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Comparison of Central Composite and Box-Behnken Design in Optimization of Turbidity Removal using Nanocellulose Filter Paper (*Neolarmarckia cadamba*)

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Abstract: Filtration process using *Neolamarckia cadamba* filter paper for dye effluents removal from dye industrial wastewater offer a favorable solution which suits well with the definition of sustainability. This study was aimed at using the Central Composite Design (CCD) and Box-Behnken Design (BBD) to compare the efficiency and to elucidate the main interacting parameters of turbidity removal using MINITAB 18 Statistical Software. At optimized conditions, the filtration process would be able to achieve 99.39 % turbidity removal efficiency for CCD and 99.69 % for BBD, respectively. The optimum conditions for the filtration process of CCD model on turbidity removal are initial turbidity of 66NTU, pH of 6.4 and initial temperature of 36.00 ºC while BBD is 75 NTU for initial turbidity, pH of 5.5 and initial temperature of 30.00 ºC. Both design of experiments were successfully applied in this study. R^2 values of 73.42 % and 90.95 % for CCD and BBD models, which indicate that both models are statistically significant with each other. However, BBD consumes limited time with fewer experimental runs, is efficient, and is commonly used in wastewater treatment. Therefore, this study showed that BBD model was the best option in terms of number of required experiments and quality of the obtained data.

Keywords: Filtration Process, Turbidity Removal, Central Composite Design, Box-Behnken Design, Response Surface Methodology, Filter Paper

1. Introduction

Water pollution has become a serious environmental problem over the last few decades due to the release of many types of pollutants which then become one of the noticeable threats to humans, animals and also to the ecosystem and [1]. Meanwhile, textile industry produces a wide range of polluting dye waste which is considered as threatening the water industry [2]. Dye color are usually visible at a dye concentration above 1 mg/L and has been reported to surpass the concentration in textile manufacturing

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effluent because mainly almost 10.00 % to 15.00 % of the dyes lost into the water during the dyeing processes [3]. The transport of these effluents through the food chain could seriously affect human health, animals and environment. Some of the dyes were produced from hazardous chemicals are carcinogenic and mutagenic in all forms of life and can interfere with sunlight transmission, which reduces the photosynthetic activity of aquatic organisms and also affects aesthetic beauty [2]. Presently, physical treatment, chemical treatment and biological treatment are the usual used techniques for treating the pollution. Most sophisticated methods are required for treating wastewater in textile industries such as chemical precipitation, ion exchange, electrolysis, coagulation, solvent extraction, reverse osmosis, and electrocoagulation [1]. However, most of these technique is less efficient in the removal of contaminants and are characterized by low selectivity, applications of high reagent, chemical and energy requirements, the generation of other toxic wastes that need to be handled carefully, and high cost [1]. Among all the available purification techniques, filtration treatment is a quick and effective process for removal of all types of dye [1]. It is one of the oldest and simplest methods of removing contaminants in the wastewater [3]. Renewable approaches involving the use of natural materials for dye effluents removal from wastewater offer a favorable solution which suits well with the definition of sustainability [4]. *Neolamarckia cadamba* are abundant and renewable plants that can produce environmental friendly, cost effective and biodegradable starting materials for the production of filter paper for the treatment of textile industrial wastewater which meet the development of environmental and economic targets in many regions of the world, aiming not only at improving wastewater treatment processes but also at minimizing the negative impacts of wastewater treatment on human health [5]. Implementing environmentally friendly green alternatives at low cost with superior performance and lower carbon footprint is important for a sustainable future.

Response surface methodology (RSM) is a multivariate statistical tool, consists of a group of mathematical and statistical techniques that are based on the fit of empirical models to the experimental data obtained in relation to experimental design. This statistical tools also helps assist to design optimization which is aimed at saving time and reducing cost of expensive analysis methods. Moreover, it is an establishment of strategies to protect and reduce negative environmental effects, and to replace material that has degraded the environment. Driven by the need of reducing the number of experiments, cost, time, and physical efforts, design of experiment (DOE) is an important statistical and mathematical tool for evaluating which types of the designs would fit for the number of process parameter. The types of the design includes Doehlert Design (DD), Central Composite Design (CCD), Box-Behnken Design (BBD) and a three-level full factorial design [6]. However, central composite design (CCD) and Box-Behnken are found to be widely used optimization techniques for filtration process because of the advantage of optimizing multifactor problems with optimum number of experimental runs [6]. In order to improve the efficiency of a turbidity removal, it is expected that the operational parameters are operated at an optimum. The previous studies on optimization of turbidity removal from textile wastewater has been carried out mostly with the use of one factor at time (OFAT) approach, which does not consider the interactional effects on the response. The use of RSM for the optimization of both input variables either independently or in combination will be necessary. In addition, the modelling and optimization of the process parameter through experimental design approach for filtration treatment using nanocellulose filter paper that focused on the turbidity removal between two different design experiments, CCD and BBD has not been applied much in the others research literature. Therefore, this study employed this methodology for optimization of removal of turbidity from industrial dye wastewater and compared common response surface designs in order to investigate their advantages and limitations in the filtration treatment process.

