Optimization of the drying process of edible film-based cassava starch using response surface methodology

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Abstract. Most food packaging consists of plastic which is difficult to degrade. One strategy for addressing this issue is the development of biodegradable polymers from cassava starch, a known of raw material easily produced at low cost and biologically to degrade, hence becoming a low-cost for edible film production. The edible film was prepared by gelatinization method using cassava starch and glycerol as plasticizers. The study was subjected to determine the optimum drying process for cassava starch-based edible film based on the drying condition process with two independent variables: drying temperature (40, 50, and 60°C) and drying time (4, 5, and 6 h) on the mechanical properties. The response surface methodology approach with a central composite design was used for optimization. The experimental data for the optimum drying condition were analyzed to obtain the optimized variables using plots and contours. The optimized edible film occurred at a drying temperature of 63.28 °C and drying time of 3.58 h resulting in a tensile strength of 6640.24 Pa, elongation at break of 1.051%, and water solubility of 55.575%. The study concluded that the optimized drying condition process significantly affected the tensile strength, elongation at break, and water solubility.

1 Introduction

One of Indonesia's most promising agricultural commodities, cassava (Manihot esculenta Crantz), is expected to yield 18.5 million tons of production in 2020, exceeding rice as the top source of carbohydrates [1], [2]. Cassava starch is basic material, whether natural or modified, is becoming more used in the food business since it has several desirable inherent qualities Cassava starch contains of amylopectin and amylose, of 62.51% and 21.70% respectively [3]. Due to its high viscosity, it is also suitable for desserts, puddings, fillings, and gums. It could replace existing starches as an alternative starch because it is less expensive. For these reasons, it could be fascinating to investigate the prospect of producing new products based on cassava starch, as well as to investigate other uses and to address the lack of information about its role as an alternative source of starch, with the goal of increasing its added value.

Additionally, there have been numerous investigations into polysaccharide sources; hence, edible coatings used to coat various types of food known as edible films containing components such as cassava starch have been widely developed [4]–[6]. Because of its availability and low production costs, cassava starch-based edible films are increasingly used in various types of food packaging and more sustainable option than synthetic films or non-biodegradable films. Synthetic films and non-biodegradable films which are made from conventional plastic materials are not eatable and cause problems due to the presence of microplastics in marine environments [7].

Furthermore, cassava starch has low-cost commercial biopolymer, odourless, tasteless, and colourless qualities [5], [8]. To address this issue, various ways have been investigated, including concentration of starch, the type and concentration of plasticizer, temperature drying time and other factors. However, specific scientific papers and studies on process optimization to produce edible films from cassava starch are limited.

The production and mechanical properties of edible film can be affected by two factors: drying temperature and drying time [2], [9], [10]. Response surface methodology (RSM) is a statistical analysis technique used to optimize edible films from cassava starch, which includes determining the drying temperature and drying time on the thermal and physical characteristics of the edible films produced [10], [11]. It also serves as a tool to determine the interaction between the pretreatment and drying processes and their effects on the edible film. [1]. Several studies related to making edible films from cassava starch have been carried out. A study by Pulungan et al. optimize the fabrication of biodegradable plastic from starch and cassava peel flour by determining the optimum drying temperature and drying time and responses of tensile strength, elongation at break, and swelling. The optimum condition for producing biodegradable plastic was found of 57.79°C drying temperature and 5 h of drying duration films, resulting in a tensile strength of 2554.65 N/m^2 and elongation at break

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of 16.67% [2]. Dewi et al found that the optimum solubility of cassava starch-based edible film was 37.95% with a drying temperature of 60°C [12] whereas Wanita et al found of 37.43% the optimum solubility with a drying temperature of 40 C [13].

Hence, the present study was carried out with the objective is to optimize of drying conditions for edible film-based cassava starch as the packaging with quality materials for protective functions. This study also investigated the interaction effects of variables on selected mechanical attributes of cassava edible film-based cassava starch by using a central composite design of response surface methodology.

