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Optimizing Edible Film from Corn Cobs with Surface Response Method

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Abstrak. The increase in plastic production worldwide has created quite a serious environmental problem. Edible film is an alternative packaging that can decompose naturally, one of the materials that can be used to make edible films is starch. This study aims to determine the composition of corn cob starch and plasticizers that can produce edible films with the best properties. The starch used is derived from corn cobs and the plasticizers used are glycerol and sorbitol. The edible film in this study was made by the casting method by dispersing the raw materials, heating the mixture, printing the edible film and drying the edible film. This research was conducted with variations in the corncob of 5, 6 and 7 in grams and the variation of the ratio of glycerol to sorbitol plasticizer is 2:8; 3:7; 5:5; 7:3; 8:2 (ml). The more starch content increases the thickness of the edible film and tensile strength, but the elongation and water vapor permeability decreases, the best edible film is obtained at the glycerol-sorbitol composition ratio of 5:5 with the amount of corncob starch of 7 grams.

Keyword: corn cobs, edible film, optimization, surface response method

1 Introduction

The increasing production of plastic around the world is causing serious problems for the environment. This is because plastic is difficult to decompose in nature, Edible film is an alternative packaging that can decompose naturally. Edible film is defined as a thin polymer layer that serves as a barrier to gas and moisture that can be consumed. The components that are often used to make edible films are divided into three categories, namely hydrocolloids, lipids, and composites. A non-volatile plasticizer was added to the hydrocolloid film formation as a solution to renew the flexibility of the edible film. Plasticizers that are often used are: glycerol, sorbitol, polyethylene glycol and oligosaccharides. Glycerol is known as a hydrophilic plasticizer, so it is suitable to be added to hydrophobic film-forming materials such as starch, pectin, gel, and protein. Glycerol acts as a plasticizer to increase the flexibility of the film [1]. Starch, a polymer that is often used as a raw material in the manufacture of edible films, is often used in the food industry as a biodegradable film that aims to replace plastic polymers because it is economical, renewable, and provides good physical characteristics [2]. The use of a single material in the manufacture of edible films still has several shortcomings, including brittle and rigid properties, it is necessary to add additional materials, namely plasticizers [3]. Plasticizer is known as an additive in the manufacture of edible films which serves to increase the elastic properties [4].

Edible film has optimum conditions, namely when the composition of the mixture can produce physical and mechanical test values according to the standard, the more starch concentration used, the better the edible film properties obtained. Corn starch with the best composition obtained was 3% corn starch concentration and 7% black turmeric juice, with the characteristics of the edible film being water vapor transmission 0.50 gram/m^2.hour, thickness 0.17 mm, tensile strength 7.90 N/cm^2, and elongation. concentration of plasticizer, the type of plasticizer used also greatly affects the characteristics of the edible film. The raw material of kolang kaling with plasticizer is glycerol, sorbitol, and polyethylene glycol [5]. The best treatment obtained is the use of sorbitol with a concentration of 3% with the resulting parameter values are 0.12 mm thick, water vapor transmission rate 4.34 gram/m^2 hour, tensile strength 2.83 N/cm^2 and percent elongation 44.65%. edible films such as tensile strength, percent increase in length of edible film and water vapor permeability [6]. Starch is a material that is often used by the food industry as a biodegradable film that functions to replace plastic polymers because it is economical, renewable, and provides good physical characteristics [6].

Corn cobs are the largest part of corn waste. The content contained in corn cobs is cellulose as much as 40-60%, hemicellulose as much as 20-30% and lignin as much as 25-30%. The starch content in corn cobs is 27.1% [7]. The nature of starch is suitable for edible films because it can form a fairly strong film. Starch-based edible films have weaknesses, namely low water resistance and low moisture barrier because the hydrophilic nature of starch can affect its stability and mechanical properties [8].

Sorbitol which acts as a plasticizer in the formation of edible films can reduce the permeability of the film to oxygen, reduce the brittleness of the film so that the elasticity of the film increases. Sorbitol is also commonly used as an additive in edible films as an artificial sweetener [9]. Hydrothermal modification of physical

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properties in making edible film from red bean starch [10], use of sweetener food additives [11].

