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Optimization of Nanocellulose Filter Paper from Forest Resources (*Shorea Roxburghii*) for Water Purification in Textile Wastewater using RSM

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Abstract: In this study, results of parametric effects and optimization of turbidity removal from textile wastewater using response surface methodology (RSM) based on a statistically designed experimentation via the Central composite design (CCD) are reported. A five-level, three-factor CCD was employed using initial turbidity (X_1), pH (X_2) and initial temperature (X_3) as process variables. The RSM model predicted an optimal turbidity removal efficiency of 98.88 % at conditions of X_1 (75 NTU), X_2 (5.5 pH) and X_3 (40 °C). The model was by checking the coefficient of determination, R^2 (0.80) and proved to have strong effect size. The lack of fit for the model with high probability (P = 0.956) and low F-value (F = 0.19) supported the efficiency of model to predict turbidity removal percentage. X_3 have a negative coefficient value which indicates the directly proportional relationship with the turbidity removal. Other two terms (X_1X_2) and (X_1X_3) shown negative coefficient as well which demonstrate the relationship between these three variables. Confirmation of experimental results was found to be close to the prediction derived from the models. This demonstrates the benefits of the approach based on the RSM in achieving good predictions while minimizing the number of required experiments.

Keywords: Turbidity removal, CCD, RSM, R^2, Lack-of-fit

1. Introduction

The earliest modern human was evolved and discovered for about 6 million years ago and still exists until today [1]. Textile plays an important role in human civilization as it is used to make clothes such as carpets, towel and other applications. Over the past centuries, textile industry undeniably contributes a lot in the world economic, textile industry as one of the biggest industries in the world, the global textile industry worth around 1 billion US dollar and it contributes 7.00 % of the world economic such as exports and employments [2]. However, textile industry is also known as major contributor to the

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environmental pollution as dye effluent is produced and released to the environment during the process [2].

Table 1 : Characteristics of Textile Effluent [3]

Parameter	Unit	Quantity
Dye concentration	mg/L	700
pH	-	10
TSS	g/mL	61-87

The very first problem caused by textile wastewater would be the visual pollution which the original colors lost during the dyeing process. It blocks the sunlight from reaching the bottom of water bodies when mixed and dissolved in water, affecting the photosynthesis process of the plants in the water. Some studies also stated that the dyes in waterbodies would undergo chemical and biological reactions and caused eutrophication [4]. These both situations would end up causing low dissolved oxygen of water bodies, oxygen deficiency to aquatic life leading to the death of aquatic life and then changes in the biological cycles of aquatic biota [5]. The presence of heavy metals in water bodies also affecting the aquatic life as the heavy metals cations can be assimilated by the fish gills since negative charges are present on their gills lead to the accumulation of heavy metals in their tissues [6]. After accumulation occurred for a long term and reach a high level, the toxicity of heavy metals could lead to the death of aquatic life and effect in the biosystem. High turbidity can significantly reduce the aesthetic value of a water body which could lead to serious effect in recreation and tourism sector [7]. High turbidity also can degrade spawning beds and affect the gill function of fish [7]. Symptoms such as headaches, cramps, nauseas, diarrhea, flatulence could be found as if human consuming water with high turbidity for long period [8].

Filtration is one of the examples of physical treatment and can be considered as the most important method in treating textile wastewater [9]. It can remove materials in the range from large visible particles to molecular to ionic chemical species. This indicates that filtration method able to clean and separate the suspended solid from textile wastewater with high efficiency. The most common and conventional filtration method is direct filtration since it is simple and economically attractive as the sedimentation process is avoided in this method [9]. In filtration process, the selection of the filter media is important depends on the characteristics of the industries [9]. Proper selection of filter media ensures the efficiency of liquid-solid separation in the filtration process. The media used for filtration can be categorized by their materials of construction, for example, cotton, wool, linen, glass fiber, porous carbon, metals and rayon. Although many types of filters are existed in market, but not all of them are used commercially. More and more materials are still being created by researchers to enhance filtration process as well as replacing the old materials for sustainability [10].

