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The Efficiency of