2 Materials and methods

2.1 Materials

The equipment used in this study was an oven, hot plate, blender, plate (20 x 25 cm), beaker glass (250 ml), measuring cylinder (10 ml), desiccator, stir bar, universal testing machine, and digital pocket scale. The ingredients used in this study were cassava from lampung regency, lab-made distilled water, and glycerol P.A Merck.

2.2 Preparation of edible film-based cassava starch

Cassava was brought from local market in Bandar Lampung. Food grade glycerol as plasticizer and distilled water were procured from local chemical supplier. In the laboratory, the cassava was sliced, then dried with the oven to remove its moisture content at 70°C. Cassava was meshed and sieved until 100 mesh to form starches. Cassava starch was employed as a raw material for preparation of edible film. 4 g of cassava starch, 100 ml of distilled water and 4 ml of glycerol were manually stirred to form an edible film. Subsequently, edible film was poured in a plat dishes (10 × 7 cm) and dried at two independent variables: i.e. drying temperature (40, 50 and 60°C) and drying time (4, 5 and 6 h) in an oven. Edible film used for testing were conditioned at 25 °C by placing in a desiccator containing silica gel for 2 h.

2.3 Edible film-based cassava starch characterization

2.3.1 Mechanical properties

The standard method plastic-tensile properties (ISO 527) were used to determine tensile strength (Pa) and elongation at break (%) of the films. The samples were cut into rectangular shapes with dimensions of 80 mm x 10 mm, placed in a *Universal Testing Machine* Exceed Electromechanical Test System Type E43 and stretched at a constant rate of 0,001 mm.s^{-1}.

2.3.2 Water solubility

Film solubility was measured by soaking the film in water at room conditions for 24 h [14]. After solubilization, samples were dried at 105ºC for 30 minutes. The samples with dimensions of 250 mm x 50 mm weighed before and after solubilization period using an analytical balance. Water solubility (%) was determined using equation below:

$$\%Water\ solubility = \frac{w_0 - w_{24}}{w_0} \ x\ 100\% \qquad (1)$$

Where w_0 denotes the first dried material (before to solubilization) and w_{24} denotes the dried material after 24 h of solubilization.

2.4 Experimental design

The drying process parameters of edible film-based cassava starch were optimized using Response Surface Methodology (RSM) and the Central Composite Design (CCD) technique. Optimization using RSM was conducted using Minitab® 19 *software*. 13 experimental runs were conducted using three independent variables: drying temperature and drying time. The statistical analysis of these data was performed at the 95% ($P < 0.05$) levels, the responses measured were tensile strength, elongation at break, and solubility of the obtained second-order models are listed in Table 2. The experimental design as listed in Table 1.

Table 1. Experimental range of independent variables used for central composite design (CCD)

Independent variables	Units	Levels		
		(-1)	(0)	(+1)
Drying temperature (X_1)	°C	50	60	70
Drying time (X_2)	h	4	5	6

3 Results and discussion

The results of the edible film-based cassava starch with drying temperature and drying time factors and its responses to mechanical properties are shown in Table 1. This present study showed that the use of drying temperature at 50°C and drying time of 6 h produced edible film with the highest tensile strength of 7760 Pa (run 3). However, at 74.1°C of drying temperature and 5 h of drying time (run 6), the resulting edible films possessed the lowest tensile strength of 4480 Pa. The percent tensile strength behaviour of edible films was reported to decrease at higher temperatures. The highest response to the elongation at break resulted in 1.44% at 50°C of drying temperature and 6 h of drying time (run 3), while the lowest elongation at break obtained 0.20% at 74.1°C of drying temperature and 5 h of drying time (run 6). The higher levels of elongation were obtained at temperatures ranging of 50 - 60 °C (run 6). The uneven thickness of the film, which changes the mechanical properties of the edible film, could be the cause of the lowest percent of elongation [2]. It was observed that tensile strength of 10-100 MPa and elongation at break of 10-50% among edible films decreased at higher temperatures and are regarded to have moderate mechanical characteristics [15]. The highest percentage water solubility was obtained from drying temperature at 50°C for 4 h, with the value of 75.88% (run 1). While, the lowest percent solubility was 36.74% resulted from drying temperature of 74.1°C and drying time of 5 h (run 6). The solubility of the edible films reduced as heating time and temperature were increased. The edible films prepared at higher temperatures was found to be lower than that of films prepared at room temperature[9].

