



A Modelling Study by Response Surface Methodology (RSM) on Turbidity Removal Optimization using Nanocellulose Filter Paper (Neolamarckia Cadamba)

Nurul Aienna Ismail¹, Nor Hazren Abdul Hamid^{2*}

^{1,2}Department of Civil Engineering Technology,
 Universiti Tun Hussein Onn Malaysia, Johor, 84600, MALAYSIA

*Corresponding Author

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Abstract: This study observed the influence of initial turbidity, pH and initial temperature on the turbidity removal from the textile wastewater using nanocellulose filter paper from *Neolamarckia Cadamba*. Response Surface Methodology (RSM) model was employed to optimize and create a predictive model to evaluate the turbidity removal performance on the nanocellulose filter paper. The performance of the RSM model was statistically evaluated in terms of coefficient of determination, R^2 . The optimum value of turbidity removal of 99.39% were found at 66 NTU, pH 6.4 and 35.9°C. The value of prediction that obtained from modelling (RSM) was in agreement with the experimental values with $R^2 = 88.23\%$, AAD = 6.87% and RMSE = 0.18 towards the efficiency of turbidity removal.

Keywords: Textile wastewater, filtration, Cellulose Nanofibers (CNF), filter paper, turbidity removal, RSM optimization, CCD

1. Introduction

Textile products are one of the essential human requirements daily, for example, clothes. In 2018, according to the Malaysian Investment Development Authority (MIDA), Malaysia becomes the 13th largest of exporting industry in the textile and textile products. About RM12 billion of the total manufactured goods exports to Malaysia. However, textile industries used a large volume of water and chemicals for processing the wet units. It produced about 22% of the total volume of industrial wastewater that contained high concentrations of organic and inorganic compounds [1]. The textile industry has classified as one of the environmental difficulties that contributed to water pollution. It is caused by high-untreated wastewater that discharges into the environment. The turbidity of textile wastewater causes the liquid becomes opaque or cloudy. Thus, alternative treatment processes should be taken by the textile industry to back up from the pre until the post wastewater treatment process.

The filtration process can be considered as one of the great option treatments for effluent due to its universal nature, inexpensiveness and ease of operation [2]. It is a physical process in removing the particulate matter from the water suspension. Filtration using natural resources also has potential in treating textile wastewater. The process is more cost-effective compared to other treatment technologies. Nowadays, it is quite challenging to know which natural resources not explored yet by others to meet the needs of material in society. It is because most of the natural resources that have cellulose, especially in wood, already been exploit. *Neolamarckia cadamba* (*Kelampayan*) is one of the

biggest Malaysian renewable forest resources that will provide high-performance of filtration in turbidity wastewater treatment by chemically modified the cellulose material into Cellulose Nanofibers (CNF) to act as filtrated. Nanocelluloses is an element of nano-sized that can extract from cellulose in natural plant fibres that are known as nanocelluloses. CNF is high in the porous structure, which provides a sub-micron range of pore size. It is because of their amenability in surface modification and easy in the formation of thin membranes. By increasing the thickness of CNF, the contaminants in effluent or the water flux will decrease. It will result in different water flux values and pressure needed to measures the water flux depending on its membrane thickness [3]. Since cellulose is one of the most sources on this planet this study focused on the effectiveness of nanocellulose filter paper from local forest resources where *Neolamarckia cadamba* used as a starting material for the production of this nanocellulose filter paper.

