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ORIGINAL ARTICLE

Blending Process of Cellulose Nanofiber/Polyvinyl Alcohol (NFC/PVA) For Paper Coating Application

N. M. Sudri[1], Aniek Sri Handayani[2,*], Ratnawati[2], A. A. Septevani[3], M. E. Puspita[4], Y. A. Husnil[2], Y. Arifin[5], A. N. Syabila[2], and M. R. Z. Qois[1]

[1] Industrial Engineering Study Program, Institut Teknologi Indonesia, Indonesia
[2] Chemical Engineering Study Program, Institut Teknologi Indonesia, Indonesia
[3] Badan Riset dan Inovasi Nasional (BRIN), Indonesia
[4] Management Study Program, Institut Teknologi Indonesia, Indonesia
[5] PT Bukit Muria Jaya, Indonesia

ABSTRACT – This paper describes how to make films using polyvinyl alcohol (PVA) and nanofiber cellulose (NFC). In this study, different NFC and PVA ratios were varied. To achieve ideal condition during the film making process, several characteristics, including as density and viscosity, have been investigated using Taguchi method. The results showed two main outcomes. To obtain optimal viscosity, the NFC/PVA ratio factor is 5/95, the volume/volume ratio is 10%, and the sonication time is 7 minutes. For optimal density, several conditions can be used, including a NFC/PVA ratio of 10/90, volume/volume ratio of 3%, and a sonication time of 5 minutes. The experimental results confirmed the average viscosity response of 129.99 ± 13.33 and the S/N ratio of -42.64 ± 0.56. The experimental results of the Taguchi method were -42.64 ± 0.56 and the S/N ratio was -42.92 ± 0.67. Meanwhile, the response data for the Taguchi methodal density averaged at 1.042 ± 0.013 and the S/N ratio was -0.365 ± 0.104. For the results of the experimental condition were 1.126 ± 0.015 and the S/N ratio of -1.031 ± 0.124.From these data, it can be concluded that there is an increase in the average and variability which indicates that the blending process is getting better with the use of NFC.

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INTRODUCTION

The government's commitment through the Coordinating Ministry for Maritime Affairs and Investment of the Republic of Indonesia in the 2030 Sustainable Roadmap is getting a target for reducing 70% of plastic waste in 2025 and reaching net zero in 2040. In 2021, Indonesian Statistic Bureau (BPS) reports that Indonesia produces 66 million tons of plastic waste and 9 out of 10 garbage found in the oceans is food and beverage packaging waste. Packaging has a 5–10% of product value and generates 40% of household waste. The problem is then created. The use of food packaging made from plastic makes issues in waste that are difficult to degrade. An 80% of plastic ends up in the soil and causes environmental problems. The challenge is raising the use of environmentally friendly materials in packaging which has characteristics like fossil packaging. However, the use of that materials has a limitation in the mechanical and barrier properties. The use of biopolymers as a food packaging material is constrained by costs and low characteristics and cannot be compared with materials from fossils.

Nanocellulose has been developed as a cheap and environmentally friendly material which has been tested and proven to be used as a gas and moisture barrier. Due to the merits possessed by cellulose, it increases the use of this type of material, including its effectiveness in the specific surface area and strong hydrogen bonds. Thus, this makes it difficult for cellulose to be passed by other molecules. This property makes cellulose a potential barrier to gas and water vapor in packaging. Nanocellulose can be classified as Nanofiber cellulose (NFC) or Nanocrystal Cellulose (NCC). NFC has diameter particle between 20 and 50nm and length between 500 and 2000nm, while NCC has short stick-shaped crystalline nanoparticles with a diameter ranging from 5 to 50 nm and a length between 100 and 500 nm [1]. Nanocrystal cellulose (NCC) has high crystallinity, causing the density on the surface of the paper to be small. Nevertheless, NCC still has weaknesses in surface bonding with paper. The strength of paper depends mainly on the strength of the fibers themselves, fiber length, fiber orientation distribution, contact area between fibers, and the strength and number of hydrogen bonds in the matrix. NFC generally achieves its function for the strength enhancement of paper by increasing the bonding area of the sheet [2]. Nano fibrillated cellulose (NFC), commonly obtained by mechanical treatment, presents an entangled network structure with flexible, longer, and wide nanofibers [3] Hence, another natural polymer matrix is required to cover these weaknesses, such as polyvinyl alcohol (PVA). PVA is a natural polymer derived from environmentally friendly vinyl acetate. PVA is an ideal polymer to be composed/blended with NFC as their hydrophilic nature due to the presence of OH group and PVA can form hydrogen bonds with NFC due to the presence of (–OH)/alcoholic group [4].