Table 2. Central composite design matrix with two actual factors and responses to tensile strength, elongation and solubility

	Code variable		Actual factors		Responses		
Run	X_1	X_2	Drying temperature (°C)	Drying time (h)	Tensile strength (Pa)	Elongation (%)	Solubility (%)
1	-1	-1	50	4	4880	0.44	75.88
2	1	-1	70	4	5440	0.88	51.82
3	-1	1	50	6	7760	1.44	52.04
4	1	1	70	6	4720	0.36	37.86
5	-1.41421	0	45.8	5	5600	1.04	47.34
6	1.41421	0	74.1	5	4480	0.2	36.74
7	0	-1.41421	60	3.6	7360	1.20	47.75
8	0	1.41421	60	6.4	4560	0.32	52.43
9	0	0	60	5	4960	0.44	61.81
10	0	0	60	5	5040	0.56	63.25
11	0	0	60	5	4960	0.48	61.63
12	0	0	60	5	4880	0.48	62.20
13	0	0	60	5	4880	0.48	62.11

3.1 Mechanical properties of edible film-based cassava starch

3.1.1 Tensile strength of edible film-based cassava starch

The tensile strength of edible film-based cassava starch prepared in this study ranged from 4880 to 7760 Pa as shown in Table 2. The decrease in tensile strength at higher drying temperature and time might be due to moisture evaporation or a loss in inter/intra molecular contacts between the polymers, which could reduce the homogeneous compact structure of the film matrix [16].

Table 3. The analysis of variance (ANOVA) results for edible film-based cassava starch's tensile strength

Source	**DF**	**Adj SS**	**Adj MS**	***F-Value***	***P-Value***
Model	5	7879850	1575970	2.26	0.042
Linear	2	2469339	1234669	1.77	0.239
X_1	1	2064430	2064430	2.96	0.129
X_2	1	404909	404909	0.58	0.471
Square	2	2170511	1085255	1.56	0.276
X_1* X_1	1	66810	66810	0.10	0.766
X_2* X_2	1	2166010	2166010	3.11	0.121
2-Way Interaction	1	3240000	3240000	4.65	0.038
X_1* X_2	1	3240000	3240000	4.65	0.038
Error	7	4879781	697112		
Lack-of-Fit	3	4861861	1620620	361.75	0.078
Pure Error	4	17920	4480		
Total	12	12759631			

R^2=61.76%

Analysis of variance (ANOVA) was conducted on the results of tensile strength to determine the significance of the model. Based on table 3, the P-value of the model is 0.042 and the P-value of 2-way interaction is 0.038, the value is smaller than the significance value (≤5%), so it can be interpreted that these variables affect the tensile strength. A nonsignificant (≥ 5%) means that the model fits well. From the regression analysis R^2 value of 61.76% was obtained, which indicated directly means of sample variation was related to the independent variable, hence the model is suitable.

The adequacy of the model was determined using regression coefficient (R_2) analysis. A second-order polynomial model fitted to the CCD and the resulting quadratic model yield the following equation, which can also be used to calculate the predicted tensile strength (Pa) at the optimum condition of edible film:

$$\textit{Tensile strength (Pa)} = -405 + 282\,X_1 - 405\,X_2 + 0.98\,X_1^2 + 558\,X_2^2 - 90\,X_1X_2 \quad (2)$$

From the equation, tensile strength represents the obtained response factor. X_1 and X_2 are linear factors, X_1^2 and X_2^2 are quadratic factors and X_1X_2 is the interaction between the independent variables. The experimental design and analysis of variance (ANOVA) method to determine the effects of significant interactions in the model ($p < 0.01$) were performed using the statistical software Minitab® 19.

