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Optimisation of Tensile Strength and Weight Loss via Taguchi and Response Surface Method for Natural Rubber Latex Film Consisting Cassava Peel as a Bio-Based Filler

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Abstract: Disposal latex and synthetic rubber gloves is troublesome such that disposal via incineration and land fill may release poisonous gasses and contaminate soil and water, respectively. As solution to latex and synthetic rubber, biodegradable glove is extensively studied. A bio-based filler is extracted from food waste and blended into natural rubber latex (NRL) as a composite NRL. The effect of biodegradability of composite NRL was studied by varying the loading of bio-based filler in a form of starch dispersion and blended into NRL mixture. Herein some amount of starch can be extracted from cassava peel to be incorporated in NRL for a sustainable and yet biodegradable glove. Previous work on incorporation of cassava-peel filler in NRL has shown a biodegradability without compromising the pristine strength of NRL film at 50% loading starch. In this project, tensile strength and weight loss of prepared composite NRL films were optimised via Taguchi and Response Surface Method (RSM) by means of Design Expert software by varying starch/filler loading, curing temperature and curing drying duration. Due to inadequate data, the optimisation from that previous prepared composite NRL was compared with similar work which utilising NRL and bio-based filler. For Pulungan (2020) study, it can be concluded that the tensile strength of cassava peel starch biodegradable film has the best condition at 50°C to 60°C at approximately 5.5 hours. Elongation optimum conditions shows contrast value of temperature and time. Meanwhile, for Wendy (2020) study, it shows the best percentage loading of cassava-peel starch is at 20% to achieve high stress and strain at break. The optimised mechanical properties via Taguchi and RSM are rather different and hence validation on mechanical properties at above mentioned conditions need to be performed experimentally.

Keywords: Biodegradable glove, tensile strength, cassava peel, taguchi, response surface method

1. Introduction

Despite the benefits from natural rubber that resulted to the modernisation of the world, natural rubber results in a serious environmental problem due to its highly polluted effluents. The growth of this natural rubber industry generates large amount effluents from its processing operations. This phenomenon becomes a great problem because of wastewater discharged contains high level biological oxygen demand (BOD), chemical oxygen demand (COD), suspended solids (SS) and ammonia [1]. During the manufacturing of rubber materials, there are high possibility of pollutants and green-house-effect gases emitted due to the leaching out of chemicals as well as during vulcanisation that undergoes at high temperature [2]. These gases are coming from the original compounding ingredients namely plasticizers, antioxidants and residual monomers or polymer oligomers and reaction products from the crosslinking [2]. Improper ventilation during disposal process may produce unpleasant odour and dust. This can have adverse effect on the workers mainly the respiratory system [2]. High concentration of ammonia in the latex posed a danger to the

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environment. This could lead to endanger to aquatic organisms due to high level of nitrogen and ammonia content in the discharged wastewater. Instead of environment, this gives a serious threat to human health and sustainability in the long term and become health risks to human in future [3]. Large amounts of one-use disposable latex gloves are thrown into the environment annually. Medical glove wastes undergo thermal destruction (incineration) and gloves from domestic and industrial wastes end up in municipal landfills. The solid waste disposal problem has highlighted issues of scarce landfill space and has stirred up public interest in material biodegradability.

Due to environment pollution caused by non-biodegradable gloves, biodegradable gloves are extensively sought. Bio-based filler in NRL gloves such as starch-based materials have been proven to be a magnificent biodegradable source [4,5]. Consequently, the additive manufacturing part produced can be improved by optimizing the process parameters. The predicted models obtained show good correlation with the measured values and can be used to generalize prediction for process conditions outside to the industry. As its name, the purpose of optimisation is to achieve the "best" design relative to a set of prioritized the mechanical characteristics. In this study, the optimum condition mechanical properties of cassava peel starch composite NRL film by Wendy (2020) and drying parameters of biodegradable film by Pulungan (2020) was identified [6]-[7]. Methods employed to identify the conditions were Taguchi method and Response Surface Method (RSM) with a Central Composite Design (CCD) in order to obtain a durable biodegradable glove which is not only safe to environment, but also possesses high stress and high strain.

1.1 Taguchi

Taguchi is commonly known due to its practicality in designing high quality systems that provide much-reduced variance for experiments with an optimum setting of process control parameters. This technique has been widely used in different fields of engineering to optimize the process parameters, and to determine the impacts of different parameters within the combination of design parameters [8]. Thus, the integration of design of experiment with parametric optimization of process is achieved in the Taguchi method. An orthogonal array provides a set of well-balanced experiments and Taguchi's signal-to-noise (S/N) ratios, which are logarithmic functions of the desired output, serve as objective functions for optimization. It helps to learn the whole parameters space with a small number (minimum experimental runs) of experiments only. These are used to study the effects of control factors and noise factors, and to determine the best quality characteristics for particular applications [8].