2. Material and methodology

- Nanocellulose filter paper (*Neolamarckia cadamba*)

Nanocellulose can classified into three categories which are cellulose nanocrystals (CNC), cellulose nanofibers (CNF) and bacterial cellulose (BC). This study focuses on CNF due to its availability of cellulose and ability to form very thin membranes with nanoporous structure [7]. In addition, this research uses cellulose-based materials (Neolamarckia cadamba) as starting material in textile industrial dye wastewater treatment. Both of the starting materials are the most popular fast-growing wood species in the tropics which able to grow in diverse soil conditions and mostly free from serious pests and diseases that are suitable to use for pulp and small construction purposes [8].

- Design of experiments

Statistical approach based on RSM were applied in order to investigate the effect of three parameters in this experiment (initial temperature, pH and initial turbidity) with the dye effluent removal as the response. The selection of the parameters for the experimental designs is based on the literature review and scopes of study that were mentioned before. Optimum parameters for fast and efficient dye removal was determined by employing CCD and BBD experimental designs. The models were verified by Analysis of Variable (ANOVA) and the performance of the CCD and BBD models were statistically evaluated using a continuous error metric, such as the coefficient of determination (R^2), absolute average deviation (AAD), and root mean squared error (RMSE).

- Selection of independent variables and ranges

Parameters and their ranges were selected based on the existing literature in order to set the boundary conditions for both CCD and BBD models since in many studies, the range of investigated parameters for the filtration process is often not representative of the actual conditions in a WWTP [4]. Therefore, the initial turbidity (60-90 NTU), pH (4.0-7.0) and temperature (20.00 -40.00 °C) dosage were used as independent (input) variables and were investigated for their impact on the efficiencies of the turbidity removal from the dye industrial wastewater as shown in Table 1.

Table 1: Ranges of the investigated parameters

Parameter	Ranges
Initial turbidity (NTU)	60-90
pH	4.0-7.0
Initial temperature (ºC)	20-40

- Central Composite design and Box-Behnken design of experiments

The independent input variables that affect the turbidity removal were analyzed using Central Composite and Box-Behnken design with a total run of 20 and 15 experiments using MINITAB 18 Statistical Software. CCD adopts five levels (-α, -1, 0, +1, +α) designs while BBD adopts a three levels (-1, 0, +1) designs. The obtained results were analyzed by applying coefficient of determination (R2), analysis of variance (ANOVA), response contour plots and residuals plots. The levels of the chosen independent variables used in the CCD and BBD experiments are given in Table 2 and Table 3.

Table 2: CCD experimental ranges and levels of the independent variables

Independent variables	Factor code	Ranges and Levels				
		-α	-1	0	+1	+α
Initial turbidity (NTU)	X_1	60.0	66.0	75.0	84.0	90.0
pH	X_2	4.0	4.6	5.5	6.4	7.0
Initial temperature (ºC)	X_3	20.0	24.1	30.0	36.0	40.0

Table 3: BBD experimental ranges and levels of the independent variables

Independent variables	Factor code	Ranges and Levels		
		-1	0	+1
Initial turbidity (NTU)	X_1	60.0	75.0	90.0
pH	X_2	4.0	5.5	7.0
Initial temperature (ºC)	X_3	20.0	30.0	40.0

- Response Surface Methodology (RSM)

RSM is a collection of mathematical and statistical technique that are useful for modelling and analysis of problem in which a response of interest is influenced by several variable. RSM was used in this research to create an experiment design that allows achieving the optimal operating conditions. The data were obtained from MINITAB 18 statistical software where it offers full regression method to analyze responses and it was used to fit the mathematical models of the experimental data. The predicted percentage of the removal of dye effluents is explained by the following quadratic equation (1):

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

Where, Y is the predicted response, xi and xj are the input variables, βo is the intercept term, βi is the coefficient of linear effect, βii is the coefficient of squared effect, βij is the coefficient of interaction effect and ε is the random error.

