Optimization of biodegradable plastic production using response surface methodology

M Riza1*, S Syaubari1, A Andriansyah2, R Dewi3 and L Ernita1
1 Chemical Engineering Department, Universitas Syiah Kuala, Jl. Tgk. Syech Abdul Rauf No.7 Banda Aceh 23111 Aceh, Indonesia
2 Laboratory of Industrial Computation and Optimization, Industrial Engineering, Universitas Syiah Kuala, Jl. Tgk. Syech Abdul Rauf No.7 Banda Aceh 23111 Aceh, Indonesia
3 Chemical Engineering Department, Universitas Malikussaleh Lhokseumawe, Aceh, Indonesia

*email: medyan_riza@unsyiah.ac.id

Abstract. Optimization is a searching technique for variable values considered optimum, effective and efficient, in order to reach the desirable results. An experiment was carried out to see the relationship between response variable and the independent variable. Several comparison tests can be obtained to gain the level that will create optimal response. The aim of this research is to apply the Response Surface methodology in order to obtain the optimum condition for the process variable in producing biodegradable plastic with lemongrass as antioxidant. Fixed variables in this research are tapioca starch, 69-79°C gelatinization temperature and the total mixture weight that consists of starch, poly(NIPAM) chitosan, lemongrass oil, acetic acid and water. The response variables are tensile strength and break of elongation of each biodegradable plastic that is produced. The application of response surface methodology on each biodegradable plastic can be used to obtain the independent variable that makes the optimal response variable. The predicted result of the optimum value for tensile strength and elongation of break lies on the composition treatment of Poly(NIPAM)-chitosan 0.35 grams, glycerol 3.5 grams and 36.55% lemongrass oil with tensile strength result of 3.98 MPa and elongation of break of 36.55%.

1. Introduction
The biggest waste around the world today is from plastic-based materials [1]. Plastic is commonly used in food packaging, packaging from household items as well as in other sectors. Plastic is a kind of material that is not easy to be degraded by microbes. The number of plastic waste is increasing every year and can cause many problems, such as environmental pollution, the spreading of diseases and also to flood problems [2]. Many efforts have been done to tackle the plastic waste landfills, they are: recycling plastic and burning plastic. However on there is only a few percentage of plastic that can be recycled; therefore another alternative solution is needed to handle this situation.

Biodegradable plastic can be commonly interpreted as plastic that can be recycled and can be degraded naturally. Conventional plastic is plastic that is made up of petroleum, coal and natural gas. Meanwhile biodegradable plastic is plastic that is made up of natural polymers such as cellulose (starch), collagen, casein, protein or lipid [3]. Biodegradable plastic is a material that its chemical structure can change under certain circumstances; in which microorganisms such as, bacteria, fungi and algae, can affect its properties [5]. Starch is the main material in producing biodegradable plastic, because starch
has biodegradable properties, easier processing and has an economical value. Starch produces materials such as, cassavas, bananas, corns, potatoes and others are easily available in Indonesia [4].

Many researchers have utilized starch as main material in producing biodegradable plastic. One of them is biodegradable plastic that is made up of starch, chitosan and glycerol mixture [2]. Glycerol functions to fix the lack of plastic properties that are starch-based which is elastic, flexible and smooth. Glycerol has a low molecular weight so that it can be considered to be used as plasticizer [4]. Chitosan is an additional material in producing biodegradable plastic that can function to fix the transparency of plastic produced [5]. Chitosan contains antioxidants and antimicrobials that can be used as substances for packaging [6]. Chitosan is a substance that can inhibit oxygen on the packaging [7]. Chitosan also functions as preservative for biodegradable plastic. Chitosan’s properties are water-resistant, non-toxic and anti-bacterial that can function as preservatives and anti-microbial.