In static problems, there three approaches can be used in utilising Taguchi method such as 'nominal the better', 'smaller the better' and 'larger the better' [8]. As stated in both equations 1 and 2 the 'smaller the better' and 'larger the better' approaches are respectively calculated the quality characteristics deviating from the desired value. The results from previous study will be used for DOE of Taguchi method.

$$S/N = -10\, log \frac{1}{n} \left(\sum_{i=1}^{n} y_i^2 \right) \tag{1}$$

$$S/N = 10\, log \frac{\mu^2}{\sigma^2} \left(\sum_{i=1}^{n} y_i^2 \right) \tag{2}$$

$$S/N = -10\, log_{10} \frac{1}{n} \left(\sum_{i=1}^{n} \frac{1}{y_i^2} \right) \tag{3}$$

Where,

n : replications/experiment trial number,

y : observed response value,

i : 1, 2, 3, …n

1.2 Response Surface Method

Process optimisation RSM using Design Expert software is made to find optimal for the relevant factors of a process to maximise the response. The significance of adapting the RSM for process optimisation is based on the availability of data generated from appropriate experimental design. It is a great tool for its varieties that able to place out and ideal experiment on the process, mixture of factors as well as components [9]. This software also offers a various option to select the type of graphs that able to assist the user to identify standout effects and for visualisation. The CCD was selected because of its efficiency with respect to the number of runs required [10] for fitting a second order response surface model. In the use of the CCD, an imbedded factorial or fractional factorial design with centre points that are augmented with a group of corner and star points allows for the estimation of curvature.

2. Materials and Methodology

Optimisation methods i.e. Taguchi and Response Surface Method (RSM) were used in order to obtain optimum values for each study. Factors and factor levels for both Pulungan (2020) and Wendy (2020) studies were constructed as shown in Table 1 and Table 2 respectively.

Table 1 - Factors and factor levels for Pulungan (2020) study

Levels	Factors	
	Drying temperature (^oC)	Drying time (hours)
0	40	5
1	50	6
2	60	7

Table 2 - Factors and factor levels for Wendy (2020) study

Levels	Factors
	Starch loading (%)
0	0
1	5
2	10
3	15
4	20
5	30
6	40
7	50

2.1 Taguchi Method

A general explanation regarding the optimisation has been introduced in previous chapter and the procedure is given in flowchart Fig. 1. The signal-to-ratio (S/N) ratio was used to investigate the optimum design of factors and factor levels. The signal is desirable of output characteristics. The noise represents the undesirable output characteristics. The SN ratio was calculated and Taguchi optimisation was done through Microsoft Excel. In this study, the larger the better approaches provide the better performance in mechanical strengths of the biodegradable composite films. Therefore, the “larger the better” was selected for the optimisation of parameters as described in Equation 3. In the case of swelling and weight loss percentage, the lower values give better performance. Hence, the “smaller the better” was selected as described in Equation 1. The “larger the better” approach is selected for this study it is because with higher optimised value for each parameters gives strong mechanical properties for NRL glove with cassava-peel starch loading. However, while high tensile strength is preferred, the “highest” number not indicating “best” performance. Hence, it is important to set a range of balance strength with stretch and comfort due to when the value of tensile strength is too high, it can lead to stiff and inflexible glove. Besides, high elongation of bio-based filler improves the glove material stretch instead of tear when snagged leaving comfort and great experience to the user.