Results for the percentage of the turbidity removal were obtained by performing batch experiments according to the CCD and BBD model of conditions. The percent of dye effluent removal of both CCD and BBD models were taken as a response (y) of the experimental design and calculated as (2):

$$\text{Removal efficiency, y (\%)} = \left(\frac{C_i - C_f}{C_i} \right) \text{x } 100\% \quad \textit{Eq. 2}$$

Where y is the experimental removal efficiency, Ci is the initial turbidity while Cf is the final turbidity obtained after the filtration process [9].

A quadratic polynomial equation of CCD and BBD models that describes the behavior of turbidity removal to optimize the process can be determined from the coefficients of all the significant factors. The obtained quadratic equation in term of coded variables is shown below in (3) and (4):

$$\begin{aligned} \text{CCD} \quad Y (\%) = \; & 99.2196 - 0.1007{*}X_1 + 0.1694{*}X_2 - 0.0488{*}X_3 - 0.1234{*}(X_1{*}X_1) \\ & - 0.2484{*}(X_2{*}X_2) + 0.0748{*}(X_3{*}X_3) + 0.003{*}(X_1{*}X_2) \\ & - 0.026{*}(X_1{*}X_3) - 0.032{*}(X_2{*}X_3) \quad \textit{Eq. 3} \end{aligned}$$

$$\begin{aligned} \text{BBD} \quad Y (\%) = \; & 99.6567 - 0.0575{*}X_1 + 0.0463{*}X_2 + 0.0313{*}X_3 - 0.2021{*}(X_1{*}X_1) \\ & - 0.2196{*}(X_2{*}X_2) - 0.1546{*}(X_3{*}X_3) + 0.0{*}(X_1{*}X_2) \\ & + 0.0350{*}(X_1{*}X_3) + 0.0775{*}(X_2{*}X_3) \quad \textit{Eq. 4} \end{aligned}$$

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

3. Results and Discussion

The results of dye effluent removal percentage in each case are summarized in Table 4.

Data analysis fitting for Response

Coefficient of determination (R2) is a measurement on how close the collected data are to the fitted regression line which simply describes how well the model fits the data. Meanwhile, adjusted coefficient of determination (Adj. R2) is an adjustment for R2 to include the number of variables in a data set while the predicted R2 (Pred. R2) is a measure of the level at which the fitted model predicts a response value [10]. The CCD analysis shows a R2 value of 73.42 % while BBD is 90.95 %. This indicates that 73.42 % of the total variation of CCD model and 90.95 % of BBD model were explained by the model and the remaining variation of both models is unexplained. The Adj. R2 of CCD model is determined to be 49.50 % while BBD model is 74.66 %. The obtained R2 values of both models were relatively high than their Adj. R2 and Pred. R2. The Adj. R2 and Pred. R2 values suggested that the models were unacceptable. The lower Adj. R2 values indicate that high R2 values might have come from the forced improvement of the models achieved by addition of many terms, whereas the true value of the model is low, that is, the models are over-fitted. The Pred. R2 a low values indicating the prediction ability of these models for experimental space navigation was compromised and they cannot be used [10]. Table 3 below shows the model summary of CCD and BBD.

Table 3: Model summary of both CCD and BBD

Model	R^2	R^2 (adj.)	R^2 (pred.)
CCD	73.42 %	49.50 %	0.00 %
BBD	90.95 %	74.66 %	0.00 %

The model was assessed for its suitability by examining the lack of fit through ANOVA presented in Table 5 and the significance of each coefficient was determined by the p-values and the F-values. The smaller the p-values and the larger the F-value, the more significant is the corresponding coefficients [11]. At a confidence level of 95.00 %, the p-value of Lack of Fit for CCD and BBD models is 0.011 and 0.143, which shows that the Lack of Fit of CCD model is significant as their $p < 0.05$ while BBD model is insignificant, indicating that the quadratic models developed by BBD model does fit the data for the response to effectively predict the removal of turbidity from the dye industrial wastewater.