1.1 Characteristic Standard Edible Film

Based on the Japanese Industrial Standard (JIS), edible films have a maximum standard thickness of 0.25 mm, a minimum tensile strength of 0.392 MPa, a maximum water vapor transmission rate of 10 g/m2 day, and elongation has a minimum standard of 10% [12]. Factors that need to be considered in the manufacture of edible films are temperature, plasticizer and material concentration. Heating is carried out during mixing. This is done with the aim of achieving perfect starch gelatinization, in the manufacture of edible films a temperature of ±70°C is used [1]. Plasticizing agents are needed as additional ingredients in the manufacture of edible films with the aim of overcoming the brittleness of edible films caused by intensive intermolecular forces. Plasticizers increase the mobility of the polymer chains, thereby increasing the flexibility of edible films [13]. The concentration of raw materials is very influential, especially on the physical properties of edible films. The more concentration of raw materials added, the thicker and greater the tensile strength [14]. In the manufacture of this edible film, starch from corn cobs is used as raw material. Starch from corn cobs is very safe for human consumption because it does not endanger health. The plasticizers used are glycerol and sorbitol. According to the regulation of the POM RI No. 5 of 2013 concerning the maximum limit for the use of humectant food additives and No. 4 of 2014 concerning the maximum limit for the use of sweetener food additives, the limit on the use of glycerol and sorbitol for edible packaging, which is in accordance with CPPB. The maximum CPPB limit is the amount of BTP that is allowed to be present in food in sufficient quantities needed to produce the desired effect and its use should not be excessive [15].

1.2 Result Optimization

Response Surface Methodology (RSM) is a collection of mathematical and statistical methods used in modeling and analysis, which aims to see the effect of several quantitative variables on a response variable and to optimize the response variable. The relationship between response Y and the independent variable X:

$$Y = f(X_1, X_2, ..X_k) + \varepsilon \qquad (1)$$

Where:
Y = response variable
X = independent variable/factor
ε = error

The first step of RSM is to find the relationship between the response and the independent variable through the appropriate approach, between the response and the independent variable is a linear function, the function approach is called the first-order model, as shown in the following equation.

$$Y = \beta o + \sum_{i=1}^{k} \beta o \, Xi \qquad (2)$$

If the form of the relationship is a quadratic, then for the function approach, a higher degree polynomial is used, namely the second-order model

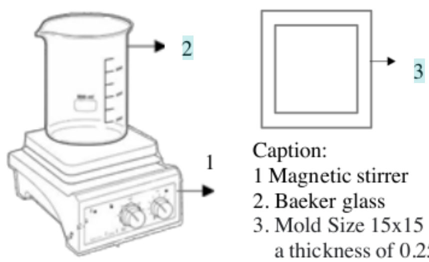
$$Y = \beta_o + \sum_{i=1}^{k} \beta_o X_i + \sum_{i=1}^{k} \beta_u X_i^2 + \sum_{i=1,j=2}^{k-1,k} \beta_{i,j} X_i X_j + \varepsilon \qquad (3)$$

After obtaining the most suitable form of relationship, the next step is to optimize the relationship. Equations and optimization results are obtained using Minitab software. To check the significance of the model, we can see the p-value of Regression. If the p-value is smaller than the degree of significance (α = 5%), it can be said that these variables make a significant contribution to the model [16].

From previous research on edible films, in this study using starch from corncob waste weighing 5,6 and 7 as raw materials, using glycerol and sorbitol as plasticizers. Edible film is made by casting method by dispersing the raw material, heating the mixture, printing the edible film and drying the edible film.

2 Methodology

The materials used in this study were corn cobs, aquadest, carrageenan, 1% NaOH, glycerol and sorbitol.



Caption:
1 Magnetic stirrer
2. Baeker glass
3. Mold Size 15x15 cm with a thickness of 0.25 mm

Fig. 1. Research tools edible film from corn cobs

2.1 Starch making

Making starch from corn cobs is done by cutting the corn cobs into small pieces, soaked in 1% NaOH for 12 hours to remove the lignin content. The corncobs were washed with water and ground, the pulp was squeezed out with a filter cloth the filtrate was allowed to stand for one day to produce starch deposits. The starch precipitate was dried in the oven to remove the moisture content, after drying the starch was ground with a mortar until smooth, then sifted so that the size was homogeneous.