The conventional optimization techniques are providing One Variable Change at a Time (OVAT) where the other variable is fixed and constant [4]. However, there are few limitations of the method which needs a large experiments number and consuming time. In this study, the removal of turbidity by filtration process was investigated. The effect of initial turbidity, pH, initial temperature and their interactions was evaluated using a Central Composite Design (CCD) that combined with RSM. CCD method is presented in two-variable rotatable for the independent variables and the number of the experiments were run all the parameters for each value. Thus, it can provide more accuracy and information in obtaining the data. There are five different levels of codes of independent variables where $-\alpha$, -1 , 0 , $+1$, and $+\alpha$. The combination of RSM with CCD is an efficient method for experimentally in venturing the relationships between the factor of investigation and system response [5]. RSM is a combination method of statistical and mathematical that used for experiments design, establishing a model in considering parameters and optimization process. It was focusing on the optimization of chemical reactions to achieved high yield, low costs of purifying wastewater, and others [6]. For the parameters, the selection is based on the scope of experiments and the frequent use in industrial designing and regulations of the environment. The selected independent variables in this study are pH (4 to 7), temperature (20 to 40°C), and initial turbidity (60 to 90 NTU), which may influence the filtration performance. According to the result of the selected experimental matrix, it is fitted with the mathematical equation that may describe the behaviour of response. The evaluation of quality model will use an Analysis of Variance (ANOVA) to study the significant impacts of the process variable for each response. ANOVA divided the variations into two-segment which is the variation of model attribution and the variation of experimental error attribution. The p -values obtained will measure the possibility of data collection slightly extreme as the model data. The lower of p -values obtained by the model, the more significant effect of the terms towards the predicted response. The pure error lack-of-fit test which utilized to evaluate either the model is sufficient to define the functional relationship among the response and experimental factors.

2. Materials and Methods

2.1 Dye Wastewater Samples

There are three main categories of materials used in this study. Firstly, the sample was taken from the textile industry located in Parit Sulong, Batu Pahat, using the method of grab sampling. Dissolved oxygen (DO) and pH were measured by HQ440d multi-parameter at the sampling point to avoid any changes during storage and transport. Next, the reagents used are sulphuric acid, H_2SO_4 and sodium hydroxide, NaOH to adjust the pH value and distilled water were used to adjust the turbidity value.

2.1.1 Characteristics of Textile Dye Wastewater

Textile dye wastewater was analyzed according to Standard Method for wastewater, which it is essential to evaluate the pollution level that could risk the environment and natural resources. There are a few characteristics of textile wastewater, such as turbidity, pH and color. The initial turbidity of the sample is 150 NTU while the maximum concentration of turbidity for aquatic life is less than 50 NTU.

2.1.2 Preparation of Textile Dye Wastewater

The sample of textile dye wastewater was adjusted based on the required experiment value of initial turbidity. Distilled water was added into the sample to reduce the turbidity concentration by using dilution method since the initial turbidity was 150 NTU.

2.2 Batch Filtration Experiments

The batch filtration experiment procedure used the sample of textile wastewater and been conducted with vacuum filtration pump. The first parameter value performed with initial turbidity (66.08 NTU), pH (4.61) and initial temperature (35.9°C) that obtained from CCD of scoping experiments. 35 mL of wastewater sample were measured which distilled was added to reduce the concentration of sample turbidity since its initial turbidity was 150 NTU. The initial pH of the sample was adjusted with 0.01M H₂SO₄ using the pH meter until 4.61 which the initial pH is 7. Lastly, the solution was heated on a hot plate until the temperature is 35.9°C. Then, the experiment was run by using vacuum filtration pump for 75 minutes. The turbidity after the filtration process was measured using TL2300 turbidity meter. All the 20 experiments were repeated with the same procedure as shown in Fig. 1. The removal efficiency of turbidity was calculated expressed by:

$$\text{Removal efficiency, \%} = \frac{C_i - C_f}{C_i} \times 100 \quad \text{Eq. 1}$$

where C_i and C_f are the initial and final concentrations of parameters.