*CORRESPONDING AUTHOR | Aniek Sri Handayani | ✉ aniek.handayani@iti.ac.id

NFC/PVA film is a product created as a paper coating material or as a liquid coater to be applied to paper media (paper coating) [1]. In the blending process in producing NFC/PVA solutions, which will later be used as paper coatings or as a coater liquid to be applied to paper surfaces, manufacturers certainly expect to reach efficiencies in the NFC or PVA usage as a raw material. To achieve efficiency in the use of these raw materials, the viscosity and density levels must be as minimal as possible yet still fulfill the requirements. This can be done by looking for several factors which affect the viscosity and density. In previous study conducted by Ratnawati et al., the viscosity of NFC/PVA nanocomposite was 32.55cp for PVA 10% and NFC 2% [5]. NFC/PVA film is a product created as a paper coating material or as a liquid coater to be applied to paper media (paper coating) [1]. In the blending process in producing NFC/PVA solutions, which will later be used as paper coatings or as a coater liquid to be applied to paper surfaces, manufacturers certainly expect to reach efficiencies in the NFC or PVA usage as a raw material. To achieve efficiency in the use of these raw materials, the viscosity and density levels must be as minimal as possible yet still fulfill the requirements. This can be done by looking for several factors which affect the viscosity and density [6]. Thus, the blending process can be more optimal according to the optimum viscosity and density, including with the experiment using the Taguchi method [7].

This study is to utilize the Taguchi method and varied several controlling parameters to discover the dominating factors as well as determine the optimal combination of the parameters to achieve optimum results supported by experimental and statistical analyses [8].

EXPERIMENTAL METHOD

Materials and Instruments

In producing NFC/PVA film, natural raw materials were used, such as microfiber cellulose (MFC) from empty palm oil bunches [9]. MFC was converted into NFC with physical treatment. The other ingredients were polyvinyl alcohol (PVA) and ultrapure water. The equipment used for producing this product included IKA T25 Digital Ultra Turrax, IKA C-MAG HS7 Hotplate Stirrer, Hielscher UP400St, HAAKE Viscosimeter 6 plus, pycnometer, thermometer, stopwatch, and coating tools.

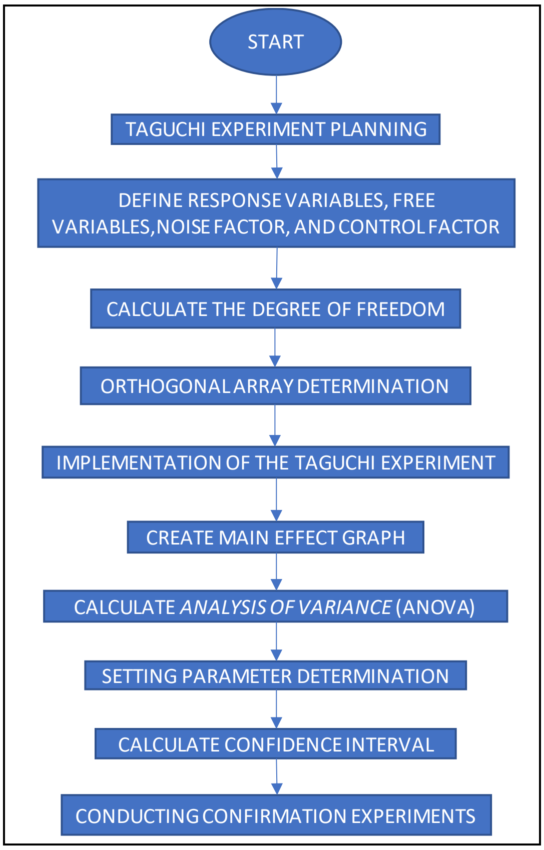

Figure 1. Design of experiment Taguchi method flow chart

Method and Procedure

Design of Experiment (DOE) is a test which is widely used to design an experiment to find out the response and characteristics of a factor to the tester's variables. The number of tests to be performed is optimized by DOE, in addition to establishes a connection between independent variables and the empirical model and, in the end, yields the experimental data's optimum responses [10]. The experimental design of the Taguchi method is a method in which quality is measured based on the deviation of the characteristic against its target value [11]. The Taguchi method is a technique used by the engineer for improving productivity during the development stage. Thus, the quality of the product or experiment can be produced rapidly at an optimal cost [12]. In this study, Taguchi method is used in the blending process. The blending process (using IKA C-MAG HS7 Hotplate Stirrer tool) is one of the stages in the manufacture of NFC Film products, where PVA and NFC solutions were mixed to become a coater liquid. This experimental design was carried out to optimize the level of viscosity and density (viscosity measurement using the HAAKE Viscosimeter 6 plus tool and density measurement using a pycnometer).

The process flow in this study is represented in Figure 1. The initial stages, include determining the response variables, noise factors, and control factors, then calculating the degrees of freedom, and choosing an orthogonal matrix. After the initial stage, experiments with the Taguchi method were carried out through a blending process between NFC/PVA. The experimental data (viscosity and density data) were processed statistically, in the form of average values, S/N ratios, main effects, and analysis of variance (ANOVA). ANOVA is used when obtaining the result of the F-ratio value of each factor. It is necessary to test the hypothesis to determine whether these factors affect the viscosity and density or not [13]. In the ANOVA calculation, if F counts are greater than the F table, then the independent variable affects the control variable. However, if the F count is smaller than the F table, the independent variable has no significant influence on the control variable. The final stage is to calculate the percentage contribution for determining the optimal of parameter settings, confidence intervals, and the test confirmation stage.

In this study, the response variables were viscosity and density in the NFC/PVA solution. For the noise factor, it is necessary to carry out a compositional analysis to find out whether there are metal elements or other materials. Table 1 is the setting of the current conditions used in the blending process for the making NFC/PVA solutions.

