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The effect of composition on mechanical properties of biodegradable plastic based on chitosan/cassava starch/PVA/crude glycerol: Optimization of the composition using Box Behnken Design

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A B S T R A C T

Synthetic plastic waste is one of the most concerning problems on our earth today. For that reason, many researchers are currently developing biodegradable plastics as a substitute for synthetic plastics for food packaging purposes. However, biodegradable plastics have poor mechanical properties because of their characterteristics as biopolymers, such as brittle and fragile. Chitosan has often been used as an additive in starch or PVA mixtures because of its biodegradability. Meanwhile, PVA itself is widely used in polymer blends because it can increase the flexibility and tensile strength of biodegradable plastic. The aim of this study is to develop a biodegradable plastic based on chitosan, cassava starch, and PVA with the addition of crude glycerol as a plasticizer. Observation of the effect of chitosan, starch, and PVA content on the mechanical properties of chitosan/cassava starch/PVA biodegradable plastics were carried out using Design-Expert Version 10.0.1 with Response Surface Methodology-Box Behnken Design. The composition of chitosan, starch, and PVA was varied from the range of 1–3 g, 1–5 g, and 1–5 g respectively. The optimization results suggest that biodegradable plastic with the composition of 3 g chitosan, 1 g starch, and 5 g PVA has the highest tensile strength and elongation values. The optimum biodegradable plastic showed that it was degraded by 50.45% of its weight after being buried in the soil for 30 days. Furthermore, the optimum biodegradable plastic can be investigated for its characteristics in further research.

1. Introduction

Currently, the transition from synthetic plastics to environmentally friendly polymer plastics derived from renewable resources is becoming a trend in the field of modern polymer materials. Synthetic plastic waste is one of the most concerning problems in Indonesia. The types of plastic circulating in the community are synthetic plastics from petroleum raw materials which are limited in number and could not be renewed. Today, most of the materials used in the packaging industry are still derived from fossil fuel sources and resistant to biological, physical, and chemical degradation [1]. So, this can cause environmental problems around us. An alternative to plastic made from petroleum or synthetic plastic is biodegradable plastic. Biodegradable plastics are plastics that can be decomposed naturally by microbial activity, because the basic ingredients are made of natural compounds taken from plants, such as starch or cellulose. Chitosan, starch, and cellulose are some of the biopolymers that are most often researched as applications for food packaging [2–4]. Starch is one of the most studied ones due to its abundant availability, low cost, as well as good biodegradability. Although it has some of the advantages already mentioned; plastics made from starch are very brittle and compared to synthetic plastics, they have inferior mechanical properties which limit their application for packaging [5]. So there need to be modifications with other materials such as; chitosan, polyvinyl alcohol, and glycerol to improve its mechanical properties.

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Chitosan is one of the preservatives from natural ingredients that has good properties to be formed into plastics and has antimicrobial properties. In the research of Ren et al (2017) adding the concentration of chitosan can significantly increase the tensile strength of the corn starch/chitosan film [6]. However, polyvinyl alcohol is also very popularly used as a mixture in starch-based biodegradable composites. Polyvinyl alcohol (PVA) is widely used as an additive in mixtures due to its excellent mechanical properties and biodegradability [7]. Polyvinyl alcohol is a synthetic biodegradable polymer used in the packaging industry due to its good film-forming properties, biodegradability, crystallinity and mechanical properties. PVA can be mixed with starch to make it more economical and increase biodegradability [8,9]. In the research of Nataliya et al (2020) the addition of PVA concentration in starch/chitosan/PVA composite films can increase the elasticity and elongation capacity of the films [10].