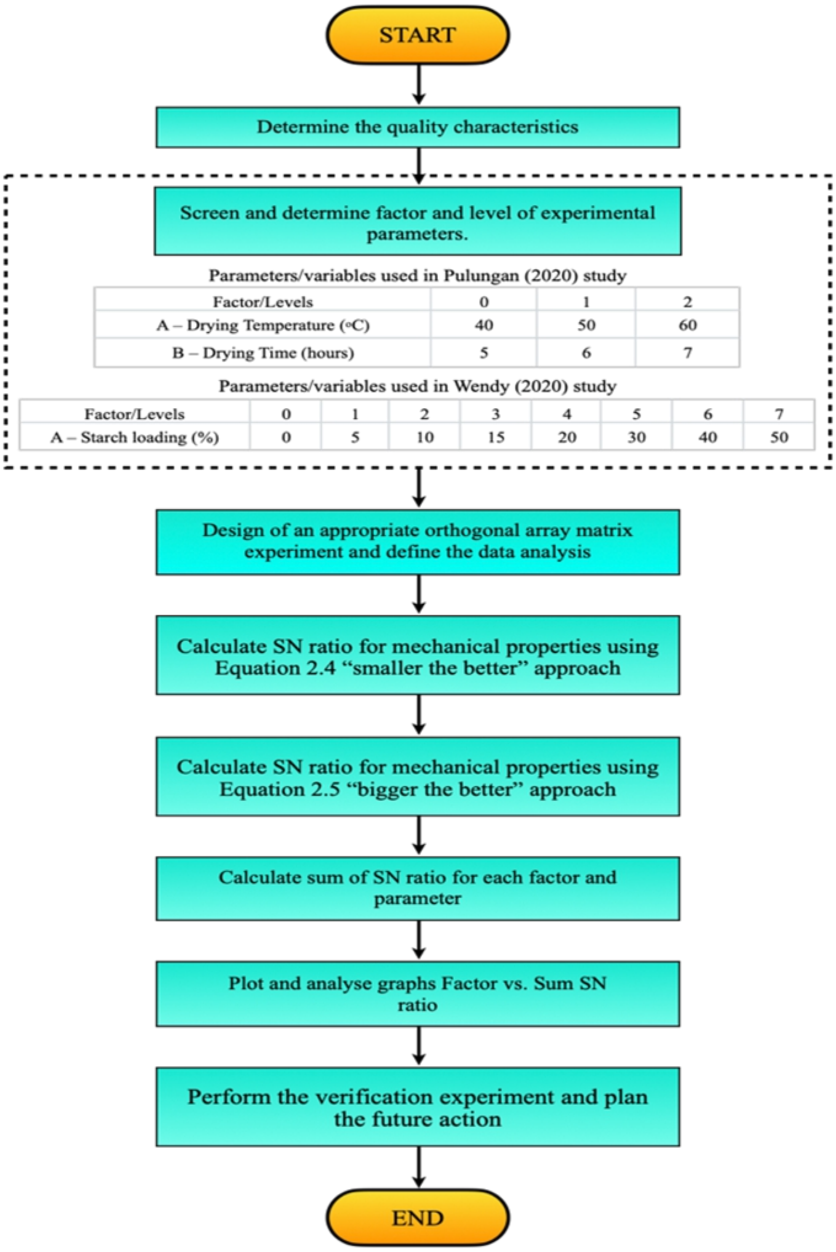

Fig. 1 - Flowchart of Taguchi method

2.2 Response Surface Method-Central Composite Design

RSM-Design Expert were chosen to discover ideal conditions for the relevant factors of a process to amplify maximise the response. It's a part of methodology to make measurable models depicting the relationship between factors and responses as a response-surface. In view of these models, it is easier to locate the necessary factor spaces that award the most conceivable yield for the procedure.

A Central Composite Design (CCD) in Response Surface Methodology (RSM) design was used and 13 of trials from the DOE have constructed in Table 1 and 2. For experiment done by Pulungan (2020) [6], the variables chosen were drying temperature and drying time and both were denoted as X1 and X2 respectively as shown in Table 3. The levels of drying temperature were 40°C. 50°C and 60°C while the drying time were 4, 5 and 6 hours. The data obtained from this study were operated using Design Expert 12 software to obtain the optimal value of biodegradable film from cassava peel starch. The responses measured were tensile stress, elongation and swelling. Design Expert 12 software was chosen to discover ideal conditions for the relevant factors of a process to amplify maximise the response. It's a part of methodology to make measurable models depicting the relationship between factors and responses as a response-surface. In view of these models, it is easier to locate the necessary factor spaces that award the most conceivable yield for the procedure.

Table 3 - Variables and responses design of experiment (DOE)

Variables		Responses		
X_1: Drying Temperature (°C)	X_2: Drying Time (hours)	Tensile stress N/m²	Elongation %	Swelling %
40	5			
50	6			
60	7			

3. Results and Discussions

Data from Pulungan (2020) [7] was studied to obtain the optimum value for the mechanical strengths of the biodegradable film with cassava-peel starch. The biodegradable film was prepared as described by Pulungan et al. which 40% of cassava peel starch were produced into starch suspension and included in a gel containing 2% (w/v) chitosan and 3% (w/v) glycerol. The mixture was mixed at 65°C and stirred for 10 minutes to form a biodegradable plastic film. For Wendy (2020) study [6], eight levels were selected for the factor chosen and the responses for the optimisation are the mechanical properties such as stress and strain at break and the weight loss. Meanwhile in Pulungan (2020) study, two factors and three levels were selected with responses of tensile stress, elongation and swelling.

