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Optimization of Batch Conditions for COD and Ammonia Nitrogen Removal Using cockle shells Through Response Surface Methodology

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Abstract: The optimal conditions for the reduction of COD and NH_3-N using cockle shells (CS) from a stabilised landfill effluent were analyzed. The influence of two variables (adsorbent dosage and pH) were analysed through the application of response surface methodology (RSM) and central composite design (CCD). Quadratic models were developed for the removals of COD and NH_3-N parameters. The optimum conditions for removal of 65.6% and 53.6% for COD and NH_3-N respectively was achieved at pH 6.34, adsorbent dosage of 20.21 g having 0.888 desirability value. The model F-value obtained for NH_3-N removal Prob. > F value of 0.0001 with F-value of 104.21 was obtained. Similarly the Prob. > F value of < 0.0001 for COD with F-value of 82.74 was obtained, these P-values confirmed the significance of the model. The predicted response versus the experimental response depicted that the experimental data were relatively close to the predicted data. Thus, the generated models significantly enclosed the correlation between the process variables and the response.

Keywords: Cockle shells, Response surface methodology, Central composite design, COD and NH_3-N.

1. Introduction

Water deficiency and contamination currently rank evenly with climate change as the most pressing environmental perturbation [1-8]. Due to increasing globalization and urbanization, which intensify ecological damage and severely endanger human health and the environment, adsorption, especially for the use of activated carbons and zeolites has been the focus of numerous studies and it is extensively applied to wastewater management [9-11]. And with the cost of conventional media reaching as high as S$602 per metric ton in the global market and the demand increasing constantly to an unprecedented degree (800,000 tons per year), the search for an economical and environmentally friendly substitute adsorbent is inevitable [1]. Agricultural waste biomass is currently a topic of interest that has been receiving considerable attention in the past few decades [12]. In recent years, different native and low-cost materials, such as peat soil, feldspar, cockle shell (CS), seaweed, rice husk, wood sawdust, banana frond, and limestone, have been considered successful alternatives for wastewater treatment [13]. Several operational parameters, such as pH, dosage, and shaking speed, mainly influence the minimization of chemical oxygen demand (COD) and ammonia nitrogen (NH_3–N).

RSM is a compilation of statistical and mathematical procedures critical to the realization, enhancement, and optimization of systems. It also helps in evaluating the influence of independent variables and gives rise to mathematical models related to chemical or biochemical systems. The primary advantage of RSM is the minimal amount of experimentation required to assess multiple parameters and their interactions [14-15]. Therefore, RSM is less strenuous compared with other approaches, such as "one factor at a time," which is lengthy and disregards the interaction between variables [16-17]. The present study aims to optimize the operating reaction conditions for the removal of COD (Y_{COD}) and that of NH_3–N (Y_{NH3-N}) by using response surface methodology (RSM) via central composite design (CCD). RSM

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performs several assays to determine the optimum experimental conditions. Y_{COD} and $Y_{NH3–N}$ were monitored throughout the experiments. Accordingly, the statistical design focused on two independent factors (i.e., media dosage and pH) and the dependent variables or responses (i.e., COD and NH_3–N).

2. Materials and method

2.1 Leachate sampling

The leachate sample was procured from Simpang Renggam municipal landfill located at $N1^0$ 53 41.64 latitude and $E1^030$ 22 34.68 longitude in Kluang District of Johor. Sterilized 20-L high-density polyethylene bottles was utilised to manually collect the leachate at site in accordance to the method outlined by [14]. Moreover, the leachate was characterized within 24 h according to the standard methods for the examination of water and wastewater [18].

2.2 Preparation of Adsorbent media

Cockle shells (CS) were obtained from commercial restaurants in Batu pahat area of Johor. The preparation is carried out according to the procedure outlined by [19,20] and crushed sorted, sieved to obtain particle between sizes of 0.6 -1.18 mm [3].

2.3 Adsorption experiments

Adsorption experiments were carried out by shaking the media in 250 mL conical flask containing 100mL sample of raw leachate solution of desired pH in an orbital shaker at 150 rpm. The experiments were carried out varying cockle shell dosage and pH. The samples were taken from the flasks at predetermined time intervals and analyzed for COD and NH_3-N content [21,22]. The COD and NH_3-N were assessed by the closed reflux and Nessler Method respectively using atomic adsorption spectrophotometer (Model HACH DR6000). The effect of pH was studied by adjusting the pH of the leachate solutions using HCl and NaOH [23,24]. Experimental results were calculated as percentage removal of COD and NH_3-N according to equation (1).

$$\% \, Removal = [[(C_i - C_f)/C_t] x \, 100 \quad (1)$$

Where C_i and C_f are the initial and final COD(mg/L) and NH_3-N(mg/L) concentration respectively.