2.2 Edible film making

Starch from corn cobs was weighed with a weight of 5gr, 6gr, and 7 grams. Starch from corn cobs was added with a mixture of glycerol and sorbitol in a ratio of 2:8, 3:7, 5:5, 7:3, 8:2 and then added with distilled water until the solution reached 100 ml in a beaker glass. The mixture was stirred with a magnetic stirrer with a rotation of 400 rpm, heated to a temperature of ± 70°C and stirred for 20 minutes. The edible film solution is still stirred while it is cooled to room temperature in order to prevent air bubbles from forming during printing. The edible film solution was printed on a glass plate and then dried at 60 °C for 7 hours. After drying, the film was cooled to room temperature.

3 Result and Discussion

After obtaining the results of the analysis and calculation of the mechanical and physical properties edible films, optimization of the results was carried out to determine the optimum results of edible films with the Surface Response Method using the Minitab software application which was carried out according to the theoretical basis reference regarding Result Optimization. The optimization results from Minitab software show that the overall data is second order. The optimization results will show the function of the response equation to the modified conditions such as the glycerol-sorbitol ratio and starch weight.

Table 1. Value of tensile strength, elongation, thickness and permeability

Starch weight (grams)	Ratio Glycerol to Sorbitol 2:8	3:7	5:5	7:3	8:2
Tensile strength (MPa)					
5	0.066	0.128	0.104	0.1	0.09
6	0.317	0.283	0.24	0.166	0.14
7	0.536	0.528	0.491	0.318	0.213
Elongation (%)					
5	10.2	14.6	15.1	19.7	21.4
6	5.1	5.8	6.9	7.9	7.5
7	4.1	4.6	6.1	6.5	6.5
Thickness (mm)					
5	0.12	0.15	0.16	0.16	0.15
6	0.18	0.19	0.19	0.19	0.17
7	0.26	0.23	0.2	0.19	0.16
Permeability (gram/m² .day)					
5	11.83	11.66	11.66	11.39	11.55
6	11.33	11.21	10.66	10.67	10.47
7	10.39	9.99	9.84	10.23	8.86

$$\text{Tensile strength} = -0{,}059 - 0{,}107\,X_1 + 0{,}198\,X_2 + 0{,}028\,X_1^2 + 0{,}0059\,X_2^2 - 0{,}04486\,X_1X_2$$

$$\text{Elongation} = 178{,}2 - 54{,}58\,X_1 + 9{,}62\,X_2 + 4{,}240\,X_1^2 - 0{,}542\,X_2^2 - 1{,}013\,X_1X_2$$

$$\text{Thickness} = -0{,}332 + 0{,}123\,X_1 + 0{,}08\,X_2 - 0{,}006\,X_1^2 - 0{,}0018\,X_2^2 - 0{,}01327\,X_1X_2$$

$$\text{Permeability} = 10{,}72 + 0{,}84\,X_1 + 0{,}499\,X_2 - 0{,}128\,X_1^2 + 0{,}0017\,X_2^2 - 0{,}1164\,X_1X_2$$

Note:
X_1 = weight of starch
X_2 = Ratio Glycerol to Sorbitol

Figure 2 shows the best tensile strength results have the darkest green color contour at a ratio of glycerol to sorbitol 2:8 and starch weight of 7 grams, this is due to the low ability of sorbitol to bind water thus limiting its ability to reduce hydrogen bonding of polymer chains compared to glycerol so that the strength The tensile strength of the film with sorbitol plasticizer is better than using glycerol plasticizer. The lowest results were shown in the ratio of glycerol to sorbitol 2:8 and starch weight of 5 grams, the small value of tensile strength was influenced by the small thickness of the edible film.