Table 1. Control factors of the initial conditions of the blending process

Parameter Code	**Factor**	***Settings***
A	$\frac{NFC}{PVA}$	$\frac{5}{95}$
B	$\frac{V_{NFC}}{V_{PVA}}$	3%
C	Temperature	50°C
D	Sonication time	5 minutes

To determine variations in level settings, the NFC/PVA ratio, volume/volume ratio, and sonication time were determined, while the temperature was not varied and was set at 50°C (see Table 2) [14].

Table 2. *Setting* the Taguchi parameter of the *blending* stage

Parameter code	**Factor**	**Level 1**	**Level 2**	**Level 3**
A	$\frac{NFC}{PVA}$	$\frac{20}{80}$	$\frac{5}{95}$	$\frac{10}{90}$
B	$\frac{V_{NFC}}{V_{PVA}}$	3%	5%	10%
C	Sonication Time	5 minutes	7 minutes	10 minutes

The degrees of freedom are calculated to determine the orthogonal array as a determinant of the number of experimental designs [15]. In this study, 6 degrees of freedom are tested using the formula V_{fl} = (number of levels–1). Based on the results of the degree of freedom, the orthogonal array chosen was L_9 with $L_9(3^4)$ [13]. To create orthogonal array $L_9(3^4)$ use Minitab 19 software. The selection of the orthogonal array is presented in Table 3.

Table 3. Orthogonal arrays

Experiment Number	**Factor**		
	A $\frac{NFC}{PVA}$	**B** $\frac{V_{NFC}}{V_{PVA}}$	**C Sonication time**
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

The next step is calculating the confidence interval. Which serves to find out the highest and lowest values where the average value and the S/N ratio are correct to cover a certain percentage of confidence. The calculation of the confidence interval on the mean value and the S/N ratio for the viscosity and density response data is presented as follows. The variables used are [16]:

(1) Calculating Predictions. The calculations are in the following:
   (i) Predicted average value for the viscosity response data: $\bar{y} + (\overline{A2} - \bar{y})$ + - + - $(\overline{B3}\bar{y})(\overline{C2}\bar{y})$ (1)
   (ii) Predictability values for viscosity response data: $\bar{y} + (\overline{A2} - \bar{y})$ + - + - $(\overline{B3}\bar{y})(\overline{C2}\bar{y})$ (2)
   (iii) Predicted average values for density response data: $\bar{y} + (\overline{A3} - \bar{y})$ + - + - $(\overline{B1}\bar{y})(\overline{C1}\bar{y})$ (3)
   (iv) Predicted variability values for density response data: $\bar{y} + (\overline{A3} - \bar{y})$ + - + - $(\overline{B1}\bar{y})(\overline{C1}\bar{y})$ (4)

(2) Calculate the Prediction of Confidence Intervals using the following Equations and Variables:
   (i) N_{eff} is the Number of effective observations.
   (ii) DoF is the Degree of Freedom, calculated using $N_{eff} = \frac{\text{number of experiment}}{1 + \text{number of DoF}}$ (5)
   (iii) CI is the Confidence Interval
   (iv) MSe is the Mean Squared Error, calculated using $CI = \sqrt{F(0{,}05; 2; 6) \times MSe \times \frac{1}{Neff}}$ (6)

RESULTS AND DISCUSSION

In the first step, the NFC/PVA blending experimental data was already obtained from the experiments which have been carried out. In this experiment, only 1 replication is used due to raw material limitations. The data will be averaged and calculated the S/N Ratio. Signal-to-noise ratios is the key technologies utilized in resilient design. A set of well-balanced tests using the signal to noise ratio, which is a log function of the desired output and emphasizes variation in quality measurements, allows for simultaneous consideration of numerous design parameters [17].Following the orthogonal array of 9 experiments, the implementation of the Taguchi method was carried out according to the combination of the predetermined level. Data from Taguchi method were processed to calculate the mean and the S/N ratio. The following is a calculation table for the viscosity and density response data presented in Table 4 and Table 5.

The S/N Ratio formula used S/N *Ratio* = -10 log $(\frac{1}{n}\sum_{i=1}^{n} y_i^2)$. (7)

Table 4. Recapitulation of mean value calculation and S/N ratio value for viscosity response data

Experiment Number	Viscosity	Mean	S/N ratio
1	330	330	-50.37
2	310	310	-49.83
3	340	340	-50.63
4	160	160	-44.08
5	160	160	-44.08
6	140	140	-42.92
7	230	230	-47.23
8	200	200	-46.02
9	170	170	-44.61
Sum		2040	-419.78
Average		226.67	-46.64

The average result from the viscosity response data according to the calculation was 226.67, while the S/N ratio result was -46.64. The average result from the density response data, from 9 experiments according to the calculation, was 1.058, while for the mean of S/N ratio result, was -0.486. Furthermore, the main effect curve based on the mean value is created and presented in Table 4. For the viscosity and density response, data are presented in Figure 1 and Figure 2. The main effect curve serves to determine the magnitude of the influence of each level from each factor on the response variable. The two main effect curves can be tabulated into the response table, as shown in Table 6 and Table 7.