Table 4: The experimentally obtained percentage of dye effluent removal by the Central Composite design (CCD) and Box-Behnken design (BBD)

Run Number	Coded values			Actual values			Dye effluent removal efficiency (%)	
	X_1	X_2	X_3	X_1	X_2	X_3	Experimental	Predicted
Central Composite Design								
1	-1	-1	-1	66	4.6	24	99.17	98.75
2	+1	-1	-1	84	4.6	24	98.94	98.59
3	-1	+1	-1	66	6.4	24	99.34	99.15
4	+1	+1	-1	84	6.4	24	99.17	99.00
5	-1	-1	+1	66	4.6	36	99.21	98.96
6	+1	-1	+1	84	4.6	36	99	98.70
7	-1	+1	+1	66	6.4	36	99.39	99.23
8	+1	+1	+1	84	6.4	36	99.13	98.99
9	$-\alpha$	0	0	60	5.5	30	99	99.04
10	$+\alpha$	0	0	90	5.5	30	99.03	98.70
11	0	$-\alpha$	0	75	4.0	30	98.69	98.23
12	0	$+\alpha$	0	75	7.0	30	99.09	98.80
13	0	0	$+\alpha$	75	5.5	40	99.13	99.35

14	0	0	-α	75	5.5	20	99.3	99.51
15	0	0	0	75	5.5	30	99.22	99.22
16	0	0	0	75	5.5	30	99.24	99.22
17	0	0	0	75	5.5	30	99.3	99.22
18	0	0	0	75	5.5	30	99.22	99.22
19	0	0	0	75	5.5	30	99.19	99.22
20	0	0	0	75	5.5	30	99.18	99.22
Box-Behnken Design								
1	-1	-1	0	60	4.0	30	99.23	99.29
2	+1	-1	0	90	4.0	30	99.24	99.33
3	-1	+1	0	60	7.0	30	99.23	99.31
4	+1	+1	0	90	7.0	30	99.24	99.50
5	-1	0	-1	60	5.5	20	99.19	99.37
6	+1	0	-1	90	5.5	20	99.34	99.48
7	-1	0	+1	60	5.5	40	99.19	99.43
8	+1	0	+1	90	5.5	40	99.48	99.54
9	0	-1	-1	75	4.0	20	99.24	99.56
10	1	+1	-1	75	7.0	20	99.27	99.50
11	1	-1	+1	75	4.0	40	99.14	99.47
12	1	+1	+1	75	7.0	40	99.48	99.71
13	1	0	0	75	5.5	30	99.68	99.66
14	1	0	0	75	5.5	30	99.6	99.66
15	1	0	0	75	5.5	30	99.69	99.66

X_1 = Initial turbidity (NTU); X_2 = pH; X_3 = Initial temperature (°C).

Table 5: ANOVA of CCD and BBD

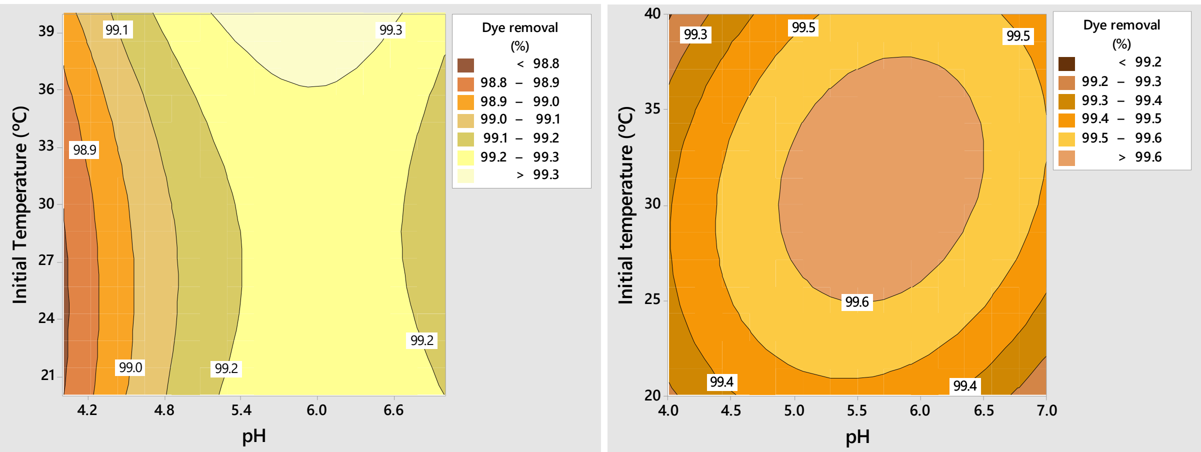
Source	DF	Adjusted Sum of Square	Adjusted Mean Square	F-Value	p-Value	
ANOVA Results and Adequacy of the Quadratic Models for turbidity removal Using CCD						
Model	9	0.354485	0.039387	3.07	0.048	Significant
Linear	3					
X_1	1	0.049527	0.049527	3.86	0.078	
X_2	1	0.139960	0.139960	10.91	0.008	
X_3	1	0.011484	0.011484	0.89	0.366	
Square	3					
X_1* X_1	1	0.027688	0.027688	2.16	0.173	
X_2* X_2	1	0.112148	0.112148	8.74	0.014	
X_3* X_3	1	0.010110	0.010110	0.79	0.396	
2-Way Interaction	3					
0000PPPPPPP	1	0.000012	0.000012	0.00	0.976	
X_1* X_3	1	0.000671	0.000671	0.05	0.824	
X_2* X_3	1	0.001032	0.001032	0.08	0.783	
Error	10	0.128335	0.012834			
Lack-of-Fit	5	0.119185	0.023837	13.03	0.007	Significant
Pure Error	5	0.009150	0.001830			
Total	19	0.482820				
ANOVA Results and Adequacy of the Quadratic Models for turbidity removal Using BBD						
Model	9	0.443552	0.049284	5.58	0.036	Significant
Linear	3					