Cellulose nanofibers also known as cellulose nanofibrils are generally defined as a natural cellulosic material, one of the divisions of cellulose nanomaterials which consists of fibrils [11]. It consists high volume and big surface area of cellulose fibrils and fibrils with dimension in nanometric scale and is obtained through mechanochemical treatments [11], [12]. Cellulose nanofibers are nano-sized smaller than 100nm or a micro-sized fiber with nano-dimension cross-sectional structures [13]. It is described as a long, flexible, rope-like fibers with both crystalline and amorphous regions [14]. Sources of cellulose nanofibers are mainly woods, however, alternate cellulose nanofibers such as agriculture crops, water plants, algae and others were found and accepted due to their characteristics of short growth periods and easy delignification [11]. The derived CNF has been proven appropriate for the adsorption of heavy metals [15]. CNF also contains dead cells and cellular debris residues providing extra sites for adsorption since they are rich in metal-binding functionalities for examples, phosphoryls, carboxyls, hydroxyls, amines, phosphates and others [15].

Elevation of environmental impact in global causing human to begin focus on eco-friendly technologies as well as renewable resources [11]. Plants will be the best fitting "renewable resources". *Shorea Roxburghii*, one of the local resources was selected to be used for production of CNF filter paper. *Shorea Roxburghii* as a useful species for silviculture in the tropics due to its characteristics of tolerance to heavy drought and high survivorship [16]. The locals also use it for medical purpose by decocting the bark for dysentery treatment. Other than that, the barks and the flowers of *Shorea Roxburghii* are edible, the bark is used as masticatory and usually will be chewed with betel nuts. In Malaysia, Research Institute Malaysia (FRIM) has planted and grew the trees to conserve it from extinction [17]. Since it is grown and used for various purposes, it can be easily obtained from local forest which the issue lack of raw material would not need to be worried. Utilizing raw materials from local forest resources also benefits the environment since the raw materials are environmental-friendly and can be easily decomposed which do not compose threat to the environment.

Response Surface Methodology (RSM) is a commercially used mathematical and statistical method with purpose of modelling and analyzing a process in which the response of interest is affected by various variables [18]. One of its advantages of application is that it can be applied to accord better understanding with minimum experiment conduct. Central composite designs (CCD) is most commonly used response surface designed experiment. It can be used to estimate first and second order terms effectively. It is especially useful in sequential experiments because you can often build on previous factorial experiments by adding axial and center points.

There is limited published study on modeling and optimization of the process parameters for turbidity removal from textile wastewater using nanocellulose filter paper. Moreover, previous studies on turbidity removal from textile wastewater seem to rely mostly on one factor at a time (OFAT) approach. Researchers have proven that such method is deficient, time consuming and unsatisfactory [19].

To achieve the aim of this study, four objectives are listed. The objectives are to study the ability of cellulose-based nanomaterials (*Shorea Roxburghii*) for turbidity removal from textile industry wastewater and as a replacement for current filter paper material. Physicochemical properties of the wastewater via benchmark experiments is going to be studied in order to provide better understanding of the impacts of wastewater matrix on the removal of turbidity from wastewater matrix. The following objectives are to build the RSM model and access its capability to determine the effectiveness of CNF filter paper at removing turbidity from industrial wastewater as well as to optimize the filtration process parameters such as pH, initial temperature, and turbidity for removal of color or turbidity from wastewater matrices.

2. Materials and Methods

Response Surface Methodology (RSM) based on CCD, was used in modelling and optimizing the factors that influence filtration process for efficient turbidity removal.

2.1 Materials

The materials that are used in the study can be divided into two parts which are the raw materials and the chemicals to prepare textile wastewater effluent. The reagents (chemicals) used were listed in Table 2 below while the raw material used was CNF filter paper produced from *Shorea Roxbughii.*

Table 2: Reagents used in textile wastewater preparation

Reagents	Function
Hydrochloric Acid (HCl)	For pH range modification
Sulphuric Acid (H_2SO_4)	For pH range modification

Sodium Hydroxide (NaOH)	For pH range modification
Methylene Blue	To adjust the color concentration of textile wastewater sample

2.2 Preparation of wastewater sample

Textile wastewater will be collected from the Anfi Industry Sdn. Bhd in Batu Pahat, Johor. 20 experiments will be carried out with different parameter values each. The initial turbidity of the textile wastewater will be analyzed using turbidimeter, TL2300. Selection of parameters and ranges was done through literature review. Table 3 shows the selected parameters and the ranges for each.