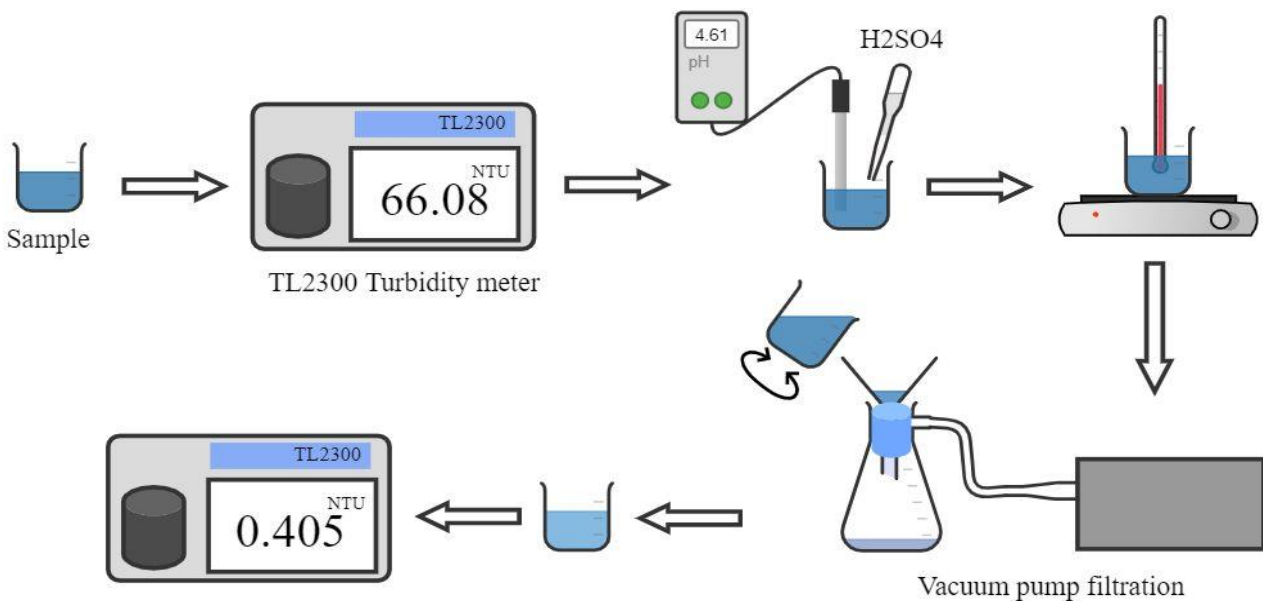


Fig. 1 - The steps of filtration process using vacuum pump filtration

After that, the optimum percentage of turbidity removal was selected to test the color removal by using DR6000 Spectrophotometer and total suspended solids. A “blank” solution is needed to “Zero” the spectrophotometers by choosing 97 color ADMI 1 inch. The efficiency of color removal was calculated as Eq. 1. Next, drying a clean piece of filter paper in the oven at 105°C for 1 hour and placed into a desiccator to cool it until becoming room temperature and weighed as initial weight using Mettler Toledo. After recorded the data, put the filter paper onto filtering vacuum filtration pump and filter the sample passing through the filter paper. Lastly, the filter paper after the filtration process was weighed by using repeated step for the initial weight. The value of TSS calculated as shown below:

$$\text{TSS mg/L} = \frac{(A-B) \times 1000}{V(L)} \quad \text{Eq. 2}$$

where A and B are the initial and final filter paper mass (g) and V is the volume of sample filtered (L).

2.3 Experimental Design and Analysis

The experiment design process that applied is Response Surface Methodology (RSM) with a combination of design strategies, Central Composite Design (CCD). Three effect variables were chosen in this study which are initial turbidity, pH and initial temperature of textile-based on water matrices types. About 20 experiments conducted to obtain the optimum operating condition of the turbidity removal via CNF filter paper from *Neolamarckia cadamba*. The parameters are shown in Table 1 with their coded levels (- α , -1, 0, +1 and + α = 1.682).

Table 1 - The ranges and levels of experimental independent variables for textile wastewater

Independent variable	Factor code	Range and level				
		- α	-1	0	+1	+ α
Initial turbidity	A	60	66.08	75	83.92	90
pH	B	4	4.61	5.5	6.39	7
Initial temperature	C	20	24.05	30	35.95	40

2.4 Response Surface Methodology (RSM)

RSM is a model that used critical mathematical and statistical methods to comprehend, enhance, and optimize a system. MINITAB 18 Statistical Software was used for the experimental data. The prediction of turbidity removal used the quadratic equation that shown below:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{1 < j} \sum \beta_{ij} x_i x_j + \varepsilon \quad Eq.3$$

where Y is turbidity removal, $\beta_0, \beta_i, \beta_{ii}$, and β_{ij} are constant coefficients and x_i and j_j the uncoded independent variables.

