTIR spectra observations. The FTIR spectra reveal that the extracted starch's peaks roughly follow the same pattern as commercial starch. A starch group (C=O) band was seen at 1206 cm^{-1} for both spectra. The broad absorption peak of the band at 3275 cm^{-1} indicates the existence of the -OH group. Additional functional groups associated with starch include C=C at wavelengths of 1637 cm^{-1} and 1077 cm^{-1}, and C-H bonds at wavelengths of 1418 and 2929 cm-1, respectively. Previous investigations have observed similar extracted starch observation peaks [15,16].

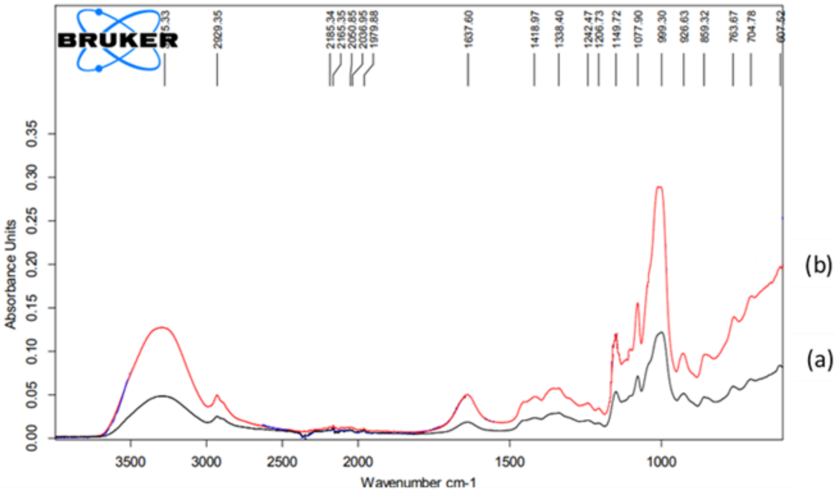

Fig. 3. FTIR spectra of (a) Commercial starch and (b) Extracted starch from sweet potato

3.2 Absorption of Water Testing

The percentage absorption of water has been observed from the soaked bioplastic in distilled water based on the ASTM D570 standard method for plastics. Starch, plasticizer, and filler are the variables that influenced the percentage absorption of water. Eq. (1) shows the equation used to determine the percentage of water absorption, meanwhile, Table 3 shows the result obtained by using the Box-Behnken Design (BBD) approach.

$$\text{Absorption of water (\%)} = \frac{W2-W1}{W1} \text{ x } 100 \quad (1)$$

where W_2 is the wet(final) weight, meanwhile W_1 is the conditioned (initial) weight.

Table 3
Design and results for BBD analysis

Experiment	Starch (g)	Plasticizer (mL)	Filler (g)	Percentage absorption of water (%)
1	2	0	0.2	28.73
2	6	0	0.2	46.28
3	2	1.6	0.2	27.52
4	6	1.6	0.2	74.73
5	2	0.8	0	25.49
6	6	0.8	0	68.63
7	2	0.8	0.4	26.88
8	6	0.8	0.4	69.85
9	4	0	0	49.06
10	4	1.6	0	49.84
11	4	0	0.4	42.05
12	4	1.6	0.4	53.67
13	4	0.8	0.2	37.74
14	4	0.8	0.2	44.71
15	4	0.8	0.2	56.69
16	4	0.8	0.2	54.92
17	4	0.8	0.2	51.71

An analysis of variance (ANOVA) was used to assess the relationship between the composition of starch, plasticizer, and filler with the percentage of water absorption for prepared bioplastics. One of the key components of statistics that are commonly recognized is the ANOVA [17]. Table 4 displays the ANOVA findings from the generated model. The model's F-value of 17.05. indicates that the model may be significant. It is unlikely for an F-value this large to be the result of noise, with a 0.01% likelihood. When the P-value is less than 0.0500, model terms are deemed significant [18]. The F-value of 1.34 for the lack of fit in Table 4 indicates that the lack of fit is not statistically significant in terms of pure error. When the lack of fit is not statistically significant, it indicates that the selected model fits the data well and can be safely used to forecast, infer, or draw conclusions about the relationships between variables [19].

Table 4
ANOVA results for response surface linear model and parameters

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3898.88	3	1299.33	17.05	<0.0001	significant
A- Starch	3649.57	1	3649.57	47.89	<0.0001	
B- Plasticizer	196.42	1	196.42	2.58	0.1324	
C- Filler	52.89	1	52.89	0.6940	0.4198	
Residual	990.75	13	76.21			
Lack of fit	744.15	9	82.68	1.34	0.4152	not significant
Pure Error	246.60	4	61.65			
Cor-Total	4889.62	16				

Table 5 displays the calculated R^2 of the model for the % absorption of water, which is 0.8237. In a regression model, the R-squared statistic, also known as the determined coefficient or R^2, quantifies the portion of the dependent variable's variance that can be attributable to the predictor variables [20]. This suggests that around 82.37% of the volatility in the model may be attributed to the data. There is a good level of agreement when the difference between the adjusted R^2 of 0.7830 and the Predicted R^2 of 0.7005 is less than 0.2. The number of independent variables (starch, plasticizer, and filler) in the model is considered by adjusted R-squared. It criticizes the inclusion of extraneous variables that do not increase the explanatory capacity of the model. Generally, a 0.7 or 0.8 corrected R-squared is seen as good. The predictive power of the model is shown as predicted R-squared. It is frequently applied to model validation or cross-validation. A model's ability to predict fresh data is shown by a greater predicted R-squared [21].