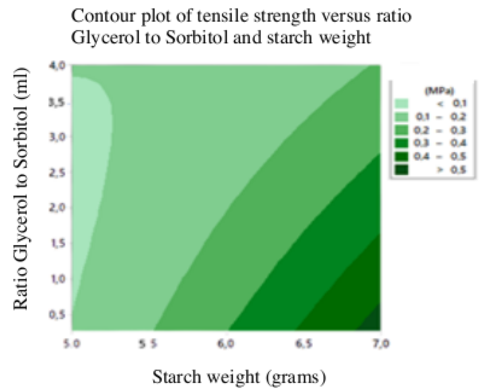


Fig. 2. Contour Plot Responses between Glycerol-Sorbitol Ratio and Starch Weight to Tensile Strength

Based on Figure 3, the best elongation results have a dark green color contour at a glycerol to sorbitol ratio of 8:2 and a starch weight of 5 grams. The lowest results were shown in the ratio of glycerol to sorbitol 2:8 and starch weight of 7 grams. This is because the increase in glycerol will decrease the intermolecular forces, as a result, the mobility between the molecular chains increases. The increase in glycerol will reduce the cohesive bonds between polymers which form a more elastic film.

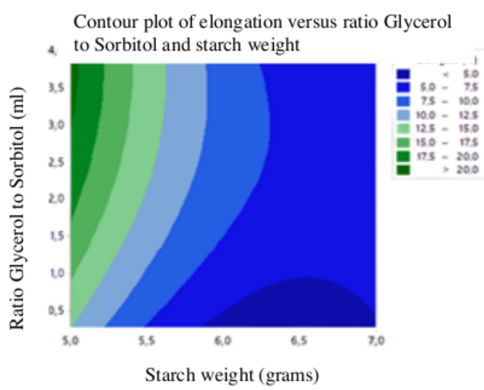


Fig. 3. Contour Plot Responses between Glycerol-Sorbitol Ratio and Starch Weight to Elongation

Based on Figure 4, the best water solubility results have a dark green color contour at a ratio of glycerol to sorbitol 2:8 and a starch weight of 7 grams. The lowest results were shown in the ratio of glycerol to sorbitol 2:8 and starch weight of 5 grams. The increase in the concentration of the material in the suspension of the edible film causes the total amount of solids contained in the edible film to increase, so that after the suspension of the edible film is dried, the edible film obtained is thicker.

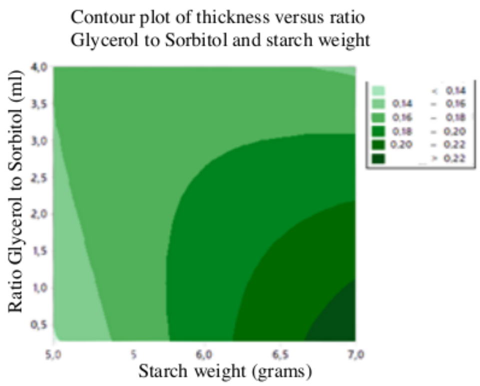


Fig. 4. Contour Plot Responses between Glycerol-Sorbitol Ratio and Starch Weight to Thickness

Based on Figure 5 the results of the best water solubility have the darkest color contour at the ratio of glycerol to sorbitol 2:8 and starch weight of 5 grams. The lowest results were shown in the ratio of glycerol to sorbitol 8:2 and starch weight of 7 grams. The effect of starch weight on the water vapor permeability of edible films can be seen that the more starch, the smaller the value of the water vapor permeability of the edible film. The high concentration of corncob starch will increase the amount of film-forming polymer. Increasing the amount of polymer will reduce the voids in the gel formed on the film. The thicker and denser the film matrix formed can reduce the rate of permeability because it is difficult for water vapor to penetrate.

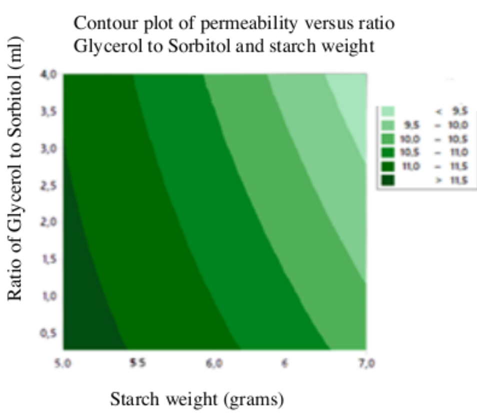


Fig. 5. Contour Plot Responses between Glycerol-Sorbitol Ratio and Starch Weight to Permeability

Based on Figure 6 shows the surface plot between the ratio of glycerol to sorbitol and starch to tensile strength. The best condition in Figure 6 is shown at the top point of the graph which shows the point of the glycerol to sorbitol ratio of 2:8 with a starch weight of 7 grams which produces a tensile strength of 0.536 MPa. While on the lowest surface showed the lowest results at the ratio of glycerol: sorbitol 2:8 with a starch weight of 5grams.