The addition of glycerol aims to improve physical properties, mechanical properties and protect plastics from microorganisms that can damage plastics. Glycerol can be obtained from used palm cooking oil through a transesterification process [11]. Used palm cooking oil or used cooking oil is used oil, which is commonly used in household needs, restaurant needs and others. These oils include palm oil and all other cooking oils. When viewed from the chemical composition, used cooking oil contains carcinogenic compounds that occur during the frying process. This used cooking oil waste pollutes the soil it passes through. Therefore, the utilization of used cooking oil waste into glycerol can reduce the negative impact it causes on the environment. Blick et al., (2015) reported that using crude glycerol produced from biodiesel did not effect on mechanical properties when compared to commercial glycerol when used as a plasticizer in starch-based films [12]. This showed that crude glycerol can be used as an alternative to plasticizers to reduce plastic production cost.

On the other side, one effort to optimize the composition of the plastic to achieve better performance is to use the Response Surface Methodology (RSM) [13]. Response surface methodology (RSM) is a collection of mathematical and statistical techniques. With careful design of the experiment, the aim is to optimize the response (output variable) that is affected by several independent variables (input variable). One of the response surface methodologies (RSM) is Box Behnken Design. The Box Behnken Design method is recommended for optimization processes with three variables [14]. The absence of axial/star runs makes the Box-Behnken more efficient in design because it involves fewer experimental units. Box Behnken Design is suitable for optimization using three variables because it uses a small number of samples and is able to predict the optimum value both linearly and quadratic [15].

The main aim of this study is the design and fabrication of biodegradable composite based on chitosan, cassava starch, and PVA with the addition of crude glycerol as the plasticizer. The fabrication process was designed using Design-Expert software via Response Surface Methodology Box-Behnken Design by determining tensile strength and elongation as response variables because they are important parameters for packaging applications. The selected compound for plastic fabrication and experimental design with RSM has been reported for the first time in the current study.

2. Experimental

2.1. Materials

Used cooking oil is obtained from household waste in the Tembalang area, Semarang, Indonesia. All chemical reagents were of analytical reagent grade. KOH, methanol, and acetic acid (99.8%) were purchased from Merck (Indonesia). Polyvinyl alcohol with the degree of hydroxylation >97% and chitosan (degree of deacetylation >75%) were purchased from Sigma Aldrich (Indonesia). Cassava starch (amylum manihot) was purchased from Planet Kimia (Indonesia). Distilled water was obtained from Laboratorium Terpadu, Universitas Diponegoro (Indonesia).

2.2. Production of crude glycerol from used cooking oil

First of all, oil waste from used oil for frying is prepared with a low acid content, making it possible for the transesterification process. KOH (1% by mass of oil mixture (1% by weight) was used as a catalyst. Furthermore, the mixture of oil and methanol is heated for 2 h and a temperature of 65 °C (the mole ratio of oil to moles of methanol is 1:6) for the transesterification process. Finally, the product was left in a separating funnel for 24 h, so that two layers were formed. The top layer is biodiesel and the bottom layer is crude glycerol. Furthermore, crude glycerol will be used in the plastic mixture as a plasticizer.

2.3. Preparation of chitosan/starch/PVA-crude glycerol biodegradable composites using Box Behnken design

Preparation of chitosan solution. For the preparation of the chitosan solution in this study using the method that has been used by Ren et al., 2017 [6] where a certain amount of chitosan (the composition can be seen in Table 1) is dissolved in 100 mL of 2% acetic acid. Then the mixture was heated on a hot plate at 60 °C and stirred for 60 min at a speed of 600 rpm.

Preparation of starch/PVA mixture. In the preparation of a mixture of cassava starch and PVA in this study using a combined method from previous research [10,16]. First, a certain amount of cassava starch (composition can be seen in Table 1) is dissolved in 100 mL of deionized water by heating on a hot plate at 90 °C and stirring at 600 rpm for 60 min. Then added 2 mL of crude glycerol which had been prepared previously. Furthermore, in different beaker, a number of PVA (compositions can be seen in Table 1) dispersed in 100 mL of deionized water and heated at 80 °C and stirred at 600 rpm for 2 h. For the final result of the preparation of a mixture of cassava starch / PVA, both beakers were mixed in the same beaker. It was then heated on a hot plate at 60 °C and stirred at 600 rpm for 60 min. Then the final result is a mixture of starch / PVA.

