



Optimization of Making Edible Films based on Glycerol, Corn Oil and Whey Cheese using Rotatable Central Composite Design



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Abstract

Public awareness is getting higher about the importance of consuming healthy and safe food, as well as a sense of care for the surrounding environment. This opens up opportunities for the application of food preservation technology, including in packaging systems that are safe for food and safe for the environment. In food packaging, plastic is generally used as packaging, but with so much plastic being wasted, plastic packaging is one of the largest contributors to the volume of non-biodegradable waste. So it is necessary to seek alternatives to substitute plastic packaging, especially for food. Food products packaged by utilizing natural materials and waste into edible films will provide added value because they can be consumed. This study aims to make edible films made from glycerol, CaCO₃, corn oil and whey cheese and to optimize to obtain edible films that have tensile strength that meets Japanese Industrial Standards (1975) using Rotatable Central Composite Design (RCCD). The conditions of optimum tensile strength predicted were glycerol of 5.40%, CaCO₃ of 0.52 g/mL, corn oil of 5% and whey cheese of 15.10%. These factors gave an optimum tensile strength of 5.9592 MPa. The significant model terms are glycerol. Analysis of variance (ANOVA) indicates that the Rotatable Central Composite Design (RCCD) model was significant as evidenced from R² of 0.8520 and the model F-value of 6.58. Therefore, the Rotatable Central Composite Design (RCCD) model can be used to predict the tensile strength in making edible films.

Keywords: edible film; glycerol; corn oil; CaCO₃; Rotatable Central Composite Design; whey cheese

Introduction

Increasing public awareness of the importance of consuming healthy and safe food [1], as well as a sense of care for the surrounding environment, opens up opportunities for the application of food preservation technology, among others in packaging efforts using natural ingredients that are safe for humans and the environment [2]. Packaging is often done with plastic, but this can have a negative impact on the environment if more and more plastic waste is produced [3]. For this reason, a solution is sought to overcome the problem of plastic waste without causing other problems. An alternative solution that can be used to overcome this problem is to replace plastic with other packaging materials that are safe for humans and the environment [4,5].

In an effort to reduce plastic waste, it is necessary to develop packaging materials that can be consumed or easily degraded (edible or biodegradable

packaging) [6–10]. Various biodegradable natural polymers have been modified to produce environmentally friendly packaging such as protein and carbohydrate-based polymers. Biodegradable polymers have renewable properties, are available in abundant quantities and are relatively inexpensive. However, in its application as an edible film, biodegradable polymers need to be modified chemically, physically, mechanically or in combination with the addition of other polymers or plasticizers.

Products in the form of edible films as packaging materials are very promising to be realized in commercial production with equipment that is not too complicated (minimalist). In addition, the raw materials for making edible film are very abundant, the price is relatively cheap and easy to obtain in Indonesia [11,12]. The existence of this potential has caused this research to make edible films from whey

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Receive Date: 08 January 2023, Revise Date: 13 March 2023, Accept Date: 04 April 2023, First Publish Date: 05 April 2023

DOI: 10.21608/ejchem.2023.180016.7307

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cheese, glucomannan and corn oil. Making edible films is expected to support food security, especially in Indonesia.

To improve the quality of edible films made from whey cheese, glucomannan and corn oil, in this study, glycerol was added as a plasticizer and CaCO_3 as a filler in the process of making edible films [13]. Therefore, this study aims to study the effect of adding whey cheese, corn oil, glycerol and CaCO_3 on the tensile strength of edible films and determine the optimum composition of whey cheese, corn oil, glycerol and CaCO_3 in producing edible films according to the Japanese Industrial Standard (1975) (having a minimum tensile strength of 3.9 MPa) using the response surface methodology (RSM) [14–16].

Materials and Methods

Making Edible Films

Whey cheese, glucomannan flour, CaCO_3 and corn oil are mixed according to a certain ratio. Mixing was carried out by adding 100 mL of distilled water at 75–80°C. At the time of mixing, the mixture is stirred until a homogeneous mixture is obtained. The homogeneous mixture is then cooled to a temperature of 50°C. It is at this time that glycerol is added to the mixture. In addition, 0.5 mL of cinnamon oil was added to the mixture as an antimicrobial. The mixture that has been added to glycerol and cinnamon oil is then stirred again until a homogeneous mixture is obtained.