The MINITAB 18 Statistical Software has used to carry out the graphical analysis, regression analysis, and Analysis of Variance (ANOVA). The efficiency of the RSM model was analyzed statistically in terms of the Determination Coefficient (R^2), Absolute Average Deviation (AAD), and the Root Mean Squared Error (RMSE).

$$AAD = \frac{1}{n} \left(\sum_{i=1}^n \left(\frac{y_p - y_e}{y_e} \right) \right) \times 100 \quad Eq. 4$$

$$RMSE = \left(\frac{1}{n} \sum_{i=1}^n (y_p - y_e)^2 \right)^{1/2} \quad Eq.5$$

where n, y_p and y_e are the number of points, predicted value and experimental value.

3. Results and Discussion

3.1 Results and Discussion of Batch Experiment

There are 20 experiments of filtration process were conducted by using the independent variables of initial turbidity, pH and initial temperature as shown in Table 2. The experiments were performed to find out the optimum parameter that influenced the turbidity removal and the data acquired was used to running in CCD.

Table 2 - Data obtained from the batch experiment of filtration

Initial turbidity (NTU)	pH	Initial temperature (°C)	Final Turbidity (NTU)	Turbidity Removal (%)
66.08	4.61	24.05	0.549	99.17
83.92	4.61	24.05	0.888	98.94
66.08	6.40	24.05	0.438	99.34
83.92	6.40	24.05	0.701	99.16
66.08	4.61	35.95	0.524	99.21
83.92	4.61	35.95	0.869	98.96
66.08	6.40	35.95	0.405	99.39
83.92	6.40	35.95	0.678	99.19
60	5.5	30	0.596	99
90	5.5	30	0.877	99.03
75	4	30	0.986	98.69
75	7	30	0.684	99.09
75	5.5	20	0.655	99.13
75	5.5	40	0.586	99.22
75	5.5	30	0.524	99.3
75	5.5	30	0.569	99.24
75	5.5	30	0.526	99.3
75	5.5	30	0.587	99.22
75	5.5	30	0.604	99.19
75	5.5	30	0.612	99.18

According to E. Agency (2006), the initial turbidity of 74 NTU shows the highest percentage removal which is 59.45% [7]. The chosen range of turbidity for the studies was 61 to 87 NTU. Turbidity is one of the most practicable measures for the performance of filtration and it also practical on the physical parameter with which to gauge filtration physical parameter [8]. The larger value of porosity will cause the low quality of filtration and decrease the efficiency of turbidity removal [9]. According to the experiment, the percentage range of turbidity removal for 5 repeated parameter value (75 NTU, pH 5.5, 30°C) is 99.18% to 99.3%. It shows that the porosity value of *Neolamarckia Cadamba* filter paper is small and capable to improve the quality of filtration. This indicates the high efficiency of CNF filter paper from *Neolamarckia Cadamba* in removing turbidity of textile wastewater. However, the highest percentage removal is 99.39% with the parameter value of 66.08 NTU, 6.4 of pH and 35.9°C. The high efficiency of turbidity removal for 66.08 NTU also might be contributed from another parameter that affecting the filtration process which is pH and initial temperature of the sample.

Next, Lade et al., (2015) stated that the maximum percentage of dye reactive blue 172 removals by using medium of wheat bran is at pH 7 where the range of pH is 5 to 11 [10]. The pH of wastewater is vital in filtration due to the combination of organic and inorganic particulates in dye effluent. The combination may be affecting the deposition of suspended solid during the depth filtration [11]. Based on the experiment, the lower pH 4 indicates the lowest percentage of turbidity removal (98.69%) while the highest pH 7 able to contribute more percentage of reduction (99.09%) with the same value of initial turbidity (75 NTU) and initial temperature (30°C). Thus, it shows that the higher value of pH makes the electrostatic repulsion between the surfaces of membrane and molecule of dye become high and cause the aggregates of pigment to become smaller and loose the form of the cake layer [12]. Therefore, it influences the turbidity of textile wastewater easy to be removed through the filtration process.