Fabrication of biodegradable plastic. A beaker containing a mixture of cassava starch / PVA will be mixed with a beaker containing a chitosan solution in the same beaker. Then stirred for 30 min at a speed of 600 rpm. Then the mixture is poured and printed in an acrylic mould measuring 20 cm × 20 cm. Then let it dry at room temperature until the plastic peels off itself from the mould (approximately 1 week). Then the final result of the plastic is analyzed to determine its mechanical properties.

2.4. Design of experiment

Box Behnken design from stastistical software Design-Expert Version 10 (Stat-Ease, Minneapolis, USA) was used to optimize

Table 1
Values and coded levels of each variable in Box Behnken design.

Variables		Coded levels	
		Low level (−1)	High level (+1)
A	Chitosan (g)	1	3
B	Starch (g)	1	5
C	PVA (g)	1	5

the compositions for the preparation of the composites. The ranges and levels of the factors investigated in this study are shown in Table 1. Three independent variables were employed by the Box Behnken design. The variables used were chitosan (A), cassava starch (B), and PVA (C). The design consists of 17 runs. The responses measured are tensile strength and elongation at break as important parameters for packaging application. One-way analysis of variance (ANOVA) was performed and *P*-value >0.05 indicated the parameters have significant impact on the response.

2.5. Mechanical test

Mechanical properties of plastic such as tensile strength and elongation at break were analyzed by using The Brookfield CT3 texture analyzer (USA) instrument. The sample was cut into rectangular pieces of 50 mm × 20 mm. The tensile speed was 5 mm/min under the load of 5 N. The stress–strain curves were drawn and the stress and strain at breaking point were reported as tensile stress (MPa) and elongation at break (%), respectively.

2.6. Soil burial test

The biodegradability test was carried out to measure the decrease in sample weight regarding other researchers [3,17] with slight modifications. The sample with size (5 cm × 5 cm) was buried in a pot containing soil with a depth of 5 cm. The soil surface is kept moist by spraying water every day. Sample weight was measured and observed after the 30th day. Eq. (1) is used to calculate the weight reduction, where Wi is the initial sample weight (g) and Wf is the final sample weight (g). Samples were taken from the soil, cleaned from residual soil using tissue to measure the weight loss of bioplastic samples.

$$weight\ \ loss\ \ (\%) = \frac{Wi - Wf}{Wi} \times 100\% \qquad (1)$$

3. Results and discussion

3.1. Experimental design

The optimization test has been successfully carried out after synthesizing the biodegradable composite by mixing each of its constituent materials. The effect of the composition of chitosan, starch, and PVA on mechanical properties was investigated by the Box Behnken design. In this study, mechanical properties were chosen as a response because it is a very important factor for designing appropriate packaging plastic. Therefore, the tensile strength and elongation of the fabricated plastics were chosen as their response and behavior to the process parameters on the composition of chitosan, starch, and PVA. Seventeen runs (Table 2) were carried out to optimize the process parameters and experiments were carried out according to the actual experimental design matrix.

The equations showing the relationship between the composition of chitosan, starch, and PVA with the relevant mechanical properties and statistical analysis are provided in Table 3. As can be seen, the suggested model is quadratic with a *P*-value >0.05 and R^2 of 0.9148 for tensile strength, meanwhile for elongation the 2FI model is recommended with a *P*-value >0.05 and R^2 of 0.8059. This indicates that the model can predict the relationship that is set correctly.

3.2. The effect of chitosan, starch, dan PVA composition on the tensile strength

A model has been presented that shows the relationship between the composition of chitosan, starch, and PVA with the tensile strength of the fabricated sample and the ANOVA results from the model obtained are presented in Table 3. parameter relationship and tensile strength with P value less than 0.01 which indicates that the quadratic equation is the right equation to correctly predict the relationship between the composition of chitosan, starch, and PVA with tensile strength. This model shows that the parameters and their interactions including *A*, *AB*, and C^2 have a significant effect on the response because they have a *P*-value of less than 0.01. Meanwhile, there are several parameters that have no significance to the response with *P*-value of more than 0.1 (data not provided here). Furthermore, the values of R^2 and Adj-R^2 (0.9148 and 0.8052, respectively) indicates that the model fits the obtained results [18].