Table 3: Table of parameter ranges

Turbidity (NTU)	pH	Initial temperature (℃)
60 - 90	4 - 6	20 - 40

2.3 Design of experiments

Response Surface Methodology (RSM) based on CCD, was used in modelling and optimizing the factors that influence filtration process for efficient turbidity removal. Three variables such as pH, temperature, adsorbent dosage and the initial concentration of dye are studied to carry out optimization studies. Figure 1 shows the design of experiment and steps of the projects.

$$N = 2^n + 2n + n_c$$

$$= 2^3 + 2(3) + 6 = 20$$

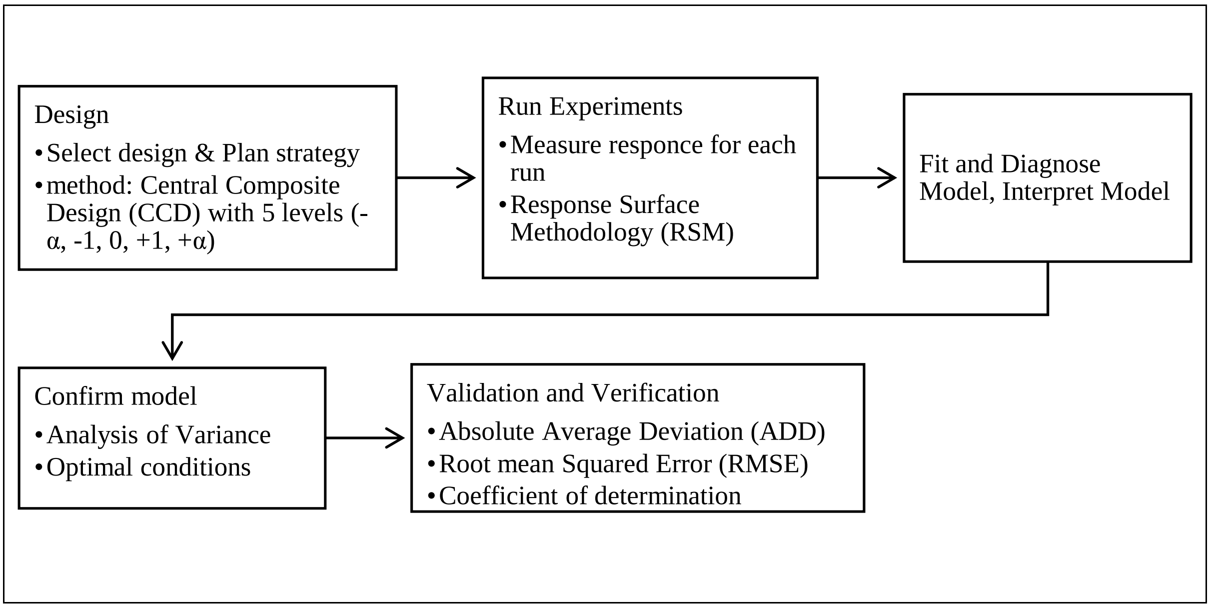

Figure 1: Design of experiment (selection of experiment design)

2.4 Application of CCD

Three independent variables are existed in this experiment which are pH (5 to 8), temperature (25 to 100) and initial concentration of textile effluent spiked with dyes (80 to 250ADMI) are optimized for the removal of dyes from textile effluent. The 20 experiments of CCD are conducted as stated at section

3.2 at constant pH of 6. The parameters are shown in Table 4 with coded levels of (–α, -1, 0, 1, α; α = 1.68).