3.1 Taguchi Method

In Pulungan (2020) study, the two factors i.e., drying temperature, drying time with three levels were used to optimise the mechanical properties of biodegradable composite film. A total nine experiments derived from L_9 (3^2) orthogonal array with parameters and their variation levels are shown as depicted in Table 4. For biodegradable film prepared by Wendy (2020), Taguchi and RSM were used to optimise tensile stress, elongation and swelling of the film. Compared to work done by Pulungan (2020), the biodegradable films were optimised based on only one factor i.e., starch loading, where the factor contained eight level quantitative measurements and resulted in eight orthogonal arrays (denoted as L_8), as shown in Table 5.

The results of the biodegradable films' mechanical properties and swelling in various conditions shown in Table 4. This study demonstrated that biodegradable film with the highest tensile stress (0.0039816MPa) was resulted from the treatment of drying temperature at 40°C and drying time of 6 hours. The lowest elongation was obtained from treatment of 60°C for 7 hours with value 2.5%. The lowest elongation value was possibly due to the uneven thickness of the film which affects the mechanical properties of the biodegradable film. The highest swelling with high elongation was obtained from the treatment of drying temperature at 60°C for 5 hours with the value of 201.6%. While the lowest swelling value was 131.3% resulted from treatment with drying temperature of 40°C and drying time of 5 hours.

Table 4 - Design of experiment of L_9 for biodegradable film by Pulungan (2020) at various conditions

Experimental Trial	Designation	Tensile Stress		Elongation	Swelling
		MPa	N/m²	%	%
1	A0B0	0.00184368	1843.68	7.50	131.3
2	A0B1	0.00214769	2147.69	8.75	193.3
3	A0B2	0.00337354	3373.54	7.50	171
4	A1B0	0.00184368	1843.68	7.50	198.4
5	A1B1	0.00184368	1843.68	7.50	201.6
6	A1B2	0.00061783	617.83	7.50	198.4
7	A2B0	0.00061783	617.83	2.50	201.6
8	A2B1	0.00398156	3981.56	16.25	172.6
9	A2B2	0.00245170	2451.70	10.00	166.7

Table 5 - Design of experiment of L_8 for biodegradable film by Wendy (2020) at various conditions

Experimental Trial	Designation	Stress at break MPa	Strain at break %	Weight loss %
1	A0	5.8830	993.48	3.01
2	A1	7.3570	938.00	4.15
3	A2	2.5601	952.64	8.49
4	A3	5.7984	911.47	7.48
5	A4	10.7320	1281.00	7.23
6	A5	8.2265	1236.10	14.53
7	A6	3.0876	829.97	19.93
8	A7	1.8633	722.30	17.24

3.1.1 Tensile Stress and Strain at Break

The graph below (Fig. 2) shows the sum of SN ratio for each drying parameters. The graph was plotted according to the table of SN ratio after sum-up from the factors. The highest value of SN ratio is at A1 (-156.34) and B1 (-161.62) which gives value 50°C and 6 hours respectively. This is because at certain high temperatures at longer period, the tensile stress of biodegradable films tends to increase and stronger compared to lower temperatures. However, if the temperature used and time taken is too long, it will affect the hydrogen binding between starch of cassava peels with chitosan eventually influence the tensile stress of the film according to Suderman et. al [11]. Hence, the optimum condition expressed by Taguchi method is A1B1 to achieve higher tensile stress of the cassava-peel starch biodegradable film.

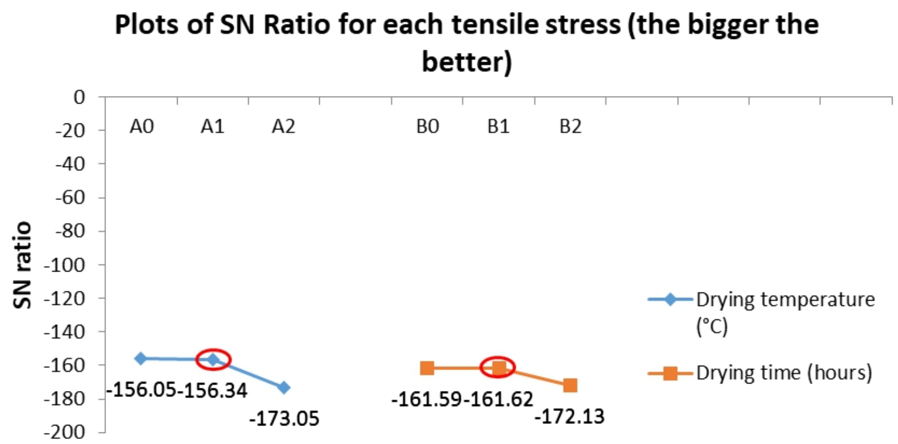

Fig. 2 - Plots of SN ratio for each Tensile stress (the bigger the better) (study by Pulungan, 2020)