The effects of drying temperature and drying time on the tensile strength of the cassava starch based edible film can be seen from the contour and surface plot in figure 1. The contour plot's employing colours ranging from red to blue suggests that the tensile strength value was increasing. Highest tensile strength was obtained from drying temperature at 50°C and drying time of 6 h produced edible film with the value of 7760 Pa. The highest tensile result was in agreement with previous research by Epriyanti et al., which found tensile strength of 1.04 MPa at condition temperature of 50 °C and drying time of 6 h, thus the tensile strength was increase in the tensile strength of the edible film produced [17]. Similar to this, a study by A. Al-Harrasi et al. suggested that the evaporation of moisture content or a reduction in the inter/intra molecular connections between the polymers could be the cause of the decrease in tensile strength values at higher drying temperatures [9]. It can be concluded that the increasing drying temperature and decreasing drying time, the smaller the tensile strength value obtained, and vice versa.

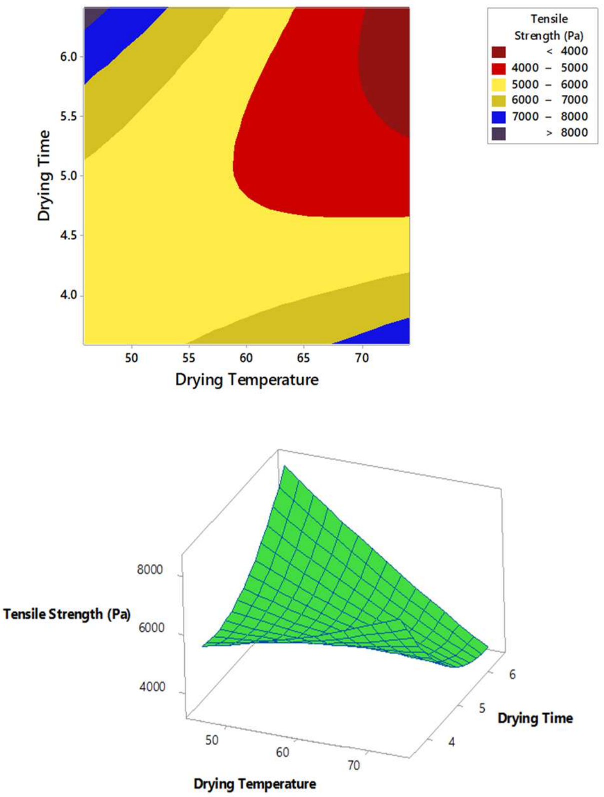

Fig. 1. Response surface and contour plot for tensile strength based edible film from cassava starch.

The influence of glycerol as a plasticizer in edible film-forming contributed to tensile strength's mechanical properties. The findings by Tessaro et al. found that the presence of glycerol in the film forming from cassava starch resulted in greater film tensile strength. Due to the significant interaction of intermolecular hydrogen bonds, glycerol has the ability to build up more stiff in edible films [16] As a result, the current study found that glycerol had a good effect on the tensile strength of cassava starch based edible film, a combination characteristic of slightly stretchy and resistant materials. Whereas films dried at lower temperatures and longer drying times showed better morphology (fewer pores/cracks), which could result in increased tensile strength [9].

3.1.2 Elongation at break of edible film-based cassava starch

According to table 2, the elongation at break of edible film-based cassava starch prepared in this present study ranged from 0.2 to 1.44%. The elongation at break demonstrates how long the edible film be stretched from the start to the break. [3]. Othman and Nasir e al. reported the elongation at break of edible films generally decreased with drying time, possibly due to recrystallization and loss of moisture and plasticizer from the film matrix [19] On the other side, the elongation at break increased as the glycerol content increased. This is because the polymer chain's greater mobility caused the material's elasticity to increase [19].