N-isopropylacrylamide (NIPAM) is an organic compound that can be synthesized to become polymer or can be crosslinking polymer that is biodegradable in nature. NIPAM is one of the acrylamide monomers that are white, odourless and shaped as solid crystals that are soluble in water and very reactive through amide reaction or by its duplicate bonds. This compound can make a long polymer chain that is called poly(NIPAM). Poly(NIPAM) is a thermo sensitive material that can be shaped into hydrogel. The application of poly(NIPAM) has a lot been done in various fields, such as, acting as electrophoresis membrane, biomedical membrane, flocculant that can separate solids and liquids in liquid waste processing, microsphere synthesis for drug transmission, thickener or to create other compounds. However, the usage of poly(NIPAM) has other deficient characteristics, so that there are other researches that took place in order to fix the deficiencies. One of them is doing crosslinking with other monomers. Poly(NIPAM) is a smart polymer that is susceptible to change in temperatures. Because of that, chemical modification in a manner of croslinking towards poly(NIPAM) and chitosan in the process of producing biodegradable plastic is hoped to increase the physical quality, thermal and plastic degradation in the plastic produced. The addition of poly(NIPAM) in the production of biodegradable plastic can create a stronger plastic structural matrix.

In order to understand how far an optimum process is affected by a number of variables, a large amount of experiment data is needed, where it requires a long time to be obtained automatically and it also requires a large cost. There are several mathematical and statistical techniques that are used to as approaches in order to gain understanding of the optimum condition of a process, without the need of many data. One of the techniques is response surface methodology (RSM). [8] uses artificial neural network to create a mathematical model for biodegradable plastic. Cinnamon oil is added in to NIPAM-chitosan and glyserol. [9] optimizes the results of linear and multilinear regression results using simulated annealing algorithm to obtain a best composition of biodegradable plastic produced.

Almost similarly with previous works, this study investigates the combination of biodegradable plastic compositions by adding lemongrass oil. RSM incorporates statistical and mathematical techniques that can be used to generate and analyze responses (composition if poly(NIPAM)-chitosan, glycerol and lemongrass oil), that are affected by some free variables. The RSM can also be used as an effort in looking for the right function to predict responses and determine the values of independent variables that can optimize responses, with achieving the aim of optimizing responses [10]. Many researcher have used this method to obtain best results. [11] uses the this method to optimize preparation of biocomposites based on poly and durian peel cellulose. The biodegradability of acrylic acid grafting onto polypropylene has been optimized by RSM [12].

2. Methodology
2.1. Equipment and materials
The equipment that will be used in this research to produce biodegradable plastic are, magnetic stirrer, beaker glass, Erlenmeyer, chemical glass, aluminium foil, digital weighing scale, hot plate, desiccator, casting glass, oven, stirrer stick, tweezers and petri dish. The materials needed for this research to produce biodegradable plastic are tapioca starch, glycerol, chitosan, lemongrass oil, 1% acetic acid,
ethanol, NIPAM monomers, distilled water, azobisisobutirnitril initiator (AIBN), and mercaptopropionionic acid initiator (MPA).

2.2. Research design
This research uses Central Composite Design (CCD) and uses Design Expert version 10 three factors, they are: what is included in the RSM to see the optimum condition towards the effects of treatment of the poly(NIPAM)-chitosan, glycerol and lemongrass oil treatment towards the results of tensile strength and elongation of break of the biodegradable plastic produced. The trial design of CCD can be seen in Table 1.

2.3. Research procedure
NIPAM polymerization is carried out by dissolving 10 grams of NIPAM into 20 ml of Ethanol in an Erlenmeyer. Then 1 ml AIBN is added and 0.6 ml MPA. The mixture is stirred until it is completely dissolved. The mixture is then incubated with a temperature of 60°C for 20 hours. The product produced is precipitate that can be taken by adding diethyl ether solution. The sediment that is obtained is then dried inside the oven for 12 hours at 80°C temperature.