Table 5. Recapitulation of mean value calculation and S/N ratio value for density response data

Experiment Number	Density	Mean	S/N ratio
1	1.044	1.044	-0.374
2	1.066	1.066	-0.555
3	1.068	1.068	-0.571
4	1.074	1.074	-0.62
5	1.056	1.056	-0.473
6	1.054	1.054	-0.457
7	1.052	1.052	-0.44
8	1.05	1.05	-0.424
9	1.054	1.054	-0.457
Sum		9.518	-4.372
Average		1.058	-0.486

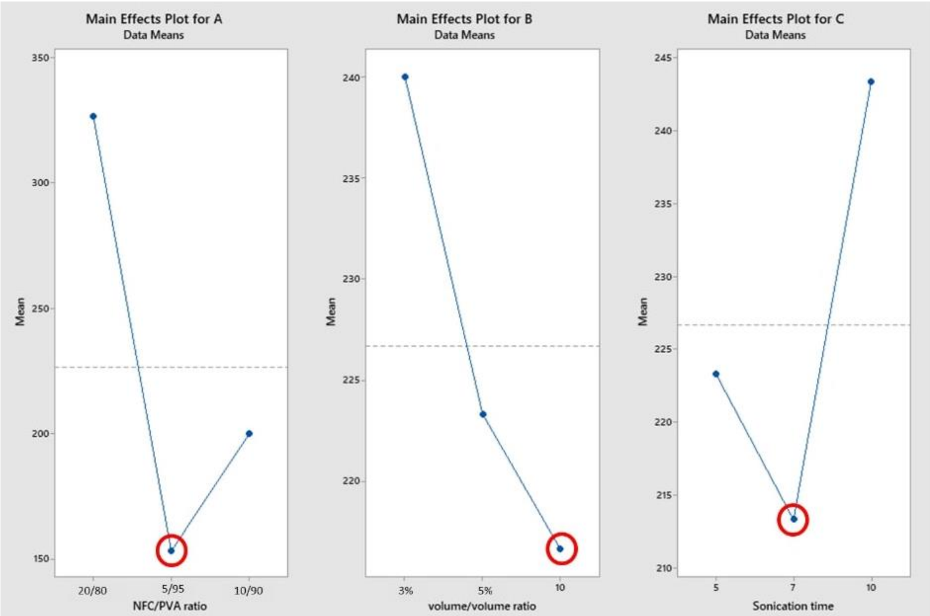

Figure 2. Main effect graph for the average values of viscosity

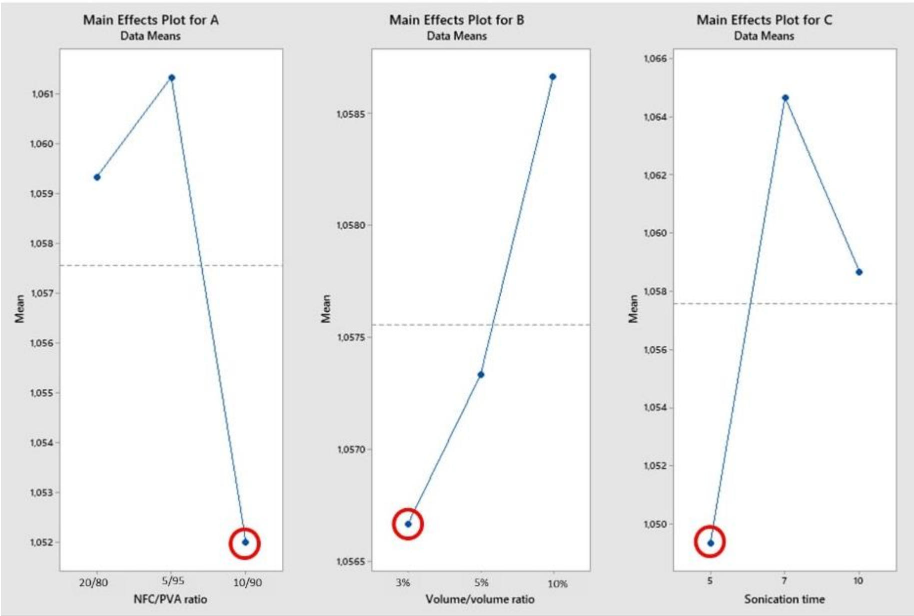

Figure 3. Main effect graph for the average value of density

Table 6. The average response of the viscosity of the NFC/PVA solution to the factor

	Mean		
	A	**B**	**C**
Level 1	326.67	240.00	223.33
Level 2	**153.33**	223.33	**213.33**
Level 3	200.00	**216.67**	243.33

Based on the Table 6, this experiment refers to the use of smaller is achieving better results. The reason for using the smaller characteristics is to save NFC raw materials. The success in obtaining the minimum viscosity and density values will less the use of NFC raw materials. It leads to choose the smallest average viscosity level of each factor. The optimal rate of each factor is the point that has the smallest value [18], [19]. The red circle on Figure 2 and Figure 3 show the chosen minimum point since it is the smallest value of each factor. For the optimal level viscosity response data, several points are obtained:

(i) factor A is level 2, which is 153.33.
(ii) factor B is level 3, which is 216.67.
(iii) factor C is level 2, which is 213.33.