Table 4. The analysis of variance (ANOVA) results for edible film-based cassava starch's elongation at break

Source	**DF**	**Adj SS**	**Adj MS**	***F-Value***	***P-Value***
Model	5	1.288	0.257	4.14	0.045
Linear	2	0.490	0.245	3.95	0.071
X_1	1	0.417	0.417	6.72	0.036
X_2	1	0.073	0.073	1.18	0.314
Square	2	0.219	0.109	1.77	0.239
$X_1 * X_1$	1	0.059	0.059	0.96	0.360
$X_2 * X_2$	1	0.183	0.183	2.96	0.129
2-*Way Interaction*	1	0.577	0.577	9.29	0.019
$X_1 * X_2$	1	0.577	0.577	9.29	0.019
Error	7	0.435	0.062		
Lack-of-Fit	3	0.425	0.141	3.09	0.091
Pure Error	4	0.009	0.002		
Total	12	1.723			

R^2=74.75%

Analysis of variance (ANOVA) was conducted on the results of the percent elongation at break to determine the significance of the model. According to table 4, the P-value of the model is 0.045 and the P-value of 2-way interaction is 0.019, the value is smaller than the significance value (≤5%), so it can be interpreted that these variables affect the elongation. A nonsignificant (≥ 5%) means that the model fits well. According to table 4, the P-value of lack of fit is 0.091 (≥ 5%), so it can be interpreted that the model made can represent the elongation at break's data on edible film. From the regression analysis R^2 value of 74.75% was obtained, which indicated directly means of sample variation was related with the independent variable, hence the model is suitable.

The adequacy of the model was determined using regression coefficient ($R2$) analysis. A second-order polynomial model fitted to the CCD and the resulting quadratic model yield the following equation, which can also be used to calculate the predicted percent of elongation at break at the optimum condition of edible film:

$$\textit{Elongation at break}\ (\%) = -1.68 + 0.056\, X_1 + 0.56\, X_2 + 0.000925\, X_1^2 + 0.1625\, X_2^2 - 0.0380\, X_1 X_2 \quad (3)$$

From the equation, percent elongation at break represents the measured response factor. X_1 and X_2 are linear factors, X_1^2 and X_2^2 are quadratic factors and X_1X_2 is the interaction between the independent variables. The experimental design and analysis of variance (ANOVA) method to determine the effects of significant interactions in the model ($p < 0.01$) were performed using the statistical software Minitab® 19.

The effects of drying temperature and drying time on the elongation at break (%) of the cassava starch based edible film can be seen from the contour and surface plot in figure 2. Red to blue colors on the contour plot indicate that the value of elongation was rising. The highest response to the elongation at break resulted in 1.44% at 50°C of drying temperature and 6 h of drying time, while the lowest elongation obtained 0.20% at 74.1°C of drying temperature and 5 h of drying time. Oliveira et al. synthesized films of cassava starch and glycerol at different temperature and time conditions (25°C/60h, 40°C/5h, 60°C/3h, and 80°C/2h), the effect of drying temperature on the properties of the film was evaluated. The results showed that temperature and drying time modifications had major effects on the barrier properties and roughness, which improved elongation at break. [20]. Luchese et al. evaluated the influence of starch content on the physicochemical properties of cassava starch-based films, at higher biopolymer content could promoted an increase in elongation at break of the starch-based films [18]. Since higher temperatures and longer drying times employed, the stiffness of the edible film will increase, making the material drier and more easily broken [15]. It can be concluded that the decreasing drying temperature and increasing drying time, the higher the elongation at break value obtained, and vice versa.