Table 1. Limitations and change level variable/independent variables.

| Variable                  | Limitations and Level |
|---------------------------|-----------------------|
|                           | -1.682 (-α) | -1 | 0 | +1 | 1.682 (α) |
| Pnipam-chitosan (X_1)     | 0.11        | 0.35 | 0.45 | 0.55 | 0.93 |
| Glycerol (X_2)            | 0.48        | 1.50 | 2.50 | 3.50 | 5.89 |
| Lemongrass oil (X_3)      | 0.08        | 0.25 | 0.35 | 0.45 | 0.76 |

Tapioca starch (10 grams) is dissolved with 50 ml of distilled water in a chemical glass and stirred for 25 minutes. Then when the mixture of starch and distilled water is homogenous, the starch mixture is placed on top of a hot plate. Hot plate is used to speed up the reaction by increasing the temperature. Magnetic stirrer was used to stir the mixture in a homogenous form. Magnetic stirrer was used to avoid lumps within the starch during heating and to ensure even heat distribution. The starch is heated until it reaches 70°C in temperature for 20 minutes.

Next, glycerol plasticizer, poly(NIPAM)-chitosan and lemongrass oil is added into the starch. During the addition of glycerol, the poly(NIPAN)-chitosan, lemongrass oil and starch mixture must be kept stirred for 15 minutes to avoid lumps and speed up the mixture homogenization between the starch and glycerol, as well as the chitosan and lemongrass oil. After the mixture thickens, the chemical glass is removed from the hot plate. The solution should be kept stirred until it reaches normal temperature around 25 – 30°C for 30 minutes to keep the thickness of the mixture stable.

After the temperature of the starch solution is normal, then casting is conducted on top of a glass plate with 1mm thickness that has been given tape on the sides. The purpose of the tape is to keep the solution intact within the glass plate. The pouring of the starch solution has to be done slowly and carefully. The thin later that is formed on top of the glass plate is entered into an oven with a temperature of 80°C and kept at it for 4 hours to harden and dry. The thin layer from the glass plate is then removed and kept inside a desiccator for a day in a place with no direct sunlight.

3. Result and Discussion
This research is conducted with 19 treatments where X_1 variable for the composition of Poly(NIPAM)-chitosan mixture, X_2 for the composition of glycerol and X_3 for the composition of lemongrass oil. The response variables are the tensile strength and elongation of break of the plastic produced.
Table 2. Experiment data results and model prediction results towards tensile strength and elongation of break.

| Run | Pnipam-chitosan (gr) X1 | Glycerol (gr) X2 | Lemongrass oil (gr) X3 | Tensile Strength (MPa) Data | Prediction | % Error | Elongation of Break (%) Data | Prediction | % Error |
|-----|------------------------|------------------|------------------------|---------------------------|------------|---------|-----------------------------|------------|---------|
| 1   | 0.35                   | 1.5              | 0.45                   | 2.97                      | 3.29       | -10.77  | 25.92                       | 25.86      | 0.23    |
| 2   | 0.55                   | 3.5              | 0.25                   | 3.57                      | 3.88       | -8.74   | 31.4                        | 25.81      | 17.81   |
| 3   | 0.45                   | 2.5              | 0.08                   | 3.86                      | 3.94       | -2.05   | 31.32                       | 28.42      | 9.25    |
| 4   | 0.45                   | 2.5              | 0.35                   | 3.77                      | 3.62       | 3.95    | 14.48                       | 26.63      | -83.94  |
| 5   | 0.45                   | 0.48             | 0.35                   | 3.44                      | 3.21       | 6.75    | 6.32                        | 9.52       | -50.6   |
| 6   | 0.45                   | 2.5              | 0.35                   | 3.1                       | 3.62       | -16.81  | 25.64                       | 26.63      | -3.88   |
| 7   | 0.45                   | 2.5              | 0.35                   | 2.78                      | 3.62       | -30.25  | 25.32                       | 26.63      | -5.19   |
| 8   | 0.35                   | 1.5              | 0.25                   | 3.02                      | 3.03       | -0.2    | 18.92                       | 10.22      | 45.98   |
| 9   | 0.35                   | 3.5              | 0.25                   | 4.08                      | 4.21       | -3.26   | 31.08                       | 36.55      | -17.6   |
| 10  | 0.45                   | 5.89             | 0.35                   | 4.24                      | 4.82       | -13.7   | 16.32                       | 17.15      | -5.09   |
| 11  | 0.45                   | 2.5              | 0.35                   | 3.73                      | 3.62       | 2.92    | 19.96                       | 26.63      | -33.44  |
| 12  | 0.45                   | 2.5              | 0.35                   | 4.17                      | 3.62       | 13.16   | 31.04                       | 26.63      | 14.19   |
| 13  | 0.35                   | 3.5              | 0.45                   | 3.77                      | 4.02       | -6.62   | 55.32                       | 41.11      | 23.19   |
| 14  | 0.93                   | 2.5              | 0.35                   | 2.79                      | 2.89       | -3.58   | 11.48                       | 11.29      | 1.61    |
| 15  | 0.45                   | 2.5              | 0.76                   | 3.66                      | 4.11       | -12.33  | 39                          | 41.6       | -6.67   |
| 16  | 0.55                   | 1.5              | 0.45                   | 3.17                      | 3.67       | -15.75  | 34.76                       | 23.64      | 32      |
| 17  | 0.11                   | 2.5              | 0.35                   | 2.75                      | 3.18       | -15.76  | 35.44                       | 37.76      | -6.56   |
| 18  | 0.55                   | 1.5              | 0.25                   | 3.08                      | 3.57       | -15.92  | 17.24                       | 24         | -39.2   |
| 19  | 0.55                   | 3.5              | 0.45                   | 2.79                      | 3.52       | -26.31  | 11.32                       | 14.37      | -26.93  |