A homogeneous mixture containing whey cheese, glucomannan flour, CaCO_3 , corn oil, glycerol and cinnamon oil was poured into the mould. The mixture that has been poured into the mold is dried using room temperature and does not use a drying device such as an oven.

Edible Film Tensile Strength Test

The tensile strength testing process is carried out using a Machine Tester with the type WDW-20E. The specimen (edible film) being tested needs to be adjusted in size and shape according to the ASTM D882 standard. In testing the tensile strength for edible films according to the Japanese Industrial Standard (1975), what needs to be considered is the minimum standard value of tensile strength. The minimum standard value of tensile strength required by the Japanese Industrial Standard (1975) for edible films is 3.9 MPa.

Design of Experiment

A rotatable central composite design (RCCD) was

employed resulting in a total of 31 experiments using the Design-Expert (State-Ease Inc., Minneapolis, MN, USA) to optimize the chosen key factors namely glycerol (A), CaCO_3 (B), corn oil (C) and whey cheese (D). These variables each at two levels, low and high: A (4–6%), B (0.25–0.75%), C (4–6%) and D (10–20%) are presented in Table 1.

A statistical program package Design-Expert (State-Ease Inc., Minneapolis, MN, USA) was used for regression analysis of the data and for estimating the coefficient of the regression equation. The equations were validated by the analysis of variance (ANOVA) test. Model and regression coefficients were considered significant when the *p*-value was lower than 0.05. The experimental design for making edible film is shown in Table 2.

Results and Discussion

Effect of Addition of Plasticizer on Tensile Strength

The results of these measurements are closely related to the amount of plasticizer added to the process of making edible films. In making edible films in this study glycerol was used as a plasticizer. Plasticizers are able to reduce the internal hydrogen bonding of molecules thereby weakening the intermolecular forces of attraction of adjacent polymer chains and reducing the tensile strength at break. The addition of more than a certain amount of plasticizer will produce a film that has a lower tensile strength [17].

In Figure 1 it can be seen that the highest tensile strength value results from the addition of 6% glycerol. The addition of glycerol which exceeds 6% causes the tensile strength value of the edible film to decrease. This is because the addition of too much glycerol can cause glycerol molecules to occupy intermolecular spaces in the polymer chain. So that it is possible for glycerol molecules in sufficient quantities to tend to reduce the energy needed by other molecules to carry out a movement (easy to move). This phenomenon might cause a decrease in the tensile strength value if too much amount of glycerol is used as a plasticizer [18–20]. Edible film obtained with the addition of 6% glycerol has met the standard of tensile strength of edible films according to Japanese Industrial Standards (1975). This is because the edible film obtained with the addition of 6% glycerol has a tensile strength value above the minimum standard value of the required tensile strength of edible film, which is 3.9 MPa.

Table 1: Design summary for making edible film

Factors	Name	Units	Low Actual	High Actual	Low Coded	High Coded
A	Glycerol	%	4	6	-1	1
B	CaCO_3	%	0.25	0.75	-1	1
C	Corn oil	%	4	6	-1	1
D	Whey cheese	%	10	20	-1	1

Table 2: Rotatable central composite design (RCCD) matrix for making edible films