From the Fig. 3 and 4, at factor A4 shows the steepest point for both stress and strain at break which is 20.61 and 62.15, respectively. The values represent the maximum tensile stress level of biodegradable composite film with 20% of cassava-peel starch filler loading. This is due to the existing of positive interaction between the rubber matrix and the filler particles therefore gives supplementary reinforcements. Compared to unfilled films, the sum of SN ratio of tensile stress is slightly lower than the films that incorporated with starch filler. In contrast, further increasing in filler loading does not strengthen the film as seen in both plotted graphs. This shows that high starch loading to NRL ratio is not suitable and desirable in intensifying the tensile stress. This condition also may cause by the strong polarising nature of starch fillers conflict with the hydrophobic natural rubber polymer. For instance, when the cassava-peel starch loading increased, the dispersion of starch in the NRL became difficult and the starch particle will tend to aggregate with one and by the formation of strong hydrogen bonding to form gelatinized starch [4]. Thus, poor interaction occurs between starch filler and NRL film.

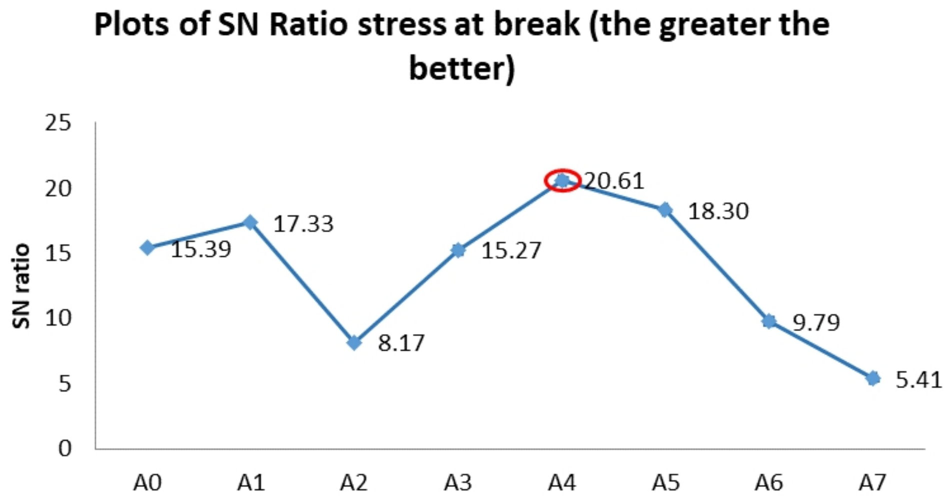

Fig. 3 - Plots of SN ratio stress at break (the bigger the better) (study by Wendy, 2020)

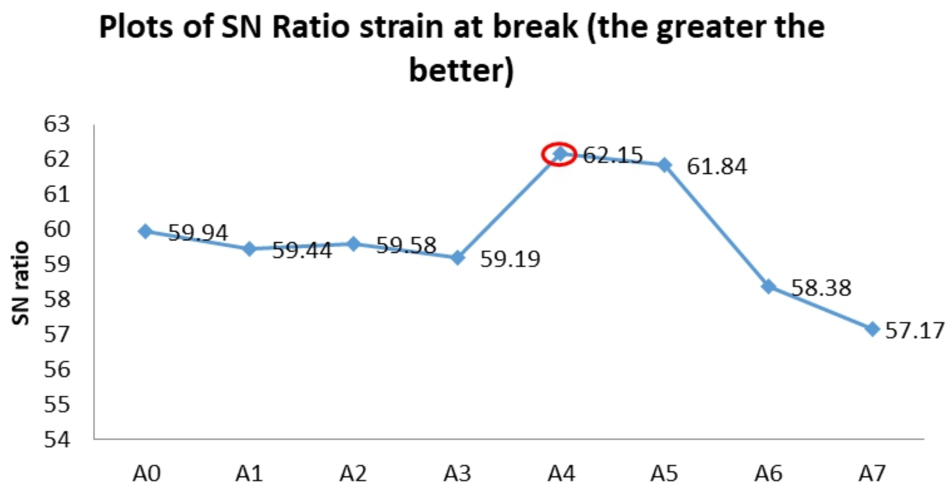

Fig. 4 - Plots of SN ratio strain at break (the bigger the better) (study by Wendy, 2020)

3.1.2 Elongation

The approach used in Taguchi method for elongation is ‘the bigger the better’ and the sum of SN ratio values from L_9 orthogonal arrays table were tabulated in Table 3. These values were plotted into graph as seen in Fig. 5, this is to recognize the optimum condition at each factor.