2.4 CCD-based experimental design.

Design Expert (Version 7.0; Stat-Ease, Inc., Minneapolis, MN 55413, USA) software was employed to design the experiments and analyze the data. CCD and RSM were used to optimize two independent variables, namely, pH and dosage. The ranges for variable selection were 4–56 g/L for dosage and 4–8 for pH. To attain the optimum dosage and pH, two dependent variables were investigated as responses, namely, COD and NH_3–N. The quadratic equation model for predicting the optimum conditions can be expressed according to Equation (2):

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i . X_i + \sum_{i=1}^{k} \beta_{ii} . X_i^2 + \sum_{t_i \leq j}^{k} \sum_{j}^{k} \beta_{ij} . X_i . X_j + \cdots + e \quad (2)$$

where *i* represents the linear coefficient, *j* the quadratic coefficient, β as the coefficient of regression, *k* is for the quantity of factors investigated and optimized by the experiment while *e* stands for the random error [25].
Analysis of variance (ANOVA) was incorporated in graphical analyses of the obtained data to determine the interactions between the system variables and their responses. The coefficient of determination R^2 determined the nature and suitability of the derived polynomial model, and its significance was measured by F-test within the program [26]. Model reliability was assessed by using a P-value with a 95% confidence limit [27]. Three-dimensional (3D) graphs and their contour graphs were created to illustrate the interactions on the basis of the effects of dosage and pH. The optimum zone was also established on the basis of the paramount variables from the graphs.

3. Results and Discussion

Table 1 presents the physicochemical quality of the CS adsorbent obtained using an XRF machine (Bruker S4 pioneer model) before and after treatment. The shells contained high amount of CaO and which makes it suitable for application in wastewater treatments. The results also indicate that several changes occurred in the percentage composition of several compounds for the pretreatment and post-treatment media composition, which is probably due to redox reaction that occurred due to adsorbent – adsorbate interaction..

Table 1 Chemical composition of CS before and after treatment.

Formula	Concentration before treatment (%)	Concentration after treatment (%)
CaO	70.70	69.70
SiO_2	0.22	0.45
Na_2	0.49	0.33
SrO	0.15	0.15
S	0 < LLD	0 < LLD
C	0.10	0.10

3.1 CCD

In this study, two salient factors, namely adsorbent dose (A) and pH (B), were selected as independent variables. The software with the application of multiple regression analysis technique proposed a quadratic model; Y_{COD} and $Y_{NH3–N}$ for COD and NH_3-N respectively, the models were selected on the basis of the highest-order polynomials [20]. Y_{COD} and $Y_{NH3–N}$ at

steady condition were selected as the dependent variables (response). Tables 2 and 3 show the empirical design array derived and the percentage removals, respectively. The percentage removals ranged from 36.86% to 71.82% and 27.32% to 54.95% for COD and NH_3–N, respectively. The quadratic model for Y_{COD} and $Y_{NH3–N}$ is expressed as follows:

$$Y_{COD} = 61.23 - 12.38*A - 1.69*B - 11.34*A*B - 3.44*A^2 - 8.31*B^2 \quad (3)$$

$$Y_{NH3-N} = 54.66 - 1.48*A + 1.21*B - 1.99*A*B - 12.52*A^2 - 5.41*B^2 \quad (4)$$

The positive or negative symbol before the expression determines the positive or negative effect of the individual or multiple coefficient factor interaction on Y_{COD} and $Y_{NH3–N}$ [15]. Positivity and negativity imply favorable and unfavorable interactions, respectively, in the remediation [17]. The occurrence of a single factor signifies a singular effect, the presence of two variables is indicative of a paired effect, and the occurrence of a second-order expression symbolizes quadratic influence [25].

Table 2 Variables and levels studied for percentage of removal of COD and NH_3-N

Factor	Name	Units	Low Level	High Level
A	dose	g	4	56
B	pH	-	4	8

Table 3 CCD of two variables and their responses.

Run	Factor A: Dose	Factor B: pH	Response (removal %)	
			COD	NH_3-N
1	-1.00	-1.00	50.12	35.55
2	1.00	-1.00	48.96	37.13
3	-1.00	1.00	71.82	40.12
4	1.00	1.00	25.28	33.74
5	-0.893	0.00	71.65	46.92
6	1.414	0.00	36.86	27.32
7	0.00	-1.414	49.13	40.92
8	0.00	1.414	40.98	46.94
9	0.00	0.00	60.94	54.95
10	0.00	0.00	64.12	52.71
11	0.00	0.00	61.95	56.21
12	0.00	0.00	58.73	54.13
13	0.00	0.00	59.15	54.72

3.2 Statistical analysis

3.2.1 ANOVA

Tables 4 and 5 present the outcomes of the surface quadratic model for Y_{COD} and $Y_{NH3–N}$ obtained through ANOVA, respectively. ANOVA is necessary to substantiate the significance and adequacy of the models. The model's F-value of the ANOVA implies the ratio of insignificance. Tables 4 and 5 show the ANOVA of Y_{COD} and $Y_{NH3–N}$, respectively. With reference to Y_{COD} percentage (Table 4), the obtained F-value of the model was 82.74 with the Prob. > F-value of < 0.0001, which confirms the model's significance. Model terms A, AB, A^2, and B^2 were significant to the response in contrast with B, which was insignificant. The lack-of-fit F-value of 1.26 indicates that the lack of fit is insignificant. Lack of fit was insignificant relative to the pure error. A lack-of-fit F-value this large could occur at a probability of 40.08% due to noise.