Run	Glycerol (%)	CaCO ₃ (%)	Corn Oil (%)	Whey Cheese (%)	Tensile Strength (MPa)		
					Experimental	Predicted	Residuals
1	5	0.50	5	15	5.5113	5.8828	-0.3715
2	5	0.50	5	5	3.9229	4.2876	-0.3647
3	6	0.75	6	20	4.2172	4.6092	-0.3920
4	4	0.25	6	20	3.9229	3.9560	-0.0331
5	5	0.50	5	15	6.1782	5.8828	0.2954
6	6	0.75	4	20	5.2477	4.8355	0.4122
7	6	0.75	4	10	4.9246	4.8066	0.1180
8	5	1.00	5	15	4.6503	4.6745	-0.0242
9	4	0.25	6	10	3.9229	3.8711	0.0518
10	5	0.50	5	15	5.5113	5.8828	-0.3715
11	4	0.75	4	20	3.9229	4.2787	-0.3558
12	5	0.50	7	15	4.1643	4.5252	-0.3609
13	5	0.50	5	15	5.8111	5.8828	-0.0717
14	3	0.50	5	15	3.1433	3.2554	-0.1121
15	6	0.25	6	20	5.2477	4.8214	0.4263
16	4	0.75	6	10	3.9229	4.0125	-0.0896
17	6	0.25	6	10	5.2477	4.8070	0.4407
18	5	0.00	5	15	3.9229	4.4477	-0.5248
19	5	0.50	5	15	6.1782	5.8828	0.2954
20	6	0.25	4	10	4.3111	4.3910	-0.0799
21	5	0.50	5	25	4.2172	4.4014	-0.1842
22	5	0.50	5	15	5.8111	5.8828	-0.0717
23	5	0.50	3	15	4.4327	4.6207	-0.1880
24	4	0.75	6	20	4.3111	3.7672	0.5439
25	4	0.25	4	20	4.3111	4.1698	0.1413
26	6	0.75	6	10	5.2477	4.9249	0.3228
27	7	0.50	5	15	4.3111	4.7480	-0.4369
28	4	0.25	4	10	4.2172	3.7403	0.4769
29	5	0.50	5	15	6.1782	5.8828	0.2954
30	4	0.75	4	10	4.2172	4.1794	0.0378
31	6	0.25	4	20	4.9246	4.7501	0.1745

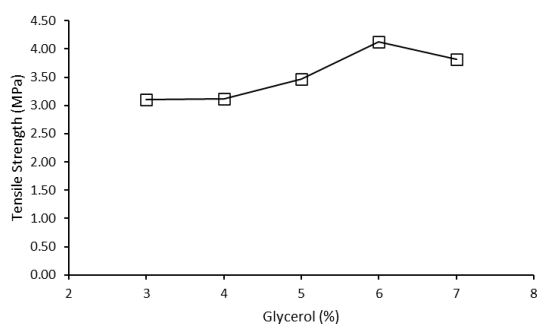


Figure. 1: The effect of adding glycerol on tensile strength (Edible film was made with the following composition: 0.25% CaCO₃; 2% corn oil; 20% whey cheese).

Effect of Addition of CaCO₃ on Tensile Strength

In addition to the amount of plasticizer, another factor that needs to be considered in making edible films is the amount of filler. In making edible films in this study CaCO₃ was used as a filler [21,22]. The effect of adding CaCO₃ on tensile strength can be seen in Figure 2. Edible films obtained without and with the

addition of 0.20-1.00% glycerol still do not meet the tensile strength standards of edible films according to Japanese Industrial Standards (1975). This is because the edible film obtained without and with the addition of 0.20-1.00% glycerol has a tensile strength value below the minimum standard value for the required tensile strength of edible film, which is 3.9 MPa.

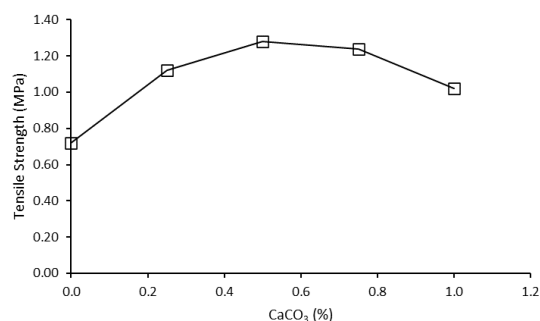


Figure. 2: The effect of adding CaCO₃ on tensile strength (Edible film is made with the composition: 2% glycerol; 2% corn oil; 20% whey cheese).