From Table 2, it can be concluded that the composition of poly(NIPAM)-chitosan, glycerol and lemongrass oil have an effect towards the values of tensile strength and elongation of break of the biodegradable plastic that is produces. The highest tensile strength value is obtained from this experiment based on the results prediction model is from run 10, which is 4.24 MPa on the 0.45 gram poly(NIPAM)-chitosan, 5.89 grams glycerol and 0.35 gram lemongrass oil composition. Meanwhile, the lowest tensile strength value is obtained from the experiment on run 17, which is 2.75 MPa with the composition of 0.11 gram poly(NIPAM)-chitosan, 2.5 grams glycerol and 0.35 gram lemongrass oil.

The highest value for elongation of break obtained from the experiment on run 13, which is 55.52 with the composition of 0.35 gram poly(NIPAM)-chitosan, 3.5 grams glycerol and 0.45 gram lemongrass oil. Meanwhile the highest elongation of break value is obtained from the results prediction model in on experiment run 15 which is 41.60% with the composition of 0.45 gram poly(NIPAM)-chitosan, 2.5 grams glycerol and 0.76 gram lemongrass oil. Meanwhile the lowest elongation of break value is obtained from the experiment run 5, which is 6.32% with the composition of 0.45 gram poly(NIPAM)-chitosan, 0.48 gram glycerol and 0.35 gram lemongrass oil. For the lowest elongation of break value that was obtained based on the results prediction model is the same with the one from run 5, which is 9.52%.

However for the centre point condition; 5 experiments were conducted with the composition of poly(NIPAM)-chitosan 0.45 gram, 2.5 grams glycerol and 0.35 gram lemongrass oil. The tensile strength value that was obtained on the centre point run 4 was 3.77 MPa, run 6 was 3.1 MPa, run 7 was 2.78 MPa, run 11 was 3.73 MPa and run 12 was 4.17 MPa. Meanwhile the elongation of break value obtained on the centre point on run 4 was 14.48%m run 6 was 25.64%, run 7 was 25.32%, run 11 was 19.96% and run 12 was 31.04%

Table 3 and table 4 results show that the statistical models are insignificant. This is due to the value of Probability F is bigger than 0.0500 which is 0.3056 for tensile strength model and 0.1098 for elongation of break. The Prob > F value serves to look into the suitability between every coefficient and the strength of interaction between each independent variables. If the Prob > F value is less than F-value, then the variable will give a significant effect towards the response.
Table 3. Tensile strength analysis of variance.