Table 4: Ranges and levels experimental of independent variables for textile effluent

Independent variable	Factor code	Range and level				
		-α	-1	0	1	α
Initial Turbidity	X_1	60	66.08	75	83.92	90
pH	X_2	4	4.61	5.5	6.39	7
Initial Temperature	X_3	20	24.05	30	35.95	40

2.5 Mathematical Modelling using RSM

Response Surface Methodology (RSM) is a commercially used mathematical and statistical method with purpose of modelling and analyzing a process in which the response of interest is affected by various variables (Aydar, 2018). One of its advantages of application is that it can be applied to accord better understanding with minimum experiment conduct. Experimental data are also processed with software MINITAB 19 Statistical Software. Predicted percentage of dyes removal from textile wastewater is explained by the following quadratic equation:

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

2.6 Evaluation of model

In models evaluation, MINITAB 16 is used to carry out the regression analysis. graphical analysis, and analysis of variance (ANOVA). The RSM model's performance is statistically evaluated in terms of the coefficient of determination (R^2), Absolute Average Deviation (AAD), and Root Mean Squared Error (RMSE). R^2 value = 1 indicates the ideal matching between predicted and actual data. The models and the parameters variation are going to be determined based on the minimum value of RMSE and AAD of the training and prediction set as shown below:

$$\boldsymbol{AAD = \frac{1}{n}(\sum_{i=1}^{n}\left(\frac{y_p - y_e}{y_e}\right)) \times 100} \quad \text{Eq.2}$$

$$\boldsymbol{RMSE = \left(\frac{1}{n}\sum_{i=1}^{n}(y_p - y_e)^2\right)^{1/2}} \quad \text{Eq.3}$$

3. Results and Discussion

3.1 Results

The ranges of three parameters were selected from literature review which were initial turbidity (60-90 NTU), pH (4-7) and initial temperature (20-40 ℃). Batch experiments developed by CCD were done and obtained an average of 98.00 % turbidity removal. Average data of six experiments with similar wastewater parameters value (75 NTU, 5.5 pH, 30 ℃) was obtained which was 98.09 % of turbidity removal percentage. Average data was obtained in order to ensure the accuracy of the result and model. The highest turbidity removal percentage among 20 experiments was 98.88 % which have parameters of 75 NTU (initial turbidity), 5.5 pH and 40 ℃ (initial temperature).

3.2 Effect of temperature

It was clear that a high and a low initial temperature (20 ℃, 40 ℃) of wastewater seems to be more effective in turbidity removal while a middle range temperature, 30 ℃ was inefficient to remove turbidity. According to a research of wastewater streams pre-treatment, the temperature of highest turbidity removal was at 22 ℃ compared to 13 ℃ and 43 ℃ which is almost similar as the initial temperature of second highest turbidity removal in this study [21]. More researches shown that higher temperature of wastewater effluent has greater influence on turbidity removal, the residual turbidity increased when the temperature decreased [22]. Since two results shown contradiction, therefore the initial temperature parameter alone might not cause much influence on turbidity removal using filtration method.

3.3 Effect of pH

The effect of pH can be seen by comparing experiments of 11st, 12rd and the average of six repeating experiments. Their values of initial turbidity and initial temperature were fixed and only pH is different. Experiment of 11st run has a pH value of 4, 12rd run has a pH of 7 while the six repeating experiments have a pH of 5.5. The final turbidity of for experiment with pH 4 was 98.28 % and 98.37 % for experiment with pH 7. The six experiments with pH 5.5 have an average final turbidity of 98.09 %. Based on these three experiments, wastewater with pH 7 was the highest turbidity removal, the second was wastewater with pH 4 while pH 5.5 got the least turbidity removal. Since the data obtained do not show a strong evidence about the relationship between pH and the turbidity removal of wastewater, it might be only effect while combine with other parameter which it did not have significant effect alone. A research the correlation coefficient of pH and turbidity only shown 0.115361 which means there is very low insignificant correlation. This indicates no direct effect of pH on turbidity of wastewater (Kumar Mandal, 2014). However, pH might still have effect on the CNF filter paper as well as react with other parameters on turbidity removal.

3.4 CCD

CCD was used to develop a correlation between three selected independent variables and one output or response. These three selected independent variables are initial temperature, initial turbidity, and pH. All these selected variables were studied for their impact on the turbidity removal from textile wastewater with a filter paper produced from CNF of *Shorea Roxburghii*. A total of 20 experiments were generated with different parameters values each and required to determine the optimum operating conditions for the filtration of TSS using CNF filter paper. The range of independent variables alongside their coded levels (-α, -1, 0, 1, α; α = 1.682, respectively) shown in Table 5. The central point was repeated six times in order to obtain good estimate of experimental error or pure error. The quadratic equation in terms of coded factors generated from the analysis for turbidity removal percentage in the form of Y is shown as below:

$$Y(\%) = 108.85 - 0.097X_1 - 0.179X_2 - 0.460X_3 + 0.001117X_1X_1 + 0.0890X_2X_2 + 0.007488X_3X_3 - 0.01101X_1X_2 - 0.00009X_1X_3 + 0.0017X_2X_3$$

Where Y is the turbidity removal percentage, X1 is initial turbidity, X2 is pH and X3 is initial temperature.