Lastly, based on Abid et al., (2012) they used nanofiltration and reverse osmosis membrane for dye removal with the highest percentage of dye removal is 97.2% with the processing conditions for feed temperature is 26 to 39°C [13].

The temperature also one of the factors affecting the filtration process in removing turbidity of textile wastewater. Based on [14], the high temperature in the filtration process has contributed to the particle collection in technology development. It also improves the performance of the membrane due to the surface roughness and decreasing the porosity of membranes. Based on the experiment, the highest initial temperature (40°C) produce a high percentage of turbidity removal compared to the lowest initial temperature (20°C), which is 99.22% and 99.13%. At high temperature, the water viscosity is reduced due to the weakened cohesive forces, where fluid viscosity is inversely proportional to the rate of filtration [15].

3.2 Determination of Optimum Parameter Affecting the Filtration Process

The value of the optimum parameter was used for test of color removal and total suspended solids. The optimum value parameter that obtained from experiment is 66 NTU, pH 6.4 and 35.9°C with 99.39% of turbidity removal. Using the parameter value of optimum, the acquired percentage of decoloration is 73.5% as shown in Table 3. However, most of the previous study able to achieve their percentage removal exceed than 90%. For example, the experiment conducted by Yu et al., (2019) had reached their efficiency removal of methyl blue and methylene blue more than 95% by using filter paper supported by nano-scale zero-valent iron (nzVI) [16]. Thus, the lack number of test experiment may influence the possibility that prevents from achieving more than 90% of decoloration.

Table 3 - The data obtained for color removal

Initial color before filtration (ADMI)	Final color after filtration (ADMI)
147	39
Removal efficiency = 73.5%	

Next, the Total Suspended Solids (TSS) experiment has been conducted using the optimum parameter value. The experiment used to determine the solids that been traps by the filter paper from *Neolamarckia Cadamba*. Based on the investigation, the obtained result of TSS is 710 mg/L as shown in Table 4. The appearance of suspended solids like silt, clay and the organic and inorganic matter had induced to the formation of turbidity. In addition, the appearance of dyes and dissolved organic matter also lead to water turbidity [17]. Thus, it indicates the total suspended solids that contain in textile wastewater is high and does not achieve the requirement parameter limits of wastewater in Standard A and B which is 50 and 100 mg/L respectively. This might due to the deficiency knowledge and awareness of textile industry about concerning the harmful of materials consisted of textile wastewater, such as wax, dyes, and fixing agents. It may cause water pollution if discharged directly to the environment.

Table 4 - The data obtained from the TSS experiment

Initial mass of filter paper (g)	Final mass of filter paper (g)	Volume of sample filtered (L)
0.0990	0.1203	30
TSS = 710 mg/L		

3.3 RSM model results from CCD matrix conditions

The combination of RSM with CCD is used to study the relationship between the three factors of experiment and one system response. The turbidity removal is the system response of the filtration process with the independent variables of initial turbidity, pH and initial temperature. There are 5 levels of codes of independent variables where $-\alpha$, -1 , 0 , $+1$, and $+\alpha$. The value of α is important in CCD to maintaining the rotatability where it depended on the amount of runs experiment. The obtained regression equation in coded units for turbidity removal in the form of Y are shown in Eq. 6.

$$Y(\text{NTU}) = 0.5838 + 0.1239A - 0.0817B - 0.0235C + 0.0346A^2 + 0.0694B^2 - 0.0174C^2 - 0.0185AB + 0.0020AC - 0.0015BC \tag{Eq.6}$$

where Y , A , B and C are turbidity removal, Initial turbidity, pH and initial temperature.