The graph in Fig. 4 shows the sum of SN ratio for each drying parameter. The graph was plotted as stated by the table of SN ratio after sum-up from the factors. The highest value of SN ratio is at A0 with value of 60.56 and B1 at 59.22. In which gives value 40°C and 6 hours respectively. Elongation at break measures on how much bending and shaping a material can withstand without breaking. The measured elongation at break is important in components that absorb energy by polymer deformation. In this study, the elongation at break increases with an increase temperature and decreases with an increase in the filler content of the composite. This is because at high temperature the water contains or moisture of the biodegradable film tends to evaporate eventually solidified and homogenise the texture particles. Thus, the most favourable condition expressed by Taguchi method is A0B1 to achieve the greatest elongation at break of the cassava-peel starch biodegradable film.

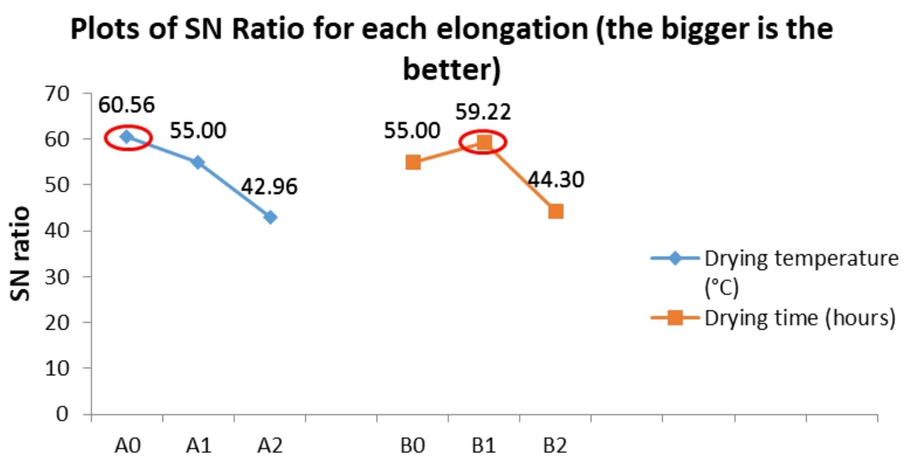

Fig. 5 - Plots of SN ratio for each Elongation (the bigger the better) (study by Pulungan, 2020)

3.1.3 Swelling and Weight Loss

Swelling is an increase in the volume of the film connected with the uptake a liquid or gas. The absorption of liquids causes changes in the mechanical properties of the swollen material and perhaps create extra pressure when it occurs in limited spaces which results various deformations of the swollen material. For instance, the swell can be defined as surface creases and wrinkles. From the graph below (Fig. 6), the swelling SN ratio stated the optimum condition is at A1B0 which represents 50°C and 6 hours respectively. The drying process influence the water content of the biofilm and causes evaporation of the moisture that are more hydrophilic in nature. To such great extent, when the biodegradable film was treated with high humidity media it will cause the film to greatly absorb water in order to achieve equilibrium. From the value achieved from Pulungan (2020) [7], the swelling is smaller due to the concentration of chitosan which is hydrophobic is bigger influence the process of water absorption. This condition can be applied to study done by Wendy (2020) [6] where the natural rubber acts as hydrophilic and cassava-peel starch as hydrophilic properties.

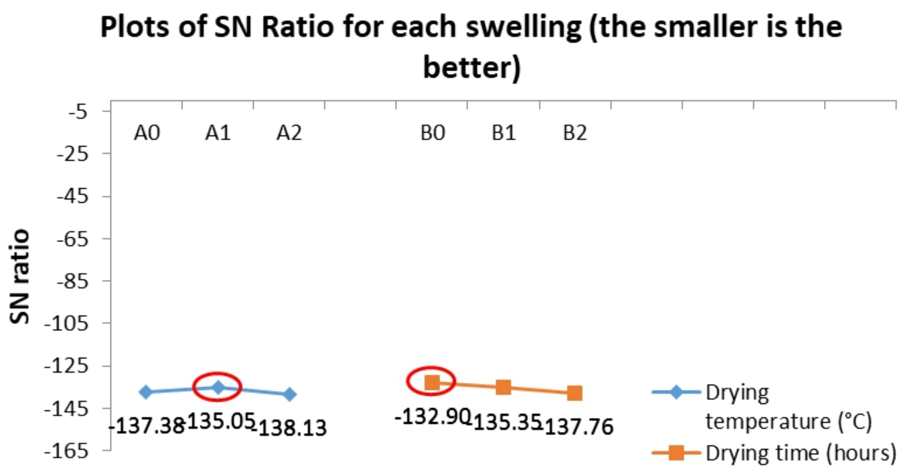

Fig. 6 - Plots of SN ratio for each Swelling (the bigger the better) (study by Pulungan, 2020)