Table 5: CCD Experimental ranges and levels of the independent variables

Independent variable	Factor code	Range and level				
		-α	-1	0	1	α
Initial Turbidity	X_1	60	66.08	75	83.92	90
pH	X_2	4	4.61	5.5	6.39	7
Initial Temperature	X_3	20	24.05	30	35.95	40

3.5 RSM

The results of turbidity removal percentage were obtained by performing batch experiments according to the CCD matrix and condition. Table below shows the experimental results obtained from the experimental runs as well as the predicted values by the build of RSM model. The percentage error was calculated as the ratio difference in experimental and predicted value to experimental value. The formula of percentage error or absolute error is experiment value minus predicted value then divide by experimental value multiply by 100. Based on previous researches by using CCD and RSM, the results of removal are varied from 10 percent to 96 percent [10], [23]. However, the results of removal of turbidity removal percentage obtained from batch experiment in this study are ranging from 97 to 98.8 percent which is too consistent. Under optimum condition, the percentage turbidity removal was found to be 76.30 % (sand bed) and 81.40 % (dual media filter) [24]. The performance of CNF filter paper is even better than and able to filter an average 98 percent of suspended solids that floating and drifting in textile wastewater. The pore size of filter paper could directly affect turbidity removal of wastewater as smaller pore size could trap and block more TSS in water [25].

Table 6: The experimentally obtained percentage of turbidity removal compared to predicted by central composite design (CCD) model

Run Number	Coded values			Actual values			Turbidity Removal (%)			
	X_1	X_2	X_3	X_1	X_2	X_3	Experimental	Predicted	Residual	Absolute Error (%)
1	0	α	0	75	7	30	98.37	97.92	0.45	0.46
2	α	0.	0	90	5.50	30	98.56	98.12	0.44	0.44
3	0	0	0	75	5.50	30	97.84	97.76	0.08	0.08
4	0	0	0	75	5.50	30	97.96	97.76	0.2	0.20
5	1	-1	-1	83.92	4.61	24.05	98.69	98.47	0.22	0.22
6	0	0	0	75	5.50	30	98.31	97.76	0.55	0.56
7	-1	-1	-1	66.08	4.61	24.05	98.44	98.16	0.28	0.29
8	-α	0	0	60	5.50	30	98.20	97.90	0.30	0.31
9	0	0	0	75	5.50	30	97.87	97.76	0.11	0.11
10	0	0	0	75	5.50	30	98.37	97.76	0.61	0.62
11	-1	1	1	66	6.39	35.95	98.50	98.09	0.41	0.42
12	0	0	-α	75	5.50	20	98.87	98.68	0.19	0.19
13	1	1	-1	83.92	6.39	24.05	98.56	98.25	0.31	0.31
14	0	0	0	75	5.50	30	98.20	97.76	0.44	0.45
15	1	1	1	83.92	6.39	35.95	98.38	98.03	0.35	0.36
16	1	-1	1	83.92	4.61	35.95	98.52	98.25	0.27	0.28
17	-1	1	-1	66.08	6.39	24.05	98.61	98.28	0.3323	0.34
18	-1	-1	1	66.08	4.61	35.95	98.24	97.96	0.28	0.29
19	0	0	α	75	5.50	40	98.88	98.34	0.54	0.55
20	0	-α	0	75	4	30	98.28	98.00	0.28	0.28

The evaluation of quality of fitted model was done by using analysis of variance (ANOVA) and the model is presented in Table 7. To access the suitability of the model, the lack of fit was examined through ANOVA. In general, the P-value are used to interpret the relationships among the variables [23]. High probability (P = 0.956) and low F-value of 0.19 indicates that the RSM mode is suitable and able to predict the turbidity removal percentage effectively from textile wastewater [10].