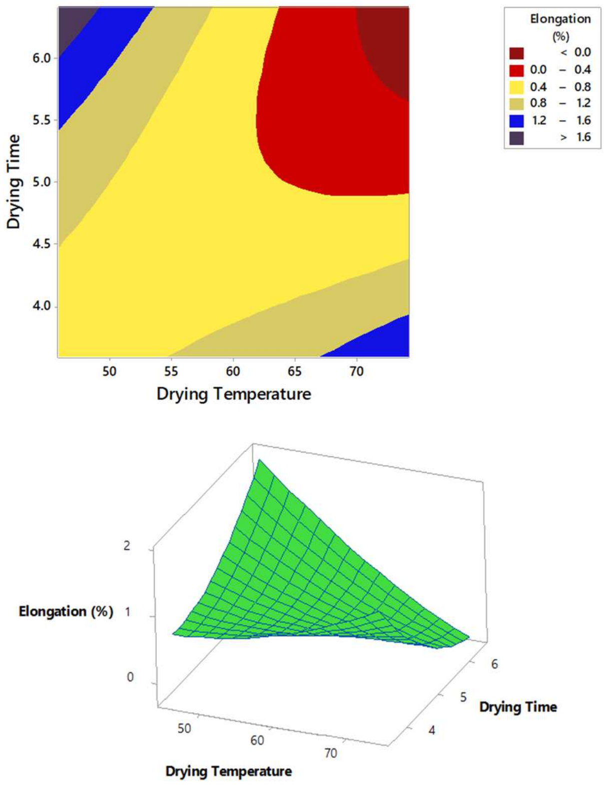

Fig. 2. Response surface and contour plot for elongation at break based edible film from cassava starch.

This present study found elongation at break of the edible film increased when drying temperature was raised while drying time was lowered. At high drying temperatures, water evaporation occurred, which stiffened and homogenized the structure of biodegradable plastic particles [21]. However, the percent elongation was discovered to be limited by a high drying temperature or drying time, probably due to glycerol evaporation [11].

3.1.3 Solubility in water test of edible film-based cassava starch

The solubility in water of edible film-based cassava starch prepared in this present study ranged from 36.74% to 75.88% as shown in Table 2. The percentage of solubility in water increased after glycerol was added, indicating that both are very hydrophilic. Similar to this, previous studies have also found that a high starch concentration improved water absorption [4], [20]. In addition, glycerol is a hydrophilic low-molecular-weight carbohydrate with the ability to absorb water.

Table 5. The analysis of variance (ANOVA) results for edible film-based cassava starch's water solubility

Source	**DF**	**Adj SS**	**Adj MS**	***F-Value***	***P-Value***
Model	5	1005.51	201.103	3.10	0.046
Linear	2	475.67	237.83	3.66	0.082
X_1	1	354.04	354.04	5.45	0.052
X_2	1	121.63	121.62	1.87	0.213
Square	2	505.44	252.71	3.89	0.073
X_1* X_1	1	444.76	444.75	6.85	0.035
X_2* X_2	1	109.71	109.70	1.69	0.235
2-*Way Interaction*	1	24.40	24.40	0.38	0.049
X_1* X_2	1	24.40	24.40	0.38	0.049
Error	7	454.49	64.92		
Lack-of-Fit	3	452.92	150.97	3.42	0.096
Pure Error	4	1.58	0.39		
Total	12	1460.00			

R^2=75.88%

Analysis of variance (ANOVA) was conducted on the results of the percent solubility of water to determine the significance of the model. According to table 4, the P-value of the model is 0.046 and the P-value of 2-way interaction is 0.049, the value is smaller than the significance value (≤5%), so it can be interpreted that these variables affect the elongation. A nonsignificant (≥ 5%) means that the model fits well. According to table 5, the P-value of lack of fit is 0.096 (≥ 5%), so it can be interpreted that the model made can represent the percent of solubility on edible film. From the regression analysis R^2 value of 75.88% was obtained, which indicated directly means of sample variation was related with the independent variable, hence the model is suitable.

The adequacy of the model was determined using regression coefficient ($R2$) analysis. A second-order polynomial model fitted to the CCD and the resulting quadratic model yield the following equation, which can also be used to calculate the predicted percent of solubility at the optimum condition of edible film:

$$\text{Solubility (\%)} = -191 + 7.69 X_1 + 21 X_2 - 0.08 X_1^2 - 3.97 X_2^2 + 0.247 X_1 X_2 \quad (4)$$

From the equation, percent solubility represents the measured response factor. X_1 and X_2 are linear factors, X_1^2 and X_2^2 are quadratic factors and X_1X_2 is the interaction between the independent variables. The experimental design and analysis of variance (ANOVA) method to determine the effects of significant interactions in the model ($p < 0.01$) were performed using the statistical software Minitab® 19.

The effects of drying temperature and drying time on the solubility (%) of the cassava starch based edible film can be seen from the contour and surface plot in figure 3. The contour plot showed colours from red to purple indicates that the value of solubility was increasing. The result of 75.88% was the highest percentage of water solubility, which was obtained after 4 h of drying at 50°C. The lowest solubility percentage, 36.74%, was produced by drying at 74.1°C for 5 h. As heating time and temperature were increased, the solubility of the edible films decreased. It was discovered that the of edible films prepared at higher temperatures was inferior to that of films prepared at room temperature [9].