Data in Table 6 will be used to carry out confirmation experiments and obtain the lowest viscosity value. If a combination of each level of factors is used, it will be able to reduce the viscosity level.

Table 7. The average response of NFC/PVA solution density to factors

	Mean		
	A	**B**	**C**
Level 1	1.0593	**1.0567**	**1.0493**
Level 2	1.0613	1.0573	1.0647
Level 3	**1.0520**	1.0587	1.0587

Based on Table 7, this experiment refers to smaller is better, which leads in choosing the smallest average density level of each factor. The optimal rate of each factor is the point that has the smallest value [20]. The red circle on the Figure 4 and Figure 5 show the chosen minimum point since it is the smallest value of each factor. For the density response data, the optimal level for factor A is level 3, which is 1.0520. For factor B, the optimal level is 1, which is 1.0567. For factor C, it is level 1, which is 1.0493. In addition, the chosen data in Table 7 will be used to confirm the experiments to obtain the lowest density value. If a combination of each level of factors is used as in the Table 7, it is predicted that it will be able to reduce the density level.

For the main effect graphs made based on the data in Table 5 for viscosity response data, the main effect graphs based on the S / N ratio value can be presented in Figure 3 and Figure 4 as follows.

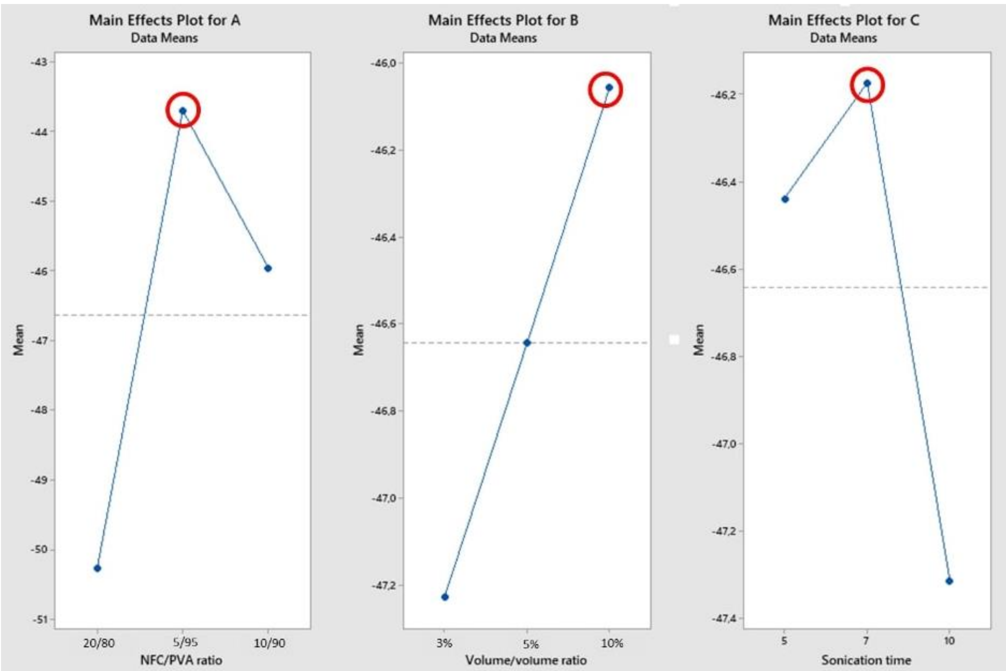

Figure 4. Main effect graph for S/N ratio values of viscosity data

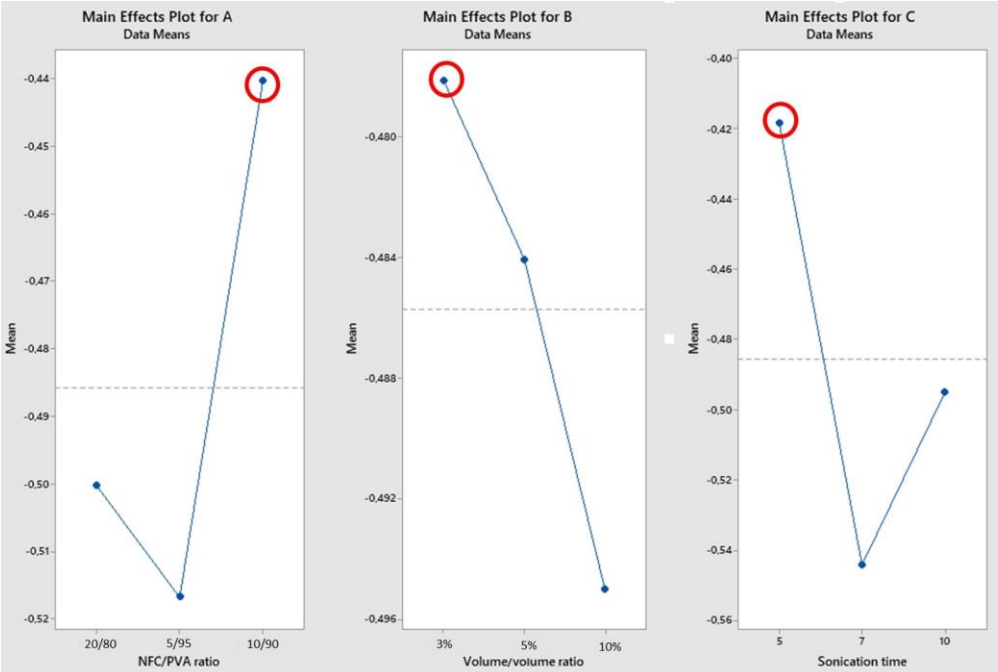

Figure 5. Main effect graph for S/N ratio value density data

The two main effect graphs on Figure 5 and Figure 6 can be tabulated into the table, as can be depicted in Table 8 and Table 9 below.

Table 8. The S/N ratio of the viscosity of the NFC/PVA solution to the factor

	S/N Ratio		
	A	**B**	**C**
Level 1	-50.28	-47.23	-46.44
Level 2	**-43.70**	-46.64	**-46.17**
Level 3	-45.95	**-46.05**	-47.32

Based on Table 8, the results of the influence of factors and levels used in the experiment can be obtained on the variability of the density of the NFC/PVA solution. This experiment refers to smaller is better which means it is getting lower, while the point that occupies the highest position on the graph in Figure 4 and Figure 5 [21] is the lowest value of the main effect S/N ratio and also the chosen point. The selection of this point is to select the minimum level of each factor. The optimal viscosity level response data at factor A is level 2 which is -43.70, at factor B is level 3 which is -46.05, and at factor C is level 2 which is -46.17. Furthermore, data in bold will be used to confirm the experiments to obtain the lowest viscosity value. If a combination of each level of factors is used as in the table above, it is predicted that it will be able to reduce the viscosity level.

Table 9. Response of S/N ratio density of NFC/PVA solution to factor

	S/N Ratio		
	A	**B**	**C**
Level 1	-0.5002	**-0.4781**	**-0.4182**
Level 2	-0.5167	-0.4841	-0.5440
Level 3	**-0.4403**	-0.4950	-0.4950