The significance of each term in the equation regarding the percentage of turbidity removal was validated by this statistical test. As can be seen from the results, most of the terms in the quadratic model were significant since P-value is 0.956 in terms of their effect on turbidity removal percentage with a model F-value of 0.19.

P-value of the model was lower than α (α = 0.05), indicating the model was statically significant. The significance of the model can be supported by the regression coefficient, R^2 obtained which is 0.80 (79.73 %) as shown in figure 4.1. R^2 value always lied within 0 to 1 and its magnitude indicates the aptness of the model (Yahya et al., 2019). A good statistical model should have a R^2 value close to 1.0. Based on this model, the R^2 (0.80) is consider acceptable as the R^2 value is greater than 0.7 which is considered a strong effect size [26]. As can be seen from the Table 7, the p-value of almost all sources is higher than alpha (α = 0.05). This indicates that the results are statically insignificant and unlikely to occur by chance alone. However, the second-order effects of initial temperature (X_3^2) is significant among other second-order effects, indicating the significant effect of this factor on the percentage of turbidity removal.

The negative value on effect of initial temperature (X_3) indicates that the increased of initial temperature would cause a decrease in percentage of turbidity removal. There are two other negative coefficient which are combination of initial turbidity and pH (X_1X_2) as well as combination of initial turbidity and initial temperature (X_1X_2). Negative coefficient of these two sources demonstrates the relationship between turbidity, pH and turbidity, initial temperature.

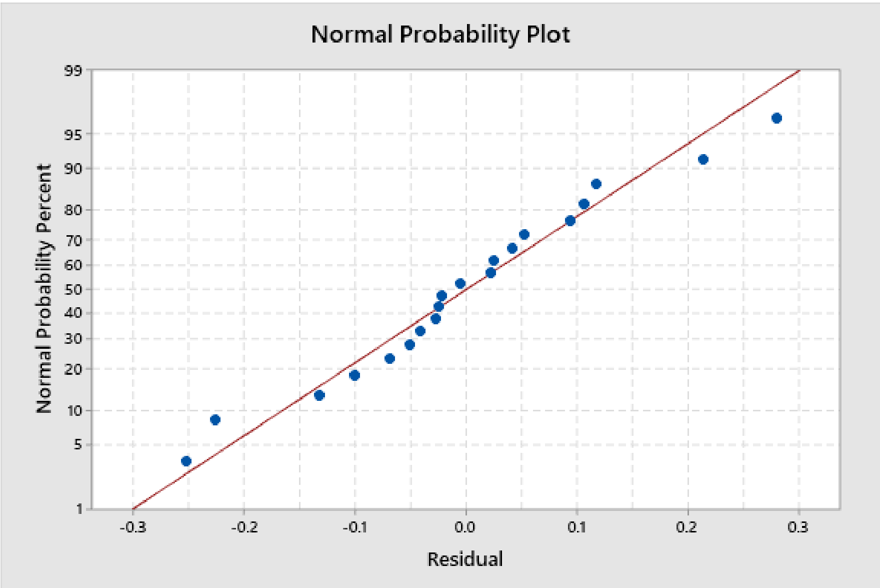

Figure 2: Graph of normal probability plot

Table 7: Predictive abilities of RSM models

Data Index	RSM		
	Coefficient of Determination (R^2)	AAD (%)	RMSE
20 CCD	0.7973	0.338	0.360147

Based on Table 7 above, the coefficient of determination (R^2) obtained from this model was 0.7973. The model was considered acceptable since the coefficient of determination is greater than 0.7 which indicates a strong size effect [26]. The average absolute deviation (AAD) is similar with standard deviation which both are used to measure of spread. The AAD of this model obtained from calculation was shown in Table 7 which was 0.338 %. The AAD percentage is so small and even less than 1 percent. The data of obtained for this model is categorized into cluster type. Smaller AAD also indicated that this model is reliable to indicate the other values using this model. Other than that, root mean square error for the model was calculated as well, which was 0.3601. Since the RMSE was smaller than 0.5, this indicated the ability of the model to predict the data was within an acceptable range and was considered accurate [27].