The optimum turbidity removal of prediction by CCD was found at 75 NTU, pH 5.5 and 40°C with 99.34% while the optimum parameter of experimental was found at 66 NTU, pH 6.4 and 35.95°C of initial temperature with 99.31%. Thus, it shows that the optimum parameter of predicted was close with the optimum parameter of experimental which it is still in agreement. R^2 is the most commonly used to measure the goodness-of-fit of the regression model. The value of R^2 was defined as a percentage in this study. Usually, the expected values of R^2 for a physical process is over than 90% which indicates a very good measurement [18]. The predicted values that produced by RSM models are in good measurements with the actual data of experimental as shown in Table 5.

Table 5 - The experimentally of turbidity removal and predicted by the CCD models

Run number	Coded values			Turbidity removal (NTU)			
	A	B	C	Experimental	Predicted	Residual	Absolute Error (%)
1	-1	-1	-1	0.549	0.634	-0.085	15.48
2	1	-1	-1	0.888	0.914	-0.026	2.93
3	-1	1	-1	0.438	0.510	-0.072	16.44
4	1	1	-1	0.701	0.717	-0.016	2.28
5	-1	-1	1	0.524	0.586	-0.062	11.83
6	1	-1	1	0.869	0.875	-0.006	0.69
7	-1	1	1	0.405	0.456	-0.051	12.59
8	1	1	1	0.678	0.671	0.007	1.03
9	$-\alpha$	0	0	0.596	0.473	0.123	20.64
10	α	0	0	0.877	0.890	-0.013	1.48
11	0	$-\alpha$	0	0.986	0.917	0.069	6.70
12	0	α	0	0.684	0.643	0.041	5.99
13	0	0	$-\alpha$	0.655	0.574	0.081	12.37
14	0	0	α	0.524	0.495	0.029	5.53
15	0	0	0	0.586	0.584	0.002	0.34
16	0	0	0	0.569	0.584	-0.015	2.64
17	0	0	0	0.526	0.584	-0.058	11.03
18	0	0	0	0.587	0.584	0.003	0.51
19	0	0	0	0.604	0.584	0.020	3.31
20	0	0	0	0.612	0.584	0.028	4.58

A = Initial turbidity (NTU), B = pH, C = Initial temperature (°C)

Table 6 - The ANOVA for prediction of turbidity removal using RSM modelling

<i>Source</i>	<i>DF</i>	<i>Adj SS</i>	<i>Adj MS</i>	<i>F-Value</i>	<i>P-Value</i>
<i>Model</i>	9	0.402322	0.044702	8.33	< 0.001
<i>Linear</i>	3	0.308466	0.102822	19.16	< 0.000
<i>A</i>	1	0.209773	0.209773	39.09	< 0.000
<i>B</i>	1	0.091180	0.091180	16.99	< 0.002
<i>C</i>	1	0.007513	0.007513	1.40	0.264
<i>Square</i>	3	0.091068	0.030356	5.66	< 0.016
<i>A²</i>	1	0.017214	0.017214	3.21	0.104
<i>B²</i>	1	0.069383	0.069383	12.93	< 0.005
<i>C²</i>	1	0.004369	0.004369	0.81	0.388
<i>2- Way Interaction</i>	3	0.002788	0.000929	0.17	0.912
<i>AB</i>	1	0.002738	0.002738	0.51	0.491
<i>AC</i>	1	0.000032	0.000032	0.01	0.940
<i>BC</i>	1	0.000018	0.000018	0.00	0.955
<i>Error</i>	10	0.053666	0.005367		
<i>Lack-of-Fit</i>	5	0.048946	0.009789	10.37	< 0.011
<i>Pure Error</i>	5	0.004719	0.000944		
<i>Total</i>	19	0.455988			