Effect of Addition of Corn Oil on Tensile Strength

In making edible films in this study corn oil was used as a source of lipids. The presence of a fairly high oleic acid content in corn oil is a consideration and reason for choosing corn oil in this study [23]. This is because oleic acid has the potential to increase the tensile strength of edible films [24].

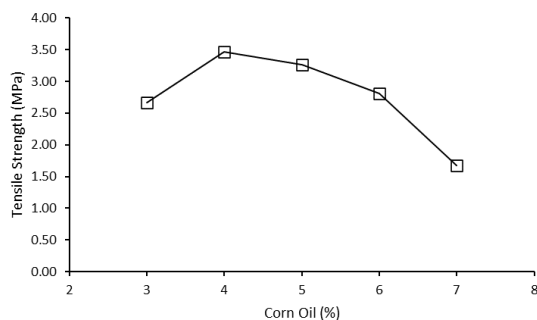


Figure 3: The effect of adding corn oil on tensile strength (Edible film is made with the composition: 2% glycerol; 0.25% CaCO₃; 20% whey cheese).

The effect of adding corn oil on tensile strength can be seen in Figure 3. Edible film obtained by adding 3-7% corn oil still does not meet the tensile strength standards of edible films according to Japanese Industrial Standards (1975). This is because the edible film obtained with the addition of corn oil as much as 3-7% has a tensile strength value below the minimum standard value for the required tensile strength of edible film, which is 3.9 MPa.

Effect of Whey Cheese Addition on Tensile Strength

One of the basic ingredients in making edible films is whey protein. Whey protein can produce edible films that are transparent, soft, flexible and have properties that can hold the aroma of the food product it is coated with [25,26]. In this study, the whey used was whey cheese from the production of mozzarella cheese.

The effect of adding whey cheese on tensile strength can be seen in Figure 4. Edible film obtained by adding 5-25% whey cheese still does not meet the tensile strength standards of edible films according to Japanese Industrial Standards (1975). This is because the edible film obtained by adding whey cheese as much as 5-25% has a tensile strength value below the minimum standard value for the required tensile strength of edible film, which is 3.9 MPa.

Table 3: Analysis of variance (ANOVA) for response surface quadratic model to identify significant factors affecting the tensile strength of edible film (Std. Dev.: 0.43; R-Squared: 0.8520; Adj R-Squared: 0.7226; Pred R-Squared: 0.2708; Adeq Precision: 8.876)

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F
Model	16.68	14	1.19	6.58	0.0003
A	3.34	1	3.34	18.45	0.0006*
B	0.077	1	0.077	0.43	0.5232
C	0.014	1	0.014	0.076	0.7869

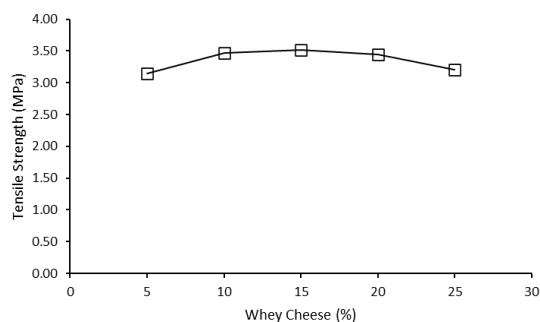


Figure 4: The effect of adding whey cheese on tensile strength (Edible film is made with the composition: 2% glycerol; 0.25% CaCO₃; 2% corn oil).

Tensile Strength Optimization using Rotatable Central Composite Design (RCCD)

Table 2 presents the Rotatable Central Composite Design (RCCD) matrix and the tensile strength obtained for each experimental run. Analysis of variance for the response surface model is presented in Table 3. The analysis indicates that the model F-value of 6.58 implies the model is significant. There is only a 0.03% chance that a "Model F-value" this large could occur due to noise. The model also has a satisfactory level of adequacy (R²). In this study A, A², B², C² and D² are significant model terms (values of "Prob > F" < 0.05).