Based on Table 9, is the lowest value of the main effect S/N ratio and is also the chosen point. The selection of this point is to select the minimum level of each factor [22] For the density response data, the optimal level for factor A is level 3 which is -0.4403, for factor B is level 1 which is -0.4781, and for factor C is level 1 which is -0.4182. Next, data in bold will be used to confirm the experiments to obtain the lowest density value. If a combination of each level of factors is used as in the table above, it is predicted that it will be able to reduce the density level [23].

Analysis of Variance (ANOVA)

After understanding the level combination of factors, the data were processed using the ANOVA. Analysis of ANOVA is a multivariate analysis technique which serves to distinguish the average of more than two groups of data by comparing their variance [20]. The ANOVA calculation functions to analyse the contribution between variables.

Thus, the accuracy of the model estimates can be determined. Furthermore, after conducting ANOVA, hypothesis testing was carried out to prove that there was a significant influence of all factors on the observed response variables. The following is the processing of data with ANOVA against the average value for viscosity and density response data. The data used for processing ANOVA analysis comes from Table 4 and Table 5 and the results were presented in Table 10 and Table 11.

Table 10. ANOVA results for mean values in viscosity response data

Source	DS	SS	MS	F-ratio
A	2	48267	24133	543
B	2	867	433	9.75
C	2	1400	700	15.75
Error	6	267	44.4	
Entire	12	50800		

Table 11. ANOVA results for mean value in density response data

Source	DF	SS	MS	F-ratio
A	2	0.000145	0.000072	1.804
B	2	0.000006	0.000003	0.077
C	2	0.000358	0.000179	4.461
Error	6	0.000241	0.000040	
Entire	12	0.000750		

Before the hypothesis test was carried out, it is necessary to understand the F table and compared it with the calculated F-ratio. Based on the definition, H_0 = Factor which has no effect on the response variable. Meanwhile, H_A = Factors influencing the response variable. According to the F test, if F < F in the table (V_1, V_2) is calculated, H_0 is accepted and the H_A is rejected. On the other hand, if F > F table (V_1, V_2) is taken, H_0 is rejected and H_A is accepted.

In this study, the assumption of the alpha (α) value of 5% (or α = 0.05) was used. This calculation receives only 5% of the possible error. V_1 had 2 of the free variable value minus one and V_2 had 6 since *the error* was calculated with 6 degrees of freedom [24]. Therefore, the F table is 5.14. The comparison of the F-ratio with the F table and hypothesis test results is in the following:

(i) Factor A: F-ratio (5.43) > 5.14. Thus, H_0 is rejected, and H_A is accepted.
(ii) Factor B: F-ratio (9.75) > 5.14. Thus, H_0 is rejected, and H_A is accepted.
(iii) Factor C: F-ratio (15.75) > 5.14. Thus, H_0 is rejected, and H_A is accepted.

From the above calculation, it can be concluded that factors A, B, and C have a significant effect on the percentage of viscosity of NFC/PVA solutions. For the density response data, the comparison of F-ratios with the F table and hypothesis test results are as follows.