$*R^2 = 88.23\%$, $Adj R^2 = 77.64\%$

The ANOVA is used to obtain the statistical parameters by using the RSM. ANOVA tests the significance and efficiency of the regression model as presented in Table 6. The F-values of the model is 8.33 which shows the models are significant respectively for the percentage of turbidity removal [19]. The F-value of the model described the ratio of the individual term means square to the residual mean square. The probability value of F-statistics is referred to the Prob > F-value (P-value) which to determine the relationship between the predictors and response and test the null hypothesis. If the value of F-statistics less than 0.05, it determined terms of the model is significant while the higher value of more than 0.10 shows the terms of the model are not significant [20]. Based on the obtained ANOVA table, the value of P > F for the model is significant due to the lower value than 0.05 which is 0.001. However, the P-value for linear and square of temperature (C and C²), the square of pH (B²) and all the 2-way interaction of independent variables (AB, AC and BC) are not significant to the turbidity removal. The parameter value of 60 NTU, pH 5.5 and 30°C has recorded the highest residual error which is 0.123 NTU and 20.64% of absolute error. Thus, the high percentage of absolute error has an impact on the value coefficient of determination (R² = 88.23%), absolute average deviation (AAD = 6.87%), and the root mean squared error (RMSE = 0.18). There are may be an error during conducting the experiment which the Buchner funnel of vacuum filtration pump does not clean properly where it needs to be cleaned using ethanol due to the dye attached on it. However, if the value of R is close to 1, it indicates that there is a good correlation between the predicted and experimental values. The P-value of lack-of-fit is significant due to its value is smaller than the level of significance, α (0.05) which P-value is 0.011. There is only a 1.1% chance lack-of-fit occurs due to the noise. Thus, the quadratic model with the predictor for the three independent variables is significant in predicting the removal of turbidity.

The normality prediction was acceptable because of the residual plot proximate on a line as shown in Fig. 2. The analysis of diagnostic case statistics of obtained data indicates the well fits the model in the optimization of turbidity removal based on the independent variables [20]. It is to measure the closeness of the points in the plot of normal probability in a straight line. Thus, the data of experiment was distributed as normally [21].