The "Pred R-Squared" of 0.2708 is not as close to the "Adj R-Squared" of 0.7226 as one might normally expect. This may indicate a large block effect or a possible problem with our model and/or data. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable [27–30]. The ratio of 8.876 indicates an adequate signal. This model can be used to navigate the design space. The response surface curves are plotted to understand the interaction of the variables and the optimum level of each variable for maximum response (tensile strength). The response surface curves for making edible film are presented in Figure 5. Final equation in terms of coded factors is given by Equation 1.

$$\begin{aligned} \text{Tensile Strength (MPa)} = & 5.88 + 0.37 \times A + 0.057 \times B - \\ & 0.024 \times C + 0.028 \times D - 0.47 \times A^2 - 0.33 \times B^2 - 0.33 \times C^2 - \\ & 0.38 \times D^2 - 5.869 \times 10^{-3} \times A \times B + 0.071 \times A \times C - \\ & 0.018 \times A \times D - 0.074 \times B \times C - 0.083 \times B \times D - 0.086 \times C \times D \end{aligned} \quad (1)$$

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F
D	0.019	1	0.019	0.11	0.7476
A ²	6.32	1	6.32	34.92	< 0.0001*
B ²	3.12	1	3.12	17.24	0.0007*
C ²	3.07	1	3.07	16.93	0.0008*
D ²	4.23	1	4.23	23.35	0.0002*
AB	5.511E-004	1	5.511E-004	3.043E-003	0.9567
AC	0.081	1	0.081	0.45	0.5123
AD	4.960E-003	1	4.960E-003	0.027	0.8706
BC	0.089	1	0.089	0.49	0.4943
BD	0.11	1	0.11	0.60	0.4491
CD	0.12	1	0.12	0.66	0.4299
Residual	2.90	16	0.18		
Lack of Fit	2.35	10	0.23	2.57	0.1299
Pure Error	0.55	6	0.091		
Cor Total	19.58	30			