(i) Factor A: F-ratio (1.804) < 5.14. Thus, H_0 is accepted, and H_A is rejected.
(ii) Factor B: F-ratio (0.077) < 5.14. Thus, H_0 is accepted, and H_A is rejected.
(iii) Factor C: F-ratio (4.461) < 5.14. Thus, H_0 is accepted, and H_A is rejected.

By the above results, it can be stated that factors A, B, and C have no significant effect on the percentage of the density of NFC/PVA solutions.

Table 12. ANOVA results for S/N ratio variant value in viscosity response data

Source	DS	SS	MS	F-ratio
A	2	67.07	33.53	430.83
B	2	2.07	1.04	13.31
C	2	2.15	1.07	13.79
Error	6	0.47	0.08	
Entire	12	71.75		

Table 13. ANOVA results for S/N ratio variant values in density response data

Source	DS	SS	MS	F-ratio
A	2	0.0097	0.0049	1.802
B	2	0.0004	0.0002	0.082
C	2	0.0241	0.0121	4.481
Error	6	0.0162	0.0027	
Entire	12	0.0504		

The following is the data processing with ANOVA on the variant value of the S/N ratio for viscosity and density response data. The data used for processing using ANOVA comes from Table 4 and Table 5 and the results were presented in Table 12 and Table 13.

The comparison of the F-ratio with the F table and hypothesis test results for viscosity response data is in the following:

(i) Factor A: F-ratio (430.83) > 5.14. Thus, H_0 is rejected, and H_A is accepted.
(ii) Factor B: F-ratio (13.31) > 5.14. Thus, H_0 is rejected, and H_A is accepted.
(iii) Factor C: F-ratio (13.79) > 5.14. Thus, H_0 is rejected, and H_A is accepted.

Thus, it can be concluded that factors A, B, and C have a significant effect on the percentage of viscosity of NFC/PVA solutions in terms of the value of the S/N Ratio.

For the density response data, the comparison of F-ratios with the F table and hypothesis test results are as follows:

(i) Factor A: F-ratio (1.802) < 5.14. Thus, H_0 is accepted, and H_A is rejected.
(ii) Factor B: F-ratio (0.082) < 5.14. Thus, H_0 is accepted, and H_A is rejected.
(iii) Factor C: F-ratio (4.481) < 5.14. Thus, H_0 is accepted, and H_A is rejected.

Therefore, it can be concluded that factors Am B, and C have no significant effect on the percentage density of NFC/PVA solutions in terms of the value of the S/N *Ratio*.

The next calculation is the percentage of contribution for the average value and S/N ratio of viscosity and density response data. After the condition of the influential and significant factors has been known, it is necessary to determine the percentage of contribution from each factor, namely factor A, B, and C. This calculation uses the results of data processing from ANOVA. The calculation for the average value is below, where the viscosity and density response data obtained comes from Table 10, Table 11, Table 12, and Table 13. The equation used for getting this matter is SS" factor = $SS"_A = SS_A - (V_A V_e)$, where SS" factor or SS"A is the sum of the pure squares of factors and V factor is the degree of freedom on factors. The equation to determine the % contribution ρ is $\rho = \frac{SS"factor}{SStotal}$ x 100%.

Three sources of variation in the results are described in the other/error term in the last row of the ANOVA tables: uncontrollable factors, factors that were not taken into account during the trials, and experimental error [25].

The percentage of contribution serves to find out how much the percentage of the contribution influences the control factor on the response variable. The results of the percentage of contribution to the average value and S/N *ratio* for viscosity and density response data are presented in Table 14.

Table 14. Contribution percentage results

Value	Responses	Factor		
		A	B	C
Average	Viscosity	98.84%	1.53%	2.58%
	Density	8.61%	-9.87%	37.05%
S/N ratio	Viscosity	93.25%	2.67%	2.77%
	Density	8.56%	-9.81%	37.18%

After going through the previous stages, the setting parameters of the NFC/PVA ratio in the mixing volume/volume ratio and the duration of the sonication time are determined (see Table 15). This stage is performed to find out the parameters of the viscosity setting and density of the NFC/PVA solution for the efficiency of NFC raw materials. The setting parameters for each factor in the viscosity and density response data are presented in Table 15.

Table 15. Blending process parameters

Parameter	Factor	Optimal Level Value
Viscosity	NFC/PVA ratio	$\frac{5}{95}$
	Volume/volume ratio	10%
	Sonication Time	7 minutes
Density	NFC/PVA ratio	$\frac{10}{90}$
	Volume/volume ratio	3%
	Sonication Time	5 minutes

The determination of the above parameters can minimize the level of viscosity and density to be more efficient in the use of NFC raw materials during the blending process. The confidence interval is the highest and lowest value where the average value and S/N ratio are correct and will be covered by a certain percentage of confidence [5]. The calculation of the confidence interval at the average value and S/N ratio for viscosity and density response data can be presented below. The variables used are:

(3) Calculating Predictions. There are four types:

(i) Predicted the average value for the viscosity response data: $\bar{y} + (\overline{A2} - \bar{y})$ + - + - $(\overline{B3}\bar{y})(\overline{C2}\bar{y})$