An insignificant lack of fit is desirable. As shown in Table 5, a Prob. > F-value of 0.0001 and an F-value of 104.21 were also obtained for NH_3–N, which indicates the model's significance. At this point, the terms A, B, AB, A^2, and B^2 were all significant. Correspondingly, a lack-of-fit F-value of 1.65 implies that the lack of fit is insignificant relative to the pure error. A lack-of-fit F-value this large could occur at a probability of 31.23% due to noise [26]. The results of statistical analyses reveal that the models are appropriate with respect to the prediction of Y_{COD} and $Y_{NH3–N}$ within the extent of the considered variables.

The merit of the models derived was determined on the basis of the coefficient of determination R^2. The R^2 values for COD and NH_3–N were 0.9834 and 0.9867, respectively (Tables 4 and 5, respectively). The "Pred R-Squared" value for COD (0.9181) was consistent with the "Adj R-Squared" value (0.9715), which implies that the model can describe a 97.15% Y_{COD}. "Adeq Precision" quantifies the signal-to-noise ratio [16]. An Adeq Precision value of more than 4 is preferable. A 30.249 ratio in this case indicates an adequate signal. The Pred R-Squared value for YNH_3–N was 0.9227 and is in reasonable agreement with the Adj R-Squared value of 0.9773 (Table 5). The model can describe the removal on the basis of the Adj R-Squared value. Furthermore, the Adeq Precision value was greater than 4, which is desirable. A 27.687 ratio indicates an adequate signal. This model can be used to navigate the design space.

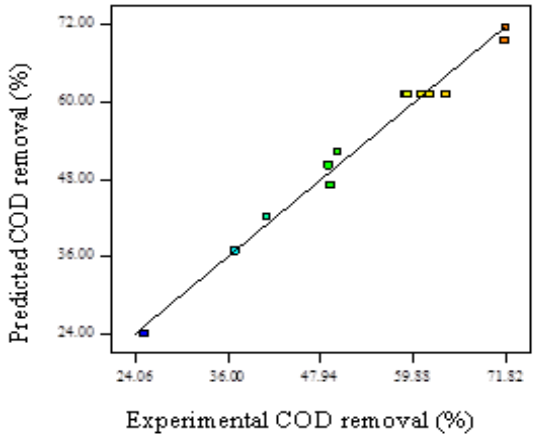
Table 4 ANOVA for Response Surface Quadratic Model of COD

Source	Sum of Squares	Degree of freedom	Mean Square	F Value	P-Value Prob > F
Model	2206.32	5	441.26	82.74	< 0.0001
A-dose	935.51	1	935.51	175.41	< 0.0001
B-pH	22.8	1	22.8	4.28	0.0775
AB	514.84	1	514.84	96.53	< 0.0001
A^2	54.33	1	54.33	10.19	0.0152
B^2	487.02	1	487.02	91.32	< 0.0001
Residual	37.33	7	5.33		
Lack of Fit	18.12	3	6.04	1.26	0.4008
R-Squared		0.9834			
Adj R-Squared		0.9715			
Pred R-Squared		0.9181			
Adeq Precision		30.249			
R-Squared		0.9834			

Table 5 ANOVA for Response Surface Quadratic Model of NH_3-N

Source	Sum of Squares	Degree of freedom	Mean Square	F Value	P-Value Prob > F
Model	1084.87	5	216.97	104.21	< 0.0001
A-dose	13.35	1	13.35	6.41	0.0391
B-pH	11.75	1	11.75	5.64	0.0492
AB	15.84	1	15.84	7.61	0.0282
A^2	718.37	1	718.37	345.04	< 0.0001
B^2	206.48	1	206.48	99.18	< 0.0001
Residual	14.57	7	2.08		
Lack of Fit	8.07	3	2.69	1.65	0.3123
R-Squared		0.9867			
Adj R-Squared		0.9773			
Pred R-Squared		0.9227			
Adeq Precision		27.687			
R-Squared		0.9867			

This model can be used to navigate the design space. Furthermore, Figs. 1 shows the predicted versus the experimental values representing Y_{COD} and YNH_3–N, respectively. The values indicate that the experimental values were relatively close to the predicted values. Thus, the generated models sufficiently captured the correlation between the response and the adsorption variables.