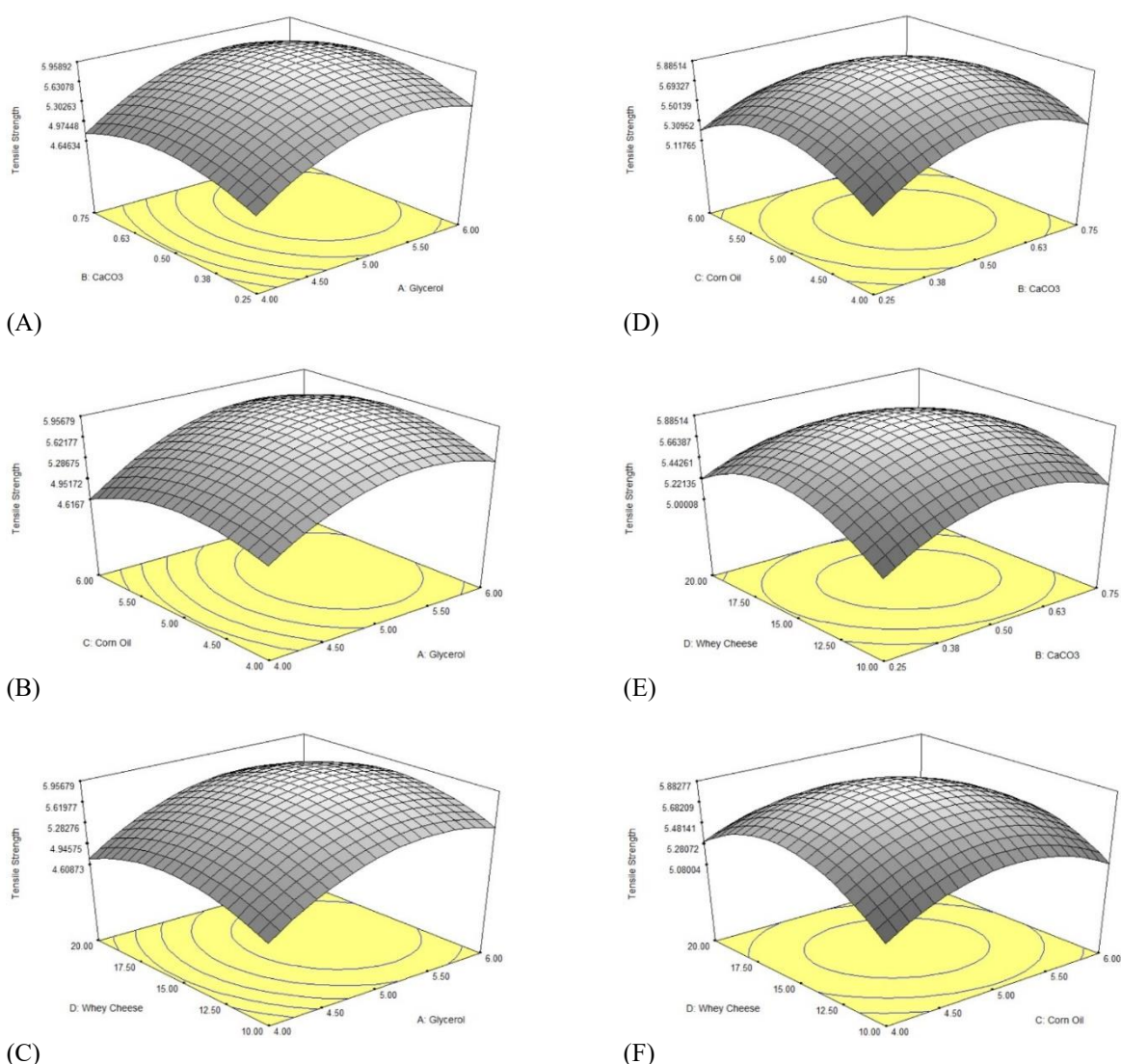


Figure. 5: Response surface showing effect of: (a) glycerol and CaCO_3 on the tensile strength at constant corn oil (5%) and whey cheese (15%); (b) glycerol and corn oil on the tensile strength at constant CaCO_3 (0.50%) and whey cheese (15%); (c) glycerol and whey cheese on the tensile strength at constant CaCO_3 (0.50%) and corn oil (5%); (d) CaCO_3 and corn oil on the tensile strength at constant glycerol (5%) and whey cheese (15%); (e) CaCO_3 and whey cheese on the tensile strength at constant glycerol (5%) and corn oil (5%); (f) corn oil and whey cheese on the tensile strength at constant glycerol (5%) and CaCO_3 (0.50%)

Experimental data generated in Table 2 in terms of actual values need to be converted first in the form of coded values before it is substituted into Equation 1 and the predicted tensile strength was obtained. Actual tensile strength and the predicted tensile strength are presented in Table 2. Comparison of these values indicates that the 2 sets of values are in close agreement. This suggests good reliability of the model as also evidenced from the statistical parameters of the model such as standard deviation of 0.43, R^2 of 0.8520 and F-value of 6.58. This shows fitness of the data for the model.

Numerical optimization method was used to predict optimum condition for the response (tensile strength) using the Design-Expert (State-Ease Inc., Minneapolis, MN, USA). The glycerol of 5.40%, CaCO_3 of 0.52 g/mL, corn oil of 5% and whey cheese of 15.10% were obtained among the solutions for the optimum conditions (maximize) for the tensile strength. This condition gives the tensile strength of 5.9592 MPa.

Conclusions

The optimization model was developed using Rotatable Central Composite Design (RCCD) for prediction of tensile strength in making edible films. The Rotatable Central Composite Design (RCCD) model fits experimental data. Glycerol is a factor that has a significant effect in making edible films. The conditions of optimum tensile strength predicted were glycerol of 5.40%, CaCO_3 of 0.52 g/mL, corn oil of 5% and whey cheese of 15.10%, which gave a tensile strength of 5.9592 MPa. The edible film obtained based on this optimization already meets the tensile strength standards of edible films according to Japanese Industrial Standards (1975). This is because the edible film obtained based on optimization has a tensile strength value above the minimum standard value of the required tensile strength of edible film, which is 3.9 MPa.

Conflicts of interest

There are no conflicts to declare

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