(ii) Predictability values for viscosity response data: $\bar{y} + (\overline{A2} - \bar{y}) + - + - (\overline{B3}\bar{y})(\overline{C2}\bar{y})$
(iii) Predicted average values for density response data: $\bar{y} + (\overline{A3} - \bar{y}) + - + - (\overline{B1}\bar{y})(\overline{C1}\bar{y})$
(iv) Predicted variability values for density response data: $\bar{y} + (\overline{A3} - \bar{y}) + - + - (\overline{B1}\bar{y})(\overline{C1}\bar{y})$

(4) The Prediction of Confidence Interval with Equations and Variables used, respectively,

$$N_{eff} = \frac{\text{number of experiment}}{1+\text{ number of DoF}} \text{ and } CI = \sqrt{F(0{,}05;2;6) \times MSe \times \frac{1}{Neff}}.$$

Table 16 shows conclusions from the interpretation results of experiments which have been carried out for viscosity and density response data.

Table 16. Prediction response results based on parameters for density response data

Responses	Value	Predictions	Interval
Viscosity	Average	129.99	116.66 ≤ μ predictive ≤ 143.32
	S/N ratio	-42.64	-43.2 ≤ μ predictive ≤ -42.08
Density	Average	1.042	1.03 ≤ μ predictive ≤ 1.06
	S/N ratio	-0.365	-0.47 ≤ μ predictive ≤ -0.26

The results of the average value confidence interval and S/N ratio in this viscosity and density response data will later be used for the validity of the optimal level value at the time of confirmation of the experiment.

Confirmation of the experiment is the final stage in the Taguchi method. In this stage, an experiment was carried out to verify the conjecture in the determination of the experimental variables. The number of replications at the confirmation stage of the experiment must be greater than the replication of experiments that have been carried out previously. The results of the experimental confirmation of the viscosity and density response data are as follows. Subsequently, the results of the experimental confirmation are used to calculate the confidence interval with the equation 5 and 6.

Table 17. Result of experimental confirmation

Experiment	Viscosity Level of Experimental Results	Density Level of Experimental Results
1	140	1.136
2	140	1.124
3	140	1.118
Mean	140	1.126
S/N ratio	-42.92	-1.031

Based on the calculation of the confidence interval results at a 95% confidence level for the Taguchi method, it is compared with the confidence interval in the confirmation experiment. The average in the confirmation experiment is at the experiential confidence Taguchi. The results of the confidence interval are presented in Table 18.

Table 18. Interpretation of NFC/PVA solution viscosity level results

Experiment	Value	Unit	Predictions	Optimization
Viscosity Taguchi method	Average	mpas	129.99	129.99 ± 13.33
	Variability	mpas	-42.64	-42.64 ± 0.56
Viscosity Confirmation Experiments	Average	mpas	140	140 ± 15.87
	Variability	mpas	-42.92	-42.92 ± 0.67
Density Taguchi method	Average	g/mL	1.042	1.042 ± 0.013
	Variability	g/mL	-0.365	-0.365 ± 0.104
Density Confirmation Experiments	Average	g/mL	1.126	1,126 ± 0.015
	Variability	g/mL	-1.031	-1.031 ± 0.124

Based on the interpretation of the calculation results as in Table 18, the variability of value shows that the process is running better since in the combination of confirmation experiments (optimal setting parameters), the average viscosity value obtained is around 124 to 156 mpa's , respectively. On the other hand, the average viscosity value in combination of Taguchi methods is obtained around 117 to 144 mpas respectively. In the combination of confirmation experiments (optimal setting parameters), the average density value obtained is around 1.11 to 1.14 g/mL per sample. Meanwhile, the average viscosity value in combination of Taguchi method is obtained around 1,029 to 1,055 g/mL reviewed to this increment is due to a combination of factor levels which results in optimal output since it minimizes the viscosity and density of the NFC/PVA solution. Therefore, the optimal setting parameters which have been previously determined are proven to be valid.

CONCLUSION

Based on the research and data processing obtained results, for viscosity response data, the optimal setting parameter is obtained at the NFC/PVA ratio factor of 5/95, the volume/volume ratio of 10, and the sonication time of 7 minutes. Whereas, for the density response data, the optimal setting parameters were obtained at the NFC/PVA ratio of 10/90, the volume/volume ratio of 3%, and the sonication time of 5 minutes. The experimental confirmation results obtained for the Taguchi method for viscosity response data are 129.99 ± 13.33 on the average value and -42.64 ± 0.56 on the value of the S/N ratio. For the experimental results, the confirmation results are -42.64 ± 0.56 on the average value and -42.92 ± 0.67 on the value of the S/N ratio. Meanwhile, the response data for the Taguchi method of density is 1.042 ± 0.013 at the average value and -0.365 ± 0.104 at the S/N ratio value. The results of the confirmation experiment are 1.126 ± 0.015 on the average value and -1.031 ± 0.124 on the value of the S/N ratio. Based on the results of the experimental confirmation, it can be stated that there is an increase in the mean and variability. This shows that, if a combination of optimum parameters obtained from the calculation results is used, the efficiency of using nano fiber cellulose (NFC) raw materials is likely to succeed in reducing the level of viscosity and density.

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