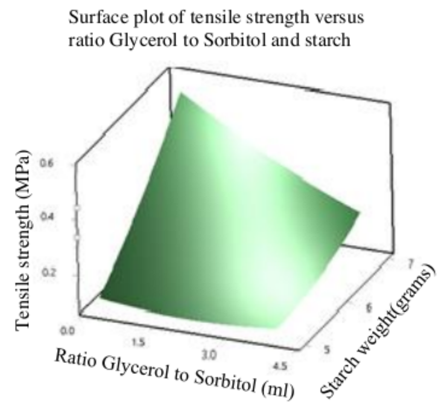


Fig. 6. Surface Characteristics Responses between Glycerol-Sorbitol Ratio and Starch Weight to Tensile Strength

Based on Figure 7 shows the surface plot between the ratio of glycerol to sorbitol and starch weight to elongation. The best condition in Figure 7 is shown at the top point of the graph which shows the point of the glycerol to sorbitol ratio of 8:2 with a starch weight of 5 grams which produces an elongation of 21.4 %. Meanwhile, on the lowest surface, the lowest yield was at the ratio of glycerol to sorbitol 2:8 with a starch weight of 7 grams.

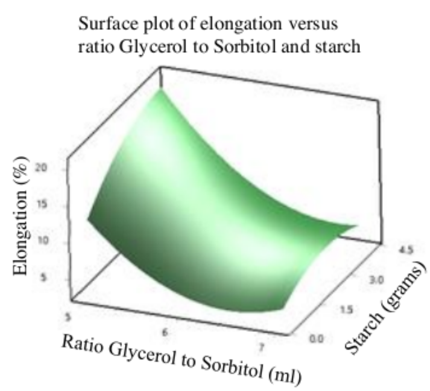


Fig. 7. Surface Characteristics Responses between Glycerol-Sorbitol Ratio and Starch Weight to Elongation

Based on Figure 8 shows the surface plot between the ratio of glycerol to sorbitol and starch weight to thickness. The best condition in Figure 8 is shown at the top point of the graph which shows the point of the ratio of glycerol to sorbitol 2:8 with a starch weight of 7 grams which produces a thickness of 0.26mm. While the lowest surface showed the lowest yield and the ratio of glycerol to sorbitol was 8:2 with a starch weight of 5 grams.

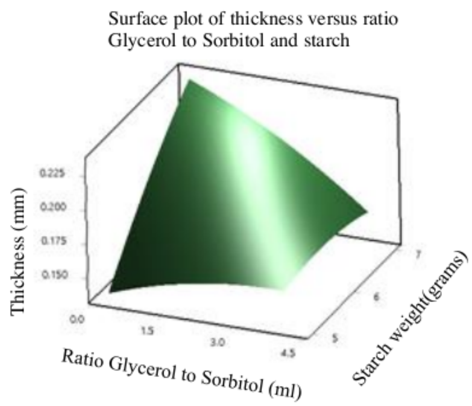


Fig. 8. Surface Characteristics Responses between Glycerol-Sorbitol Ratio and Starch Weight to Thickness

Based on Figure 9 shows the surface plot between the ratio of glycerol to sorbitol and starch weight to water vapor permeability. The best condition in Figure 9 is shown at the top point of the graph, which shows the ratio of glycerol to sorbitol 2:8 with a starch weight of 5 grams which results in a permeability of 11.83g/m2day. While on the lowest surface, the lowest yield was at the ratio of glycerol to sorbitol 8:2 with a starch weight of 7 grams.