Y_1	1	0.026450	0.026450	3.00	0.144	
Y_2	1	0.017113	0.017113	1.94	0.223	
Y_3	1	0.007813	0.007813	0.88	0.390	
Square	3					
Y_1* Y_1	1	0.150785	0.150785	17.08	0.009	
Y_2* Y_2	1	0.178031	0.178031	20.17	0.006	
Y_3* Y_3	1	0.088231	0.088231	9.99	0.025	
2-Way Interaction	3					
Y_1* Y_2	1	0.000000	0.000000	0.00	1.000	
Y_1* Y_3	1	0.004900	0.004900	0.56	0.490	
Y_2* Y_3	1	0.024025	0.024025	2.72	0.160	
Error	5	0.044142	0.008828			
Lack-of-Fit	3	0.039275	0.013092	5.38	0.161	Insignificant
Pure Error	2	0.004867	0.002433			
Total	14	0.487693				

*DF = degree of freedom

Model analysis fitting for Responses

For better graphical interpretation of the turbidity filtration process, two-dimensional response contour plots were generated to see how fitted response values relate to two continuous variables based on a model equation. Figure 1 shows the effect of the investigated parameters on the removal of turbidity, with one of the three parameters held constant at its intermediate value (75 NTU, 5.5 or 30.00 °C). In these plots, the response is represented as a function of two factors. When more than two factors are studied, the other factors that are not plotted must be set at a constant value. Therefore, only a limited part of the experimental domain is shown, which leads to the difficult establishment of optimal operating condition [12]. In addition, the plots depict the sensitivity of the responses due to the change of factor levels with the degree of their interactions and the contours are curved because the both of the models contains quadratic terms that are statistically significant [13]. Figure 1 (A) illustrated the optimum turbidity removal at initial temperature of 36.00 ºC and pH of 6.4 using CCD model while 30.00 ºC and 5.5 for BBD. The turbidity removal percentage of BBD model is higher than CCD due to the electrical repulsive force of its positive charge and the attraction between the membrane and dye molecules [13].