From the graph plotted (Fig. 7), A6 with the highest sum of SN ratio 25.99 shows the increasing activity of microorganism metabolism compared to A4 with 17.18 SN ratio. This decreasing of weight loss may be due to lower cassava-peel starch filler loading resulting low and limited microorganism activity. The starch filler is known to have hydrophilic properties that will attract and absorb water in the soil. The incorporation of starch into NRL film directly enhance the action of hydrolysis, depolymerisation of NRL film and microbial attack [12].

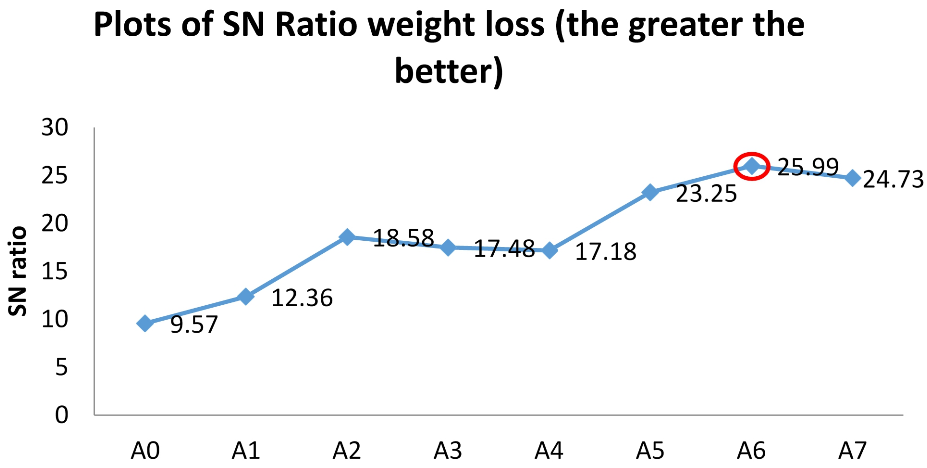

Fig. 7 - Plots of SN ratio weight loss (the bigger the better) (study by Wendy, 2020)

3.2 Central Composite Design via Design Expert 12 Software

Design-Expert 12 is the software used for this study to find the optimum drying condition for each response inserted. For this optimisation method, Wendy's study unable to be optimised due to lack of experimental data since the design of experiment is not compatible with the method.

The results of the biodegradable films mechanical properties and swelling percentage in various conditions can be seen in 3D plotted graphs (see Fig. 8, 9 and 10). In all three, the outline of the contour figure shows the highest value and the deeper shows a decrease in the responses' value. This study displayed that biodegradable film with the highest tensile stress (3981.56 N/m2) was resulted from the treatment of drying temperature at 60°C and drying time of 5 hours. Similar result was obtained for the treatment with 50°C drying temperature and 4.59 hours drying time. However, when using drying temperature of 50°C for 6 hours, the resulted biodegradable film has the lowest tensile stress of 617.83 N/m2. The similar trends were also observed for elongation parameters. Where the highest elongation value was resulted from the treatment of 60°C for 5 hours or 50°C for 4.59 hours, giving the value of 16.25%.

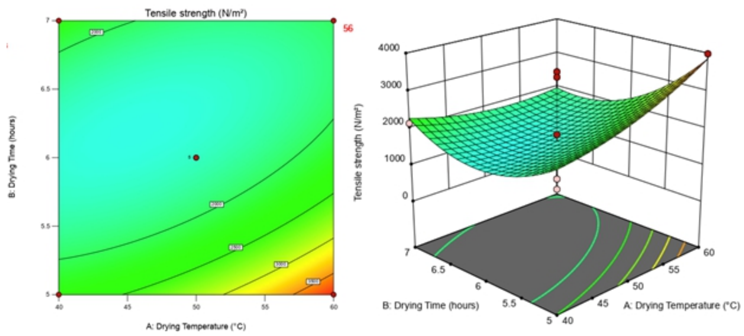

Fig. 8 - Response surface contour (a) and three-dimensional plots; (b) biodegradable film tensile stress