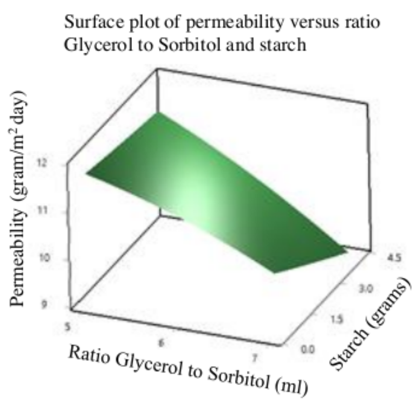
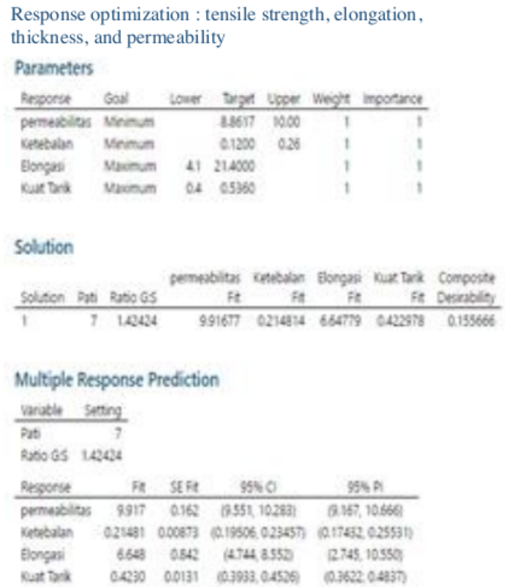


Fig. 9. Surface Characteristics Responses between Glycerol-Sorbitol Ratio and Starch Weight to Permeability

Based on Figure 10 shows treatment with the depiction of contour plots and surface plots with optimization using the Surface Response Method in the Minitab 18 software application, an optimization of the desired results with certain parameters can be carried out. The parameters of the optimization results are set to produce optimum edible film properties. The optimization results using the Surface Response Method resulted in optimum conditions at a glycerol-sorbitol ratio of 1.4242 or 5.875:4.125 with a of starch 7 g with edible film properties including tensile strength of 0.4230 MPa, elongation of 6.6478%, vapor permeability water is 9.9168 gr/m2day, and the thickness is 0.2148 mm. The values of these properties are mostly in accordance with the Japanese Industrial Standard (JIS), but for elongation it is still not suitable. Then it is taken from conditions that are close to the optimum results, namely the glycerol-sorbitol ratio of 1 or 5:5 and the amount of starch is 7 grams.


Response optimization : tensile strength, elongation, thickness, and permeability

Parameters

Response	Goal	Lower	Target	Upper	Weight	Importance
permeabilitas	Minimum		8.8617	10.00	1	1
Ketebalan	Minimum		0.1200	0.26	1	1
Elongasi	Maximum	4.1	21.4000		1	1
Kuat Tarik	Maximum	0.4	0.5360		1	1

Solution

Solution	Pati	Ratio G:S	permeabilitas Fit	Ketebalan Fit	Elongasi Fit	Kuat Tarik Fit	Composite Desirability
1	7	1.42424	9.91677	0.214814	6.64779	0.422978	0.155666

Multiple Response Prediction

Variable	Setting
Pati	7
Ratio G:S	1.42424

Response	Fit	SE Fit	95% CI	95% PI
permeabilitas	9.917	0.162	(9.551, 10.283)	(9.167, 10.666)
Ketebalan	0.21481	0.00873	(0.19506, 0.23457)	(0.17432, 0.25531)
Elongasi	6.648	0.842	(4.744, 8.552)	(2.745, 10.550)
Kuat Tarik	0.4230	0.0131	(0.3933, 0.4526)	(0.3622, 0.4837)

Fig. 10. Output Optimization Results with Minitab Software

4 Conclusion

The resulting edible films have different characteristics depending on the variation of the plasticizer ratio between glycerol and sorbitol and the amount of starch. These characteristics include the value of tensile strength with the largest value of 0.536 MPa and the smallest of 0.066 MPa, the elongation value with the largest value of 21.4% and the smallest of 4.1%, thickness with the largest value of 0.26 mm and the smallest of 0.12 mm, and water vapor permeability with the largest value 11.83 gram/m^2 day and the smallest 8.86 gram/m^2 day. Where some are in accordance with the Japanese Industrial Standard (JIS) and existing theories. The optimum results were in the composition ratio of glycerol to sorbitol 5:5 with the amount of corncob starch of 7 grams.

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