3.6 Characterization of wastewater under optimized parameters based on RSM

3.6.1 Total Suspended Solids (TSS)

For TSS experiment, the study was conducted under optimal condition, which was determined from the batch experiments developed from CCD. The optimal condition was 75 NTU (initial turbidity), 5.5 pH and 40 ℃ (initial temperature). The turbidity of textile wastewater was caused by the amount of TSS in the wastewater. Higher turbidity indicates higher TSS. The TSS obtained from the study was 2175 mg/L which is around 98% of turbidity removal as shown in Table 8 According to research on characteristics of textile wastewater, the average total suspended solids was ranging from 830 mg/L to 1580 mg/L [28]. The TSS obtained from study was even higher compared to the data obtained from other research study. In contrast, large amount of filtered TSS (2175 mg/L) or 98.00 % turbidity removal proved that the efficiency of CNF filter paper produced from *Shorea Roxburghii* on turbidity removal was excellent. Based on the legislation in Malaysia, the total suspended solids limit for wastewater discharged was 50 mg/L (Standard A) and 100 mg/L (Standard B) and the turbidity required was less than 10 NTU [29]. By having the information of 98.00 % turbidity removal and TSS of 2175 mg/L, the original TSS amount can be calculated which was 2219.39 mg/L. The residual obtained by using original TSS to minus TSS obtained from filtration was 44.39. Compared to the limitation, the TSS of wastewater after filtration was acceptable. By using the CNF filter paper, turbidity of wastewater could be filtered effectively and ensured to be within the range limited by the government.

Table 9: Result of TSS for turbidity removal

	Results
Volume of sample, mL	40
Initial weight of filter, g	0.1218
Final weight of filter, g	0.1305
Weight of solid, g	0.87
TSS, mg/L	2175

3.6.2 Color

A decolorization test was done by using DR6000 with program ADMI 1 inch to test the color removal of textile wastewater. The initial color of textile effluent with optimum parameter tested was 112 ADMI. After the textile effluent was filtered using CNF filter paper, the final color tested was 18 ADMI. The percentage of color removal can be calculated by using formula initial color minus final color divide by initial color and then multiply by 100. Color removal percentage obtained was 83.95%. Based on the observation, the color of filtered effluent is approximately similar as the color of distilled water hardly can be recognized. The efficiency of color removal by using CNF filter paper was considered great. According to Malaysia legislation of effluent discharge, fifth schedule paragraph 11(1)(a), the acceptable color ADMI for standard A is 10 ADMI and 20 ADMI for standard B [29]. The result obtained from decolorization study was 18 ADMI which was sufficed to meet the limit of effluent discharge for standard B. This result shown the performance of CNF filter paper produced from *Shorea Roxburghii* was excellent. However, the result still could not meet the limit of effluent discharge for standard A which is 10 ADMI. Further decolorization is required in order to reach the 10 AMDI limit for standard A. The potential of nanocellulose fiber filter paper in dye removal can be recognized due to its ability to remove 50.00 % to 59.00 % of color and even higher in this study [30]. Moreover, the initial color of textile effluent tested by using DR6000 cannot be more than 300 as it is the calibration range. Further research and experiments are required in order to obtain the data of color removal with higher initial textile wastewater color.

4. Conclusion

The main aim of this study is to predict the capability of CNF filter paper for the removal of turbidity from wastewater under realistic conditions using RSM modelling. From the result reported it could be concluded that the turbidity removal was successfully achieved. Process variables optimization carried out via five-level, three factors CCD suggested that removal efficiency of turbidity from textile wastewater depends actively on initial turbidity (X_1), pH (X_2) and initial temperature (X_3). The RSM model predicted an optimal turbidity removal efficiency of 98.88% at conditions of X_1 (75 NTU), X_2 (5.5 pH) and X_3 (40°C). The lack of fit for the model with high probability (P = 0.956) and low F-value (F = 0.19) supported the efficiency of model to predict turbidity removal percentage. X_3 have a negative coefficient value which indicates the directly proportional relationship with the turbidity removal. Other two terms (X_1X_2) and (X_1X_3) shown negative coefficient as well which demonstrate the relationship between these three variables. Moreover, the coefficient of determination (R^2) obtained from the model is 0.80 which have strong effect size since it is greater than 0.7. AAD and RMSE calculated to evaluate were 0.338 and 0.3601. Both values proved the reliability and the accuracy of model in predicting data for future.

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