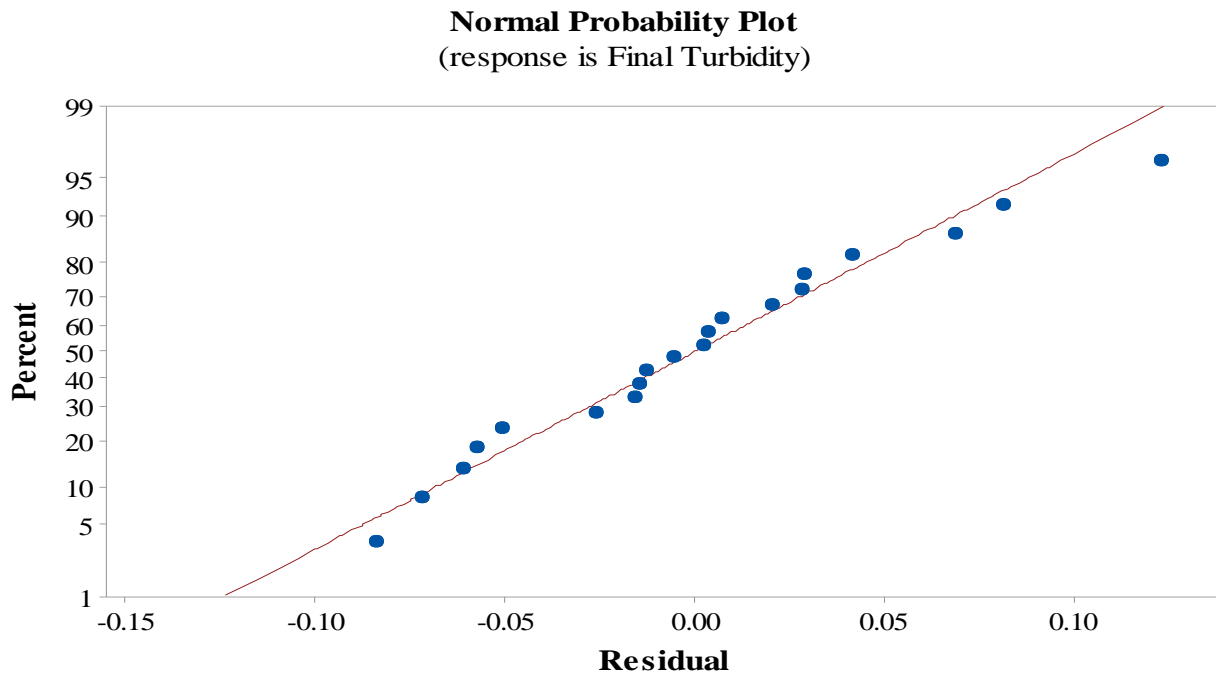


Fig. 2 - The normal probability plot of the residual

A surface plot only can provide two continuous independent variables. Fig. 3 shows the surface plot of the final turbidity versus pH and initial turbidity. Since there are three independent variables, the Minitab holds the values of the variable of 30°C initial temperature. The response surface is curved due to the terms of quadratic that contains in the model is statistically significant. The highest value of turbidity removal was at the behind of lower-left corner of the plot which the range of 60 NTU of initial turbidity and 6 to 7 of pH. Therefore, these plots only valid for the holds value of the extra variables. The exactness of the surface plot based on how well the model represents the actual relationship between the independent variables.

Surface Plot of Final Turbidity vs pH, Initial Turbidity

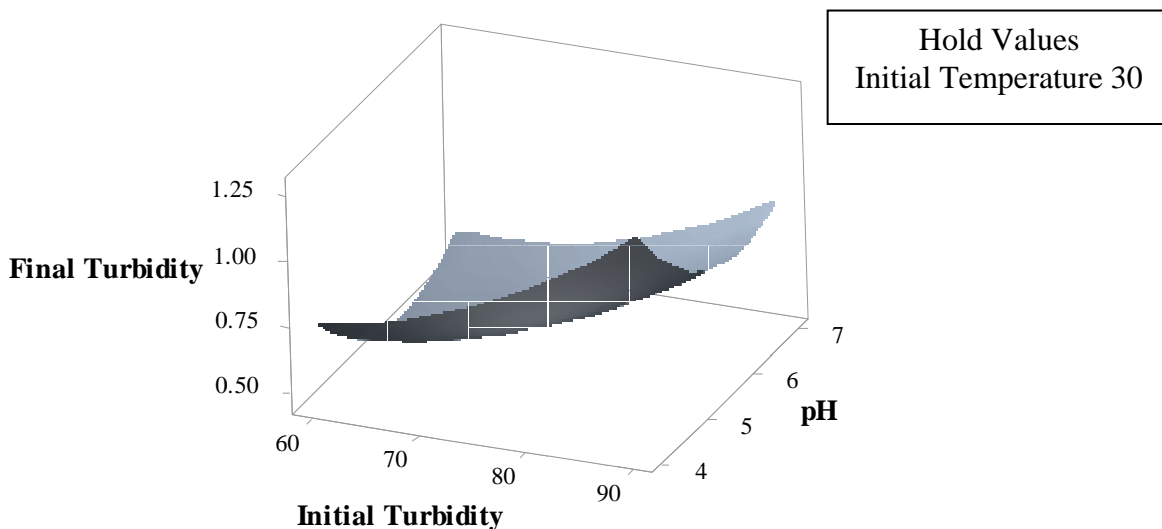


Fig. 3 - The surface plot of the final turbidity versus pH and initial turbidity

4. Conclusion

In conclusion, the cellulose-based nanomaterials that generated from local forest species of *Neolamarckia Cadamba* were efficient to remove turbidity and color from industrial textile wastewater by using the purification method of filtration. Based on the experimental data obtained, the optimum parameter for the filtration process are 66 NTU of initial turbidity, 6.4 of pH and 35.9°C of initial temperature which able to achieve 99.39% of turbidity removal. The application of Response Surface Methodology (RSM) via Central Composite Design (CCD) has proved as one of the reliable tools in optimization and modelling for treatment process parameters where it able to find out the interactive effects of the three variables: initial turbidity, pH and initial temperature on the efficiency of turbidity removal. The value of prediction obtained was in agreement with the experimental values which $R^2 = 88.23\%$, AAD = 6.87% and RMSE = 0.18 towards the efficiency of turbidity removal. Thus, *Neolamarckia Cadamba* is the best and efficient option as a starting material to be modified CNF into the filter paper.

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