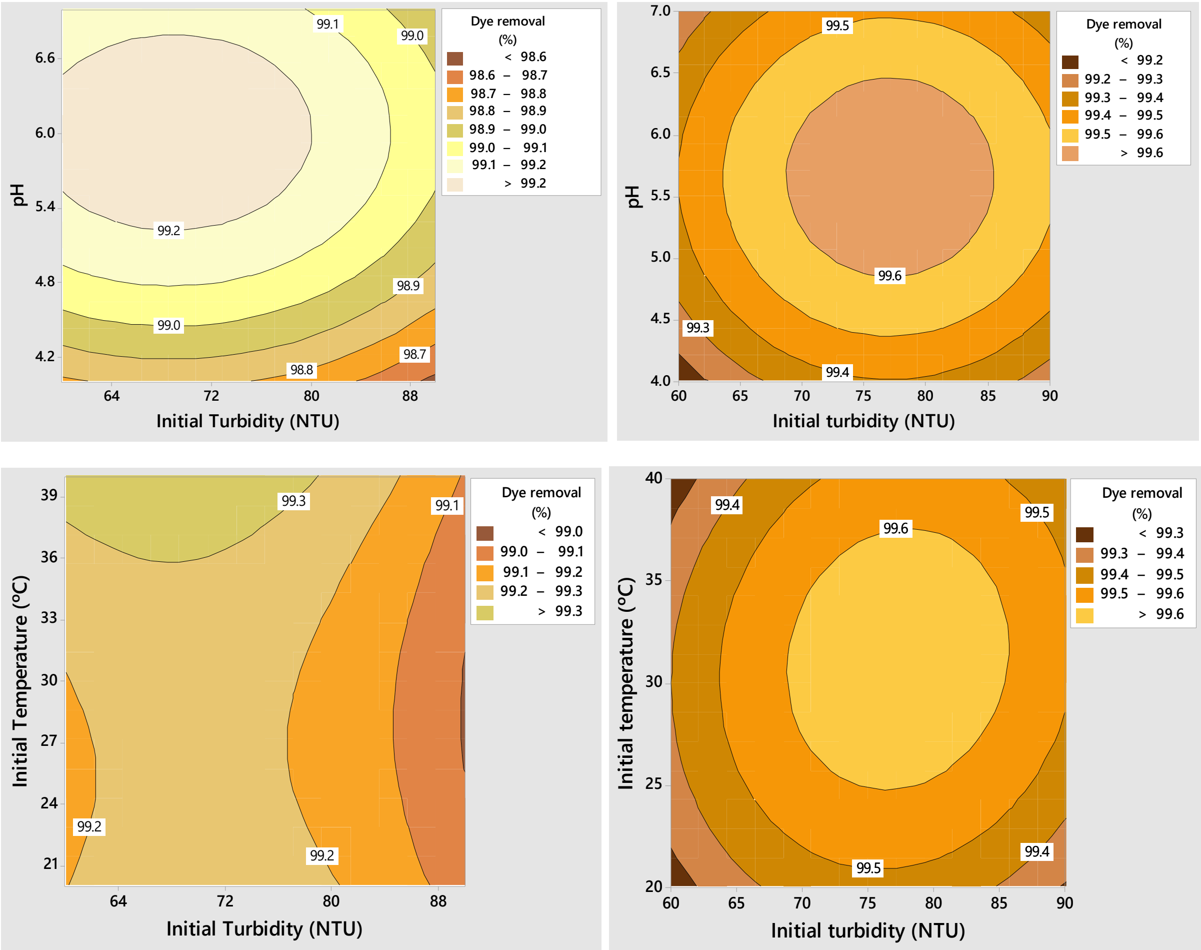


Figure 1: Contour plots of CCD (left) and BBD (right) showing the effects of filtration parameters on the dye effluent removal, with initial turbidity held constant at 75NTU (A), pH held constant 5.5 (B) and initial temperature held constant at 30 ºC (C)

BBD model shows an increased at weak acidic range and decreased at extremely basic or acidic conditions which due to the fact that lower pH is favourable for removal of anionic dyes, but extremely basic or acidic conditions gave poor removal results. Thus, pH was found to be one of the main parameters affecting the filtration process.

Figure 1 (B) depicts the response surface plots at initial turbidity of 66NTU and pH of 6.4 for CCD model and 75 NTU and pH of 5.5 for BBD model. Therefore, it shows that the removal of the turbidity increases along with the pH values which there is tendency towards sedimentation. While in Figure 1 (C) shows the optimum dye effluent removal of CCD model at initial turbidity of 66NTU and initial temperature of 36.00 ºC whilst 75NTU and 30.00 ºC for BBD model. The temperature of CCD model that is higher than BBD model leads to an increase in the pore size of the membrane and thus allows the passage of pollutants through it. At high temperature, the water viscosity decreases due to the weakened cohesive forces and thus, the filtration rate inversely proportional to fluid viscosity [14].