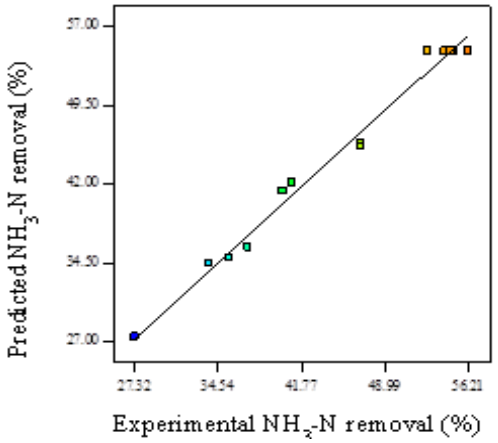
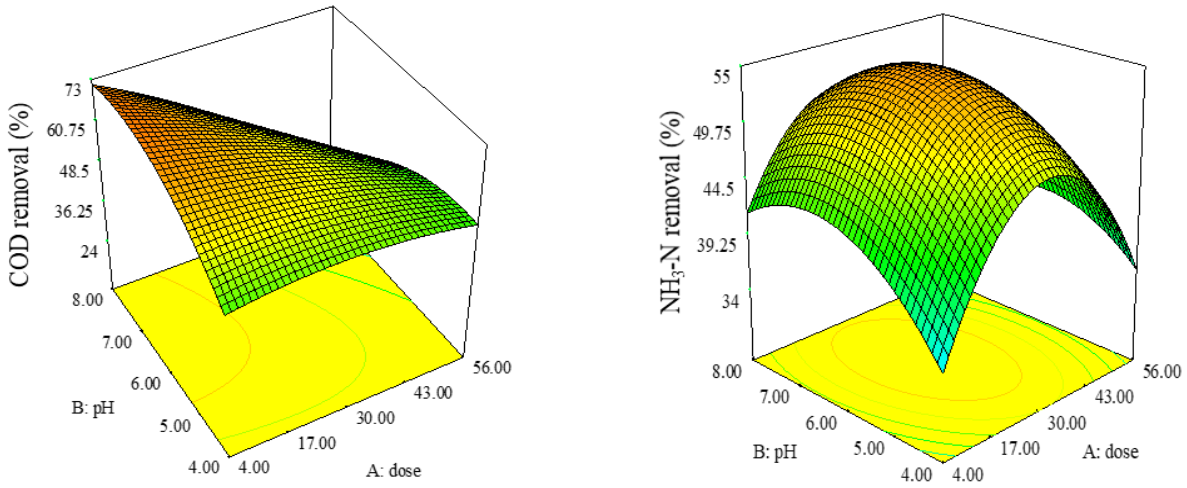


Fig.1 Predicted vs. Experimental values plot for (a) COD and (b) NH_3-N removal



Figs. 2 Response surface plots for the COD and NH_3–N parameters removal

Figs. 2 presents the 3D response surface plots for the Y_{COD} and $Y_{NH3–N}$ parameters, respectively. The plots illustrate the effect of pH–dosage interaction in the removal. The two figures show that the Y_{COD} was better than the $Y_{NH3–N}$. Daud [19] and Moideen [14] had observed similar behavior in their study.

3.2.2 Optimization

The COD and NH_3–N decontamination by CS were optimized by using Design Expert software. Graphical optimization presents the region of ideal response values of the factor scope with the area that suits the optimization criteria that have been identified at the peak. The ANOVA evaluation emphasizes the models significance for the removal (%) of COD and ammonia nitrogen with Prob > F values of < 0.0001 Table 4 and 5. Furthermore, the Prob > F of greater than 0.10 signifies the statistical inadequacy of the correlation among the process responses and variables [28-38]. Notwithstanding, some of the models' terms are have limited influence on the removal of COD and ammonia based on the F values. Through the optimization, the baseline for the two response values was set to maximum values. The ideal adsorption values obtained were pH 6.34 and dosage of 20.21 g. These configurations yield approximately 65.6% COD, 53.6% NH3–N, and a desirability value of 0.888.

Table 6 Numerical optimization solution

Number	Dose (g)	pH	COD (%)	NH_3-N (%)	Desirability
1	20.21	6.34	65.605	53.615	0.888

Conclusion

Two adsorption criteria (adsorbent dosage and pH) were optimized by using CCD for the minimization of COD and NH_3–N by CS. The optimized removal proportions were 65.6% and 53.6% for the COD and NH_3-N respectively. The maximum removal proportion of the adsorbate was achieved at the optimum adsorbent dosage of 20.21g, pH 6.34, and desirability value of 0.888. Based on the evaluated adsorbent performance, the CS prove to be suitable for application in wastewater industries.

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