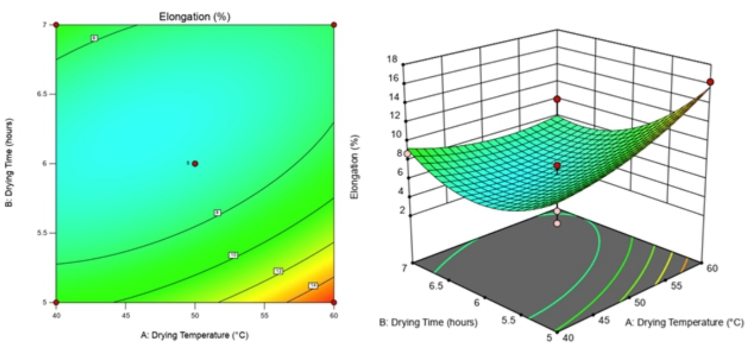

Fig. 9 - Response surface contour (a) and three-dimensional plots; (b) biodegradable elongation

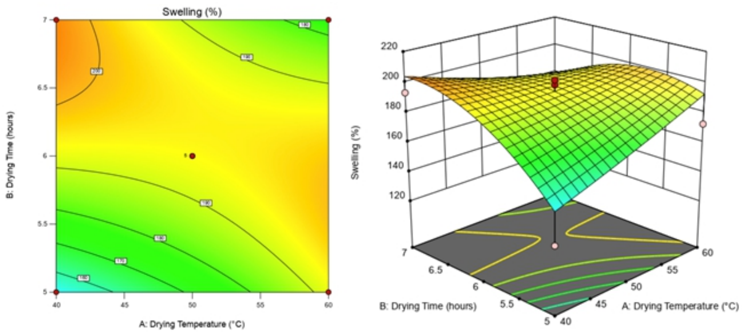

Fig. 10 - Response surface contour (a) and three-dimensional plots; (b) biodegradable swelling percentage

The lowest elongation was again obtained from treatment of 50°C for 6 hours with the value of 2.5%. The lowest elongation value was possibly due to the uneven thickness of the film which affects the mechanical properties of the biodegradable film. The highest swelling was obtained from the treatment of drying temperature at 64.14°C for 6 hours, with the value of 215.1%. While the lowest swelling value was 131.3% resulted from treatment with drying temperature of 40°C and drying time of 5 hours. This result indicates that the higher the drying temperature and the longer the drying time can increase the swelling value of the biodegradable film. This result was in accordance with Suderman et al. [11] that drying temperature and time have impact on the mechanical properties of the biodegradable film. Results obtained from response surface central composite design showed the optimum values for each output responses as seen in Table 6.

Table 6 - Optimum values for each responses RSM

Factors	Output responses		
	Tensile stress (MPa)	**Elongation (%)**	**Swelling (%)**
	0.00398156	**16.25**	**131.3**
A: Drying Temperature (°C)	60	60	40
B: Drying Time (hours)	5	5	5

4. Conclusion

The enhancement of mechanical properties could be attributed to the gelatinisation of starch in cases of higher drying temperatures. In this study, the optimum conditions of mechanical properties of biodegradable film made of 40% cassava peel starch and composite NRL-cassava peel starch were studied. Based on Wendy (2020) study stated the starch loading that has excellent mechanical and biodegradable properties is 20%. For Pulungan (2020) study, it can be concluded that the tensile stress of cassava-peel starch biodegradable film has the best condition at 50°C to 60°C at approximately 5.5 hours. Elongation optimum conditions shows contrast value of temperature and time. Meanwhile, for Wendy (2020) study, it shows the best percentage loading of cassava-peel starch is at 20% to achieve high stress and strain at break. In contrast for weight loss response which has the highest percentage (40% of starch loading) this is due to the consumption of starch by microorganisms in the favoured soil environment and hence causing increase in weight loss of composite NRL-cassava-peel starch film. From Wendy (2020) study, it can be perceived that at starch loading of 40% and above causing decreasing of tensile stress which can be explained by the imperfect distribution of the filler through the polymer matrix as well as very poor adhesion between the matrix and filler.

There are several recommendations and improvements can be incorporated into this research for future study on biodegradable composite NRL. First and foremost, it is important to form design of experiment before carried out the experiment and data collection. This is to ease the optimisation using other optimisation techniques or software for example Minitab and analysis such as ANOVA. Besides, the verification and validation test from the optimised data should be performed to prove the conditions from both Taguchi and RSM method.

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