Table 5
Fit statistics

Std. Dev.	7.08
Mean	47.54
C.V. %	14.89
R^2	0.8237
Adjusted R^2	0.7830
Predicted R^2	0.7005
Adeq Precision	13.8739

The Design-Expert 13 software offers 100 optimization solutions. Figure 4 illustrates that the optimization resulted in a predicted percentage absorption of water of 20.069 and a desirability of 0.806. The optimization value of the percentage absorption of water can be approved if the desirability value is greater than 0.75, indicating that the variable significantly affects the reaction [18].

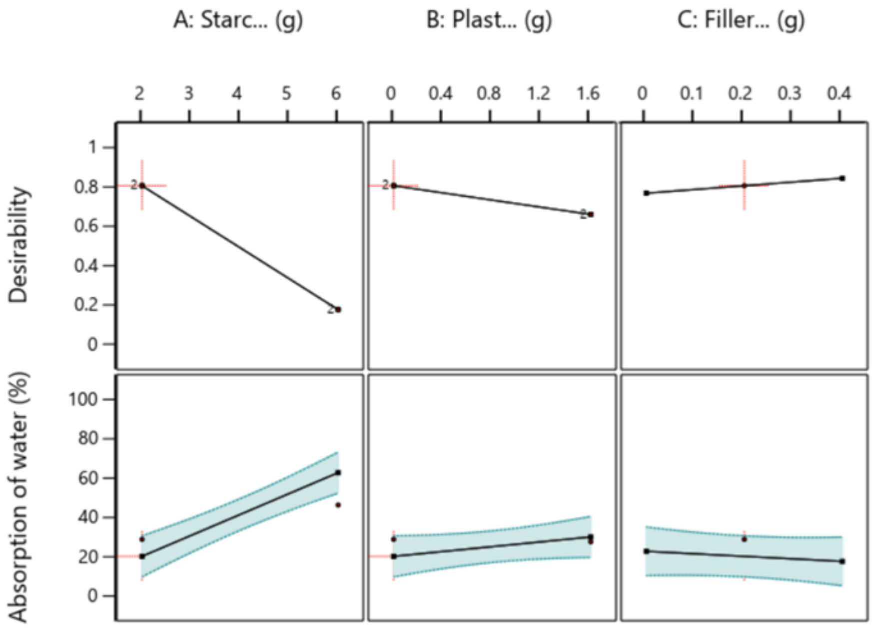

Fig. 4. Optimization percentage absorption of water using BBD approach

Eq. (2) gives the final mathematical equation in terms of the actual factors as determined by Design-Expert Software

$$\text{Percentage absorption of water} = +1.28 + (10.68 \text{ x Starch}) + (6.19\ x \text{ Plasticizer}) - (12.68 \text{ x Filler}) \quad (2)$$

The three-dimensional response surfaces (Figure 5) were shown using the linear model equations of the Box-Behnken design to illustrate the relationship between the components of starch (A), plasticizer (B), and filler (C) and the percentage of water absorption of starch-based bioplastic absorption. Plots show how the interaction effects of the factors affect the response. Figure 5 shows that when starch and plasticizer are used in small amounts, the percentage of water absorption is at its lowest, while filler is used in moderate amounts.

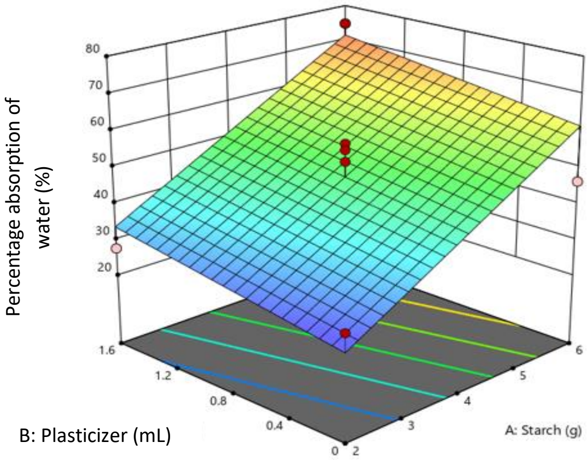

Fig. 5. 3D response surface graphs for percentage absorption of water of starch-based bioplastic

4. Conclusions

The Box-Behnken Design was applied to determine the optimum condition for achieving the lowest percentage of water absorption. The results indicate that the amount of starch, amount of plasticizer, and amount of filler significantly affect the percentage absorption of water for the prepared starch-based bioplastic from *Ipomoea batatas*. Statistical analysis based on the Box-Behnken design using Design Expert Software revealed that the ideal conditions for achieving the minimum percentage absorption of water for prepared starch-based bioplastic were 2.01 g of starch, 0.0956 mL of plasticizer, and 0.1850 g of filler.

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