The effect of the composition of chitosan, starch, and PVA on the tensile strength value can be seen in Fig. 1. As we can see, the effect of tensile strength is strongly influenced by the amount of chitosan composition. This is in line with the *P*-value of the chitosan parameter which has a value of 0.0002 which is very less than 0.05 which indicates the factor has a significant influence on the tensile strength response. In Fig. 1a it can be seen that chitosan with a content of 3 g has the highest tensile strength. This may be due to the reason that chitosan has a high degree of polymerization chain, which favors polymer–polymer interactions, resulting in a strong matrix [19]. The tensile strength increases with the addition of chitosan can be attributed to the high intermolecular hydrogen formation between NH_3^+ of the chitosan backbone and OH from starch [6]. Furthermore, the relationship between the interaction of PVA with chitosan and starch on tensile strength can be seen on the 3d surface graph (Fig. 1b and c). From the two graphs, it can be seen that the addition of PVA content can increase the tensile strength of the biodegradable composites. But, there is an optimum value at 3 g of PVA content, after the addition of more PVA content decreasing in tensile strength. This is due to the elastic and flexible nature of PVA. Where there is an inverse relationship between tensile strength and elongation at break of the biodegradable composite. The strength of the plastic increases as the elongation decreases [20]. This phenomenon was also found in other research [21].

3.3. The effect of chitosan, starch, dan PVA composition on elongation

A model has been presented that shows the relationship between the composition of chitosan, starch, and PVA with the elongation of the fabricated sample and the ANOVA results from the model obtained are presented in Table 3. Parameter relationship and elongation with a *P*-value less than 0.01 which indicates that the 2FI is the right model to correctly predict the relationship between the composition of chitosan, starch, and PVA on the elongation response. This model shows that the parameters and their interactions including *B*, *C*, and *AB* have a significant effect on the response because they have a *P*-value of less than 0.01. Meanwhile, there are several parameters that have no significance to the response with a *P*-value of more than 0.1 (data not provided here). Furthermore, the values of R^2 and Adj-R^2 (0.8059 and 0.6895, respectively) indicates that the model fits the obtained results.

The effect of the composition of chitosan, starch, and PVA on the elongation response is presented in Fig. 2. It can be seen that the 3d surface graph shows that the elongation response is strongly influenced by starch and PVA parameters. This is in line with the results of ANOVA analysis which showed that the parameters of starch (*B*),

Table 2
The software suggested values for chitosan, starch, and PVA content. The responses were measured and entered in the table.

Run	Factor 1 Chitosan (g)	Factor 2 Starch (g)	Factor 3 PVA (g)	Response 1 Tensile Strength (MPa)	Response 2 Elongation (%)
1	2	3	3	22.56	71.33
2	3	3	5	19.66	83.00
3	3	1	3	31.93	52.66
4	3	5	3	22.38	52.33
5	1	5	3	16.02	28.66
6	2	1	1	10.92	36.00
7	2	3	3	17.08	61.66
8	2	3	3	21.76	80.33
9	2	1	5	16.4	93.33
10	1	1	3	11.07	93.33
11	1	3	5	9.63	63.00
12	2	3	3	20.56	66.66
13	1	3	1	9.92	45.00
14	2	5	5	15.79	78.33
15	2	5	1	21.23	43.66
16	2	3	3	19.86	61.33
17	3	3	1	24.69	33.66

Table 3
The software suggested Equations between process parameters and selected responses and the relevant ANOVA results.