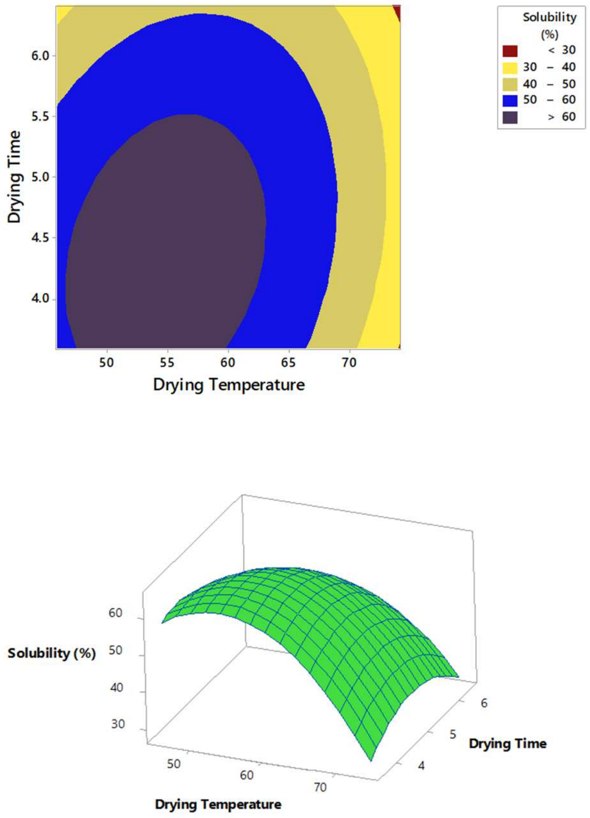

Fig. 3. Response surface and contour plot for solubility in water based edible film from cassava starch.

The percent solubility found in the present study resulted in high solubility, which means that the film is easy to dissolve in water and its ability to retain water is low [23]. Solubility is a physical property of edible film that shows the percentage of dry weight dissolved after being immersed in water for 24 h [9], [14]. Higher temperatures and longer drying times caused the water to dry and evaporate more quickly, which caused uneven surfaces, air cavities to form, and water absorption via the pores in the films structure [18], [22].

3.2 Optimization of edible film-based cassava starch

Response to a desirability (D) function that accepts values between 0 and 1 is transformed by the desirability function analysis. Desirability will be 1 if the response variable reaches its objective and zero if it falls outside of the allowable range [24]. The outcome of the edible film analysis using statistical software to obtain the optimum used to produce the desired response. The optimum edible film was produced under the operating conditions of drying temperature 63.28°C and drying time 3.58 h with (D) of 0.6015. The response resulting from the optimum value for tensile strength is 6640.24 Pa with (D) of 0.658, elongation is 1.051% with a (D) 0.686, and solubility is 55.575% with (D) of 0.481.

Desirability values that are closer to 1.0 indicate the ability of the software to produce the desired product is more perfect, but optimization is not to obtain a desirability value of 1.0, but to find the best condition that brings together all objective functions [40]. The number of external variables that affect the response such as the use of drying temperature and drying time results in the desirability value not approaching 1.0.

4 Conclusions

The lower drying temperature with longer drying time results in higher tensile strength and elongation at break value. However, the higher drying temperature and drying time the smaller solubility value. The optimum edible film from cassava starch was produced under the operating conditions of drying temperature 63.28°C and drying time 3.58 h. The response resulting from the optimum value for tensile strength of 6640.24 Pa, elongation at break of 1.051%, and solubility of 55.575%.

Based on the results of the study, future research could focus on optimizing the drying temperature and time conditions to further improve the properties of biopolymeric films made from cassava starch and glycerol. Additionally, further studies could investigate the potential of these films in other applications, such as in the food packaging industry, agricultural, pharmaceutical, and medical applications.

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