Normal Probability plots

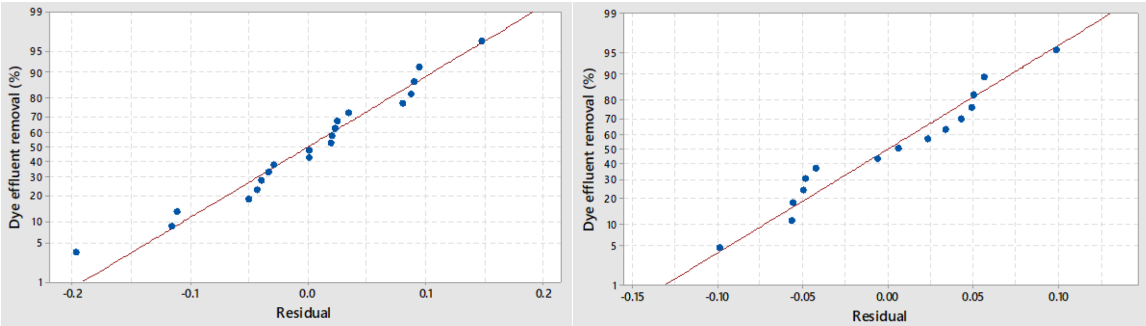


Figure 2: Normal probability plot of dye effluent removal for CCD (left) and BBD (right)

The normal plot of residuals of the turbidity removal percentage for CCD and BBD models is plotted in Figure 2. From Figure 2, it can be seen that the residuals for CCD model are plotted around the straight line which indicates that the residuals are normally distributed. Similarly, residuals for BBD model can be observed which are plotted approximately along the straight line as CCD model. The data in the normal probability plots of both models did not show strong deviation from a straight line, demonstrating a normal distribution. Hence, it can be said that the normality assumption is satisfied for the turbidity removal in this study [15].

Statistical comparison and performance of CCD and BBD for dye effluent

The performance of the built CCD and BBD models were compared and statistically measured by the coefficient of determination (R2), absolute average deviation (AAD), and root mean squared error (RMSE). AAD is a summary statistical dispersion or variability while RMSE is a standard way to measure the error of a model in predicting quantitative data. Table 6 shows the statistical comparison and performance of BBD and CCD models for dye effluent removal.

Table 6: Obtained coefficients for mathematical models after application of BBD and CCD models

Response	BBD		CCD	
	Coefficient	p-value	Coefficient	p-value
X_0	99.6567		99.2196	
X_1	0.0575	0.144	-0.1007	0.078
X_2	0.0463	0.223	0.1694	0.008
X_3	0.0313	0.390	0.0488	0.366
$X_1* X_1$	-0.2021	0.009	-0.1234	0.173
$X_2* X_2$	-0.2196	0.006	-0.2484	0.014
$X_3* X_3$	-0.1546	0.025	0.0748	0.396
$X_1* X_2$	0.0000	1.000	0.003	0.976
$X_1* X_3$	0.0350	0.490	-0.026	0.824
$X_2* X_3$	0.0775	0.160	-0.032	0.783
R^2	0.9095		0.7342	
Adjusted R^2	0.7466		0.4950	
AAD (%)	0.12		0.13	
RMSE	0.58		0.53	

Among the compared quadratic models, BBD model had the best values of R2 and adjusted R2 rather than the CCD model. The CCD model led to the optimal initial temperature of 36.00 °C although the quadratic BBD model pointed out the initial temperature of 30.00 °C as the optimal one. The AAD and RMSE for the CCD model was calculated to be 0.13 % and 0.53 respectively, while for BBD model was 0.12 % and 0.58 respectively. Besides that, the CCD models have smaller RMSE than the BBD models indicating that CCD model has better fit than BBD model. On the other hand, based on the results of the AAD, BBD model gave the smallest value than CCD model which indicates less variability were spread out. This made the BBD to be more superior over the CCD.

4. Conclusion

This study was planned to compare two optimization techniques such as central composite design and Box-Behnken design with three parameters which is turbidity, pH and temperature. At optimized conditions, the filtration process would be able to achieve 99.39 % turbidity removal efficiency for CCD model and 99.69 % for BBD model, respectively. RSM is an effective and economically viable alternative technique that can be adapted for optimizing various wastewater treatment processes to favorably maximize the output. The optimum conditions for the filtration process of CCD model on dye removal are initial turbidity of 66 NTU, pH of 6.4 and initial temperature of 36.00 ºC while BBD is 75 NTU for initial turbidity, pH of 5.5 and initial temperature of 30.00 ºC. Both designs led to almost the same optimal process conditions. This study showed that BBD model predicts better turbidity removal closer to the actual values than CCD model.

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