Response	Equation[a]	P-value	R^2
Tensile Strength (MPa)[b]	$= 20.36 + 6.50\boldsymbol{A} - 3.63\boldsymbol{B} - 4.33\boldsymbol{C}^2$	0.0053	0.9148
Elongation (%)[b]	$= 61.43 - 9.04\boldsymbol{B} + 19.92C + 16.08\boldsymbol{AB}$	0.0041	0.8059

[a] A is chitosan content, B is starch content, and C is PVA content.
[b] The modified equation was obtained after eliminating not significant terms from the original equation.

PVA (C), and the interaction of chitosan with starch (AB) had a significant effect on the elongation response, with the P-value of 0.0461, 0.0005, and 0.0169 respectively. In Fig. 1a it can be seen that the addition of starch content has a negative effect on the elongation response. This is due to the rigid and brittle nature of starch. The interaction between chitosan and starch (AB) had a significant effect on the elongation response. The addition of polysaccharides into the chitosan matrix will increase the tensile strength with a consequent decrease in elongation. This effect is caused by the incorporation of starch filler with chitosan matrix providing strong interactions between compounds, especially hydrogen bonds [22]. This was also reported by Bourtoom and Chinnan (2008) that chitosan can reduce the elongation of biodegradable composites prepared from starch-chitosan [11]. On the other hand, the addition of PVA to the biodegradable composite increased the elongation response. This is clearly seen in Fig. 1b and c, the addition of PVA content linear to increasing of the elongation. The increased flexibility of the biodegradable plastics at higher PVA concentrations could be due to the interaction of plasticizer-polymer chain that facilitates chain shear and thereby helping to increase the overall flexibility [23].

3.4. Optimization

In the previous section, we have introduced proper equations that relate the parameters of chitosan, starch, and PVA content to the selected response. Furthermore, in this section, an optimization stage has been carried out to obtain an optimum biodegradable composite composition. Several limitations were made to obtain the optimum biodegradable composite by setting the mechanical strength, both tensile strength and elongation, to the maximum value in the Design-Expert software, this is related to the parameter that is important for food packaging applications, so the mechanical strength is better to be in its maximum possible value. On another side, the compositions of chitosan, starch, and PVA are set in a certain range in the software, 1–3 g for chitosan and 1–5 g for starch and PVA respectively.

Based on the optimization process, the program Design-Expert 10.0.1 provides 36 solutions for optimization. Optimization solution with 3 g of chitosan, 1 g of starch and 5 g of PVA are recommended as solutions optimal formula because it has a value of the highest desirability is 0.822. Score desirability close to 1 can be concluded that the formula produces biodegradable composites

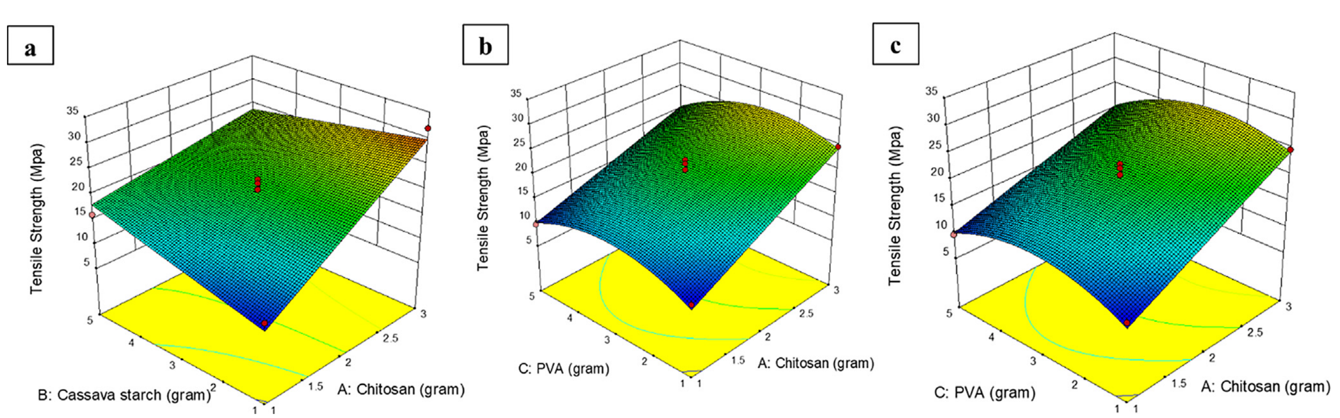

Fig. 1. The effect of (a) cassava starch and chitosan, (b) PVA and chitosan, and (c) PVA and starch interactions on the tensile strength response.