| Source | Sum of square | df  | Mean square | F value | P-value Prob>F | Characteristic |
|--------|---------------|-----|-------------|---------|----------------|----------------|
| Model  | 2.68          | 9   | 0.3         | 1.42    | 0.3056         | Insignificant  |
| A      | 0.005         | 1   | 0.005       | 0.024   | 0.881          |                |
| B      | 0.46          | 1   | 0.46        | 2.17    | 0.1745         |                |
| C      | 0.2           | 1   | 0.2         | 0.95    | 0.3548         |                |
| A²     | 0.38          | 1   | 0.38        | 1.82    | 0.2103         |                |
| B²     | 0.014         | 1   | 0.014       | 0.065   | 0.8049         |                |
| C²     | 0.16          | 1   | 0.16        | 0.76    | 0.4063         |                |
| AB     | 0.5           | 1   | 0.5         | 2.39    | 0.1562         |                |
| AC     | 0.064         | 1   | 0.064       | 0.3     | 0.5955         |                |
| BC     | 0.25          | 1   | 0.25        | 1.17    | 0.3067         |                |
| Residual | 1,890        | 9   | 0.21        |         |                |                |
| Lack of Fit | 0.64      | 5   | 0.13        | 0.41    | 0.8232         | Insignificant  |
| Pure error | 1.25        | 4   | 0.31        |         |                |                |
| Cor Total | 4.58         | 18  |             |         |                |                |

Table 4. Elongation of break analysis of variance.

| Source | Sum of square | df  | Mean square | F value | P-value Prob>F | Characteristic |
|--------|---------------|-----|-------------|---------|----------------|----------------|
| Model  | 1679.81       | 9   | 186.65      | 2.35    | 0.1098         | Insignificant  |
| A      | 358.74        | 1   | 358.74      | 4.51    | 0.0626         |                |
| B      | 302.3         | 1   | 302.3       | 3.8     | 0.083          |                |
| C      | 26.59         | 1   | 26.59       | 0.33    | 0.5772         |                |
| A²     | 300.62        | 1   | 300.62      | 3.78    | 0.0837         |                |
| B²     | 128           | 1   | 128         | 1.61    | 0.2563         |                |
| C²     | 61.38         | 1   | 61.38       | 0.77    | 0.4024         |                |
| AB     | 363.18        | 1   | 363.18      | 4.57    | 0.0613         |                |
| AC     | 81.53         | 1   | 81.53       | 1.03    | 0.3376         |                |
| BC     | 715.43        | 9   | 79.49       |         |                |                |
| Residual | 557.02       | 5   | 111.4       | 2.81    | 0.169          | Insignificant  |
| Lack of Fit | 158.41      | 4   | 39.6        |         |                |                |
| Pure error | 2395.24     | 18  |             |         |                |                |

Table 5 and table 6 are the results of variance analysis for each tensile strength and elongation of break statistical models. It can be seen that each of the tensile strength and elongation of break $R^2$ values are 0.5865 and 0.7013.

This matter identifies that variables $X_1$, $X_2$ and $X_3$ gave a big effect towards the tensile strength and elongation of break models. The suggested model for tensile strength and elongation of break is quadratic model. The actual mathematical model obtained is to predict the value of tensile strength (TS) and elongation of break (EOB) is as follows:

$$TS = -1,20552 + 10,11597X_1 + 1,50665X_2 + 2,03294X_3 - 2,1875X_1X_2 - 4,125X_1X_3 - 1,4125X_2X_3 - 3,4251X_1^2 + 0,027594X_2^2 + 3,40108X_3^2$$

(1)

$$EOB = -134,37538 + 259,98289X_1 + 51,96163X_2 + 215,34639X_3 - 61,3X_1X_2 - 400X_1X_3 - 27,7X_2X_3 + 0,9497X_1^2 - 2,0833X_2^2 + 63,42999X_3^2$$

(2)
Table 5. Tensile strength statistical model summary.