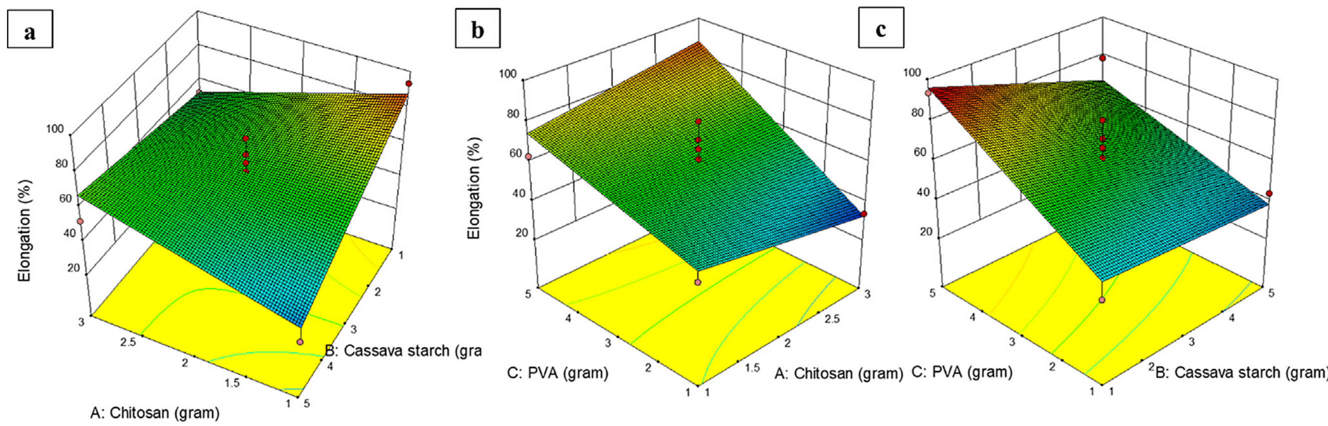

Fig. 2. The effect of (a) chitosan and cassava starch, (b) PVA and chitosan, and (c) PVA and cassava starch interactions on the elongation response.

that have the appropriate characteristics with an optimization target of 82.2%. Where if the desirability value > 0.75 then the optimization can be accepted, it means the variable has a significant influence on the response.

The results of the verification of the optimum formula recommended by the Design-Expert software with the Box Behnken Design obtained a biodegradable plastic with a tensile strength of 25.11 MPa and an elongation of 83.43 %. Fig. 3 shows a photo of the optimized plastic product. When compared to the value of predicted (Table 4), the good news is that the actual value of the experimental results is close to the value predicted by the software. This can be interpreted that the formula selected from the optimization solution recommended by Design-Experts software is good enough.

3.5. Biodegradability in soil

The optimum plastic was tested to determine its biodegradability in the soil. Fig. 4 showed the optimum plastic can be degraded by 50.45% of its weight after being buried in the soil for 30 days. After 30 days of exposure in the soil, plastic eventually diminished in size became brittle and fragile. This showed that the chitosan/cassava starch/PVA/crude glycerol optimized plastic has potential as a biodegradable plastic for packaging applications.

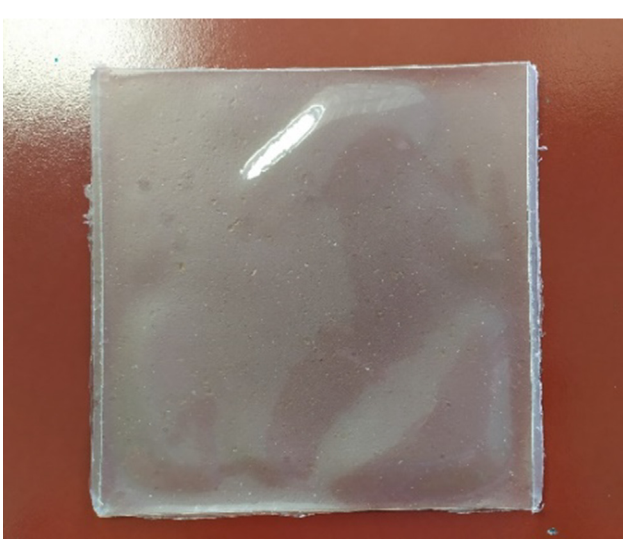

Fig. 3. Optimum fabricated biodegradable plastic.

The biodegradability of biodegradable plastics are mostly related to the absorption of water by the sample on the surface followed by the growth of microorganisms such as bacteria [24]. In addition, the high solubility of the material can also accelerate the decomposition process [25].

4. Conclusion

The chitosan/starch/PVA biodegradable plastic with crude glycerol as plasticizers have been successfully fabricated by the casting method. Fabrication process using Box Behnken Design via Design-Expert 10.0.1 software by evaluating the composition of chitosan, starch, and PVA as parameters that affecting on mechanical properties such as tensile strength and elongation. After optimization, the Design-Expert program recommended a biodegradable composite solution with a composition of 3 g of chitosan, 1 g of starch, and 5 g of PVA which is predicted to has the highest tensile strength and elongation with the highest desirability of 0.822. To verify the optimization from the software, the predicted response value is compared with the actual. The actual response has 25.11 of tensile strength and 83.43% of elongation value which is quite close to the predicted value by the software. It can be concluded that solutions recommended by the program Design-Expert is good enough.

Furthermore, the optimum sample was tested in the biodegradability test showed that could be decomposed above 50% by weight for 30 days in the soil. These results indicate that biodegradable plastic based on chitosan/cassava starch/PVA/crude glycerol can be used as an alternative to replace synthetic plastics with further research.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Table 4
Responses values predicted by the software and measured experimentally.

	Tensile strength (MPa)		% Error	Elongation (%)		% Error
Sample	Predicted	Measured	4.8%	Predicted	Measured	3,7%
Optimum sample	26.39	25.11		86.67	83.43	

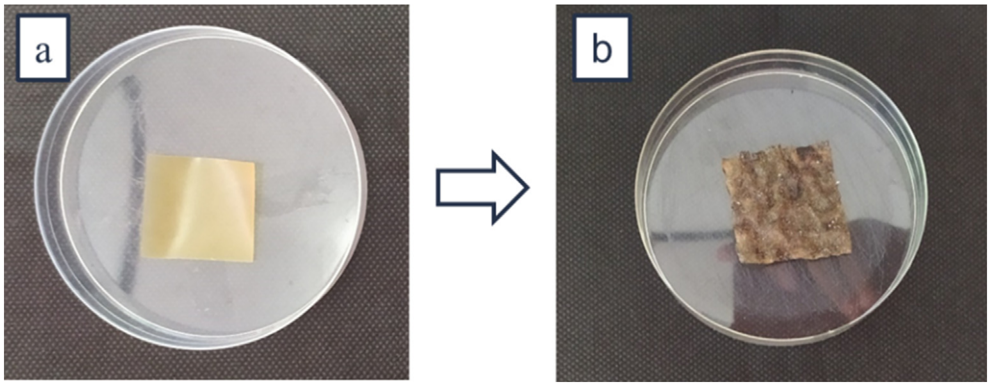

Fig. 4. The optimum plastic (a) before and (b) after buried in the soil for 30 days.

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