| Model source | Std Dev. | R-squared | Adjust R-square | Predicted R-squares | press |
|--------------|----------|-----------|-----------------|---------------------|-------|
| Linear       | 0.48     | 0.2374    | 0.0849          | -0.434              | 6.57  |
| 2FI          | 0.49     | 0.3588    | 0.0383          | -0.4637             | 6.70  |
| Quadratic    | 0.46     | 0.5865    | 0.1729          | -23.107             | 15.16 suggested |
| Cubic        | 0.56     | 0.7264    | -0.2313         | + aliased           |

Table 6. Elongation of break statistical model summary.

| Model source | Std Dev. | R-squared | Adjust R-square | Predicted R-squares | press |
|--------------|----------|-----------|-----------------|---------------------|-------|
| Linear       | 10.84    | 0.2642    | 0.117           | -0.3688             | 3278.62 |
| 2FI          | 10.30    | 0.4687    | 0.2031          | -0.6956             | 4059.96 |
| Quadratic    | 8.92     | 0.7013    | 0.4026          | -1.3278             | 5575.73 suggested |
| Cubic        | 6.29     | 0.9339    | 0.7024          | + aliased           |

Tensile strength is defined as the maximum force that can withstand by plastic until it breaks. The testing of tensile strength is conducted to find out the durability of a material towards the imposition on the flexible point and to find out the plastic’s elasticity. Figure 1(a) shows the value of tensile strength increases as the composition of poly(NIPAM)-chitosan and glycerol increase. The increase of tensile strength is affected by the higher number of poly(NIPAM)-chitosan used. This is because the higher the number of poly(NIPAM)-chitosan, the more hydrogen bonds that can be found in the plastic, therefore the chemical bonds become stronger and harder to break because it will require a larger energy to break the bond [1]. Figure 1(b) shows the value of tensile strength having no significant changes as the composition of poly(NIPAM)-chitosan and lemongrass oil composition increases. This is due to the increase of lemongrass oil can decrease the mechanical nature so that it can be easily broken. This happens because there is a dispersed oil phase within the plastic matrix that causes the plastic structure to run into discontinuity process. Elongation of break is defined as the percentage of length change in the plastic when it is pulled it breaks. The higher the value of elongation of break then the plastic is more elastic, so that the material pulled is more stretched. Plastic with lower elongation of break will be brittle in nature [8]. In figure 1[d], it can be seen that elongation of break will increase as the composition of glycerol increases and the elongation of break decreases as the poly(NIPAM)chitosan composition increases. This is due to the plasticizer’s ability to reduce brittleness and increase the flexibility of the plastic. Meanwhile with the increase of poly(NIPAM)-chitosan, it decreases the percentage of elongation of break. This is due to the smaller distance between the intermolecular bonds. The addition of lemongrass oil also tends to decrease the percentage of elongation break. This is due to the fact that oil is hydrophobic in nature so that it does not dissolve in the starch solution that will cause less elasticity on the plastic and can be broken easily.
Figure 1. 3D Plot of the Poly(NIPAM)-chitosan, glycerol, and lemongrass oil composition effects towards the values of tensile strength (a, b, c) and elongation of break (d, e, f).

4. Conclusion
This study investigates the optimum composition of biodegradable plastic production. Lemongrass oil is used as antioxidant in the experiments. The experiments are designed using RSM to get optimal
composition formed biodegradable plastic. The independent variables in this experiments are poly(NVPAM)-chitosan, glycerol, and lemongrass oil. The tensile strength and elongation of break values are affected by the composition of Poly(NIPAM)-chitosan, glycerol, and lemongrass oil. Based on analysis, the prediction results of optimum value of tensile strength and elongation of break depended on the composition treatment of 0.35 grams of Poly(NIPAM)chitosan, 3.5% glycerol and 36.55% lemongrass oil. Based on the Central Composite Design (CCD) on Design Expert 10 software, it was found out that the suitable model for tensile strength and elongation of break values is a quadratic model where each of the $R^2$ value are 0.5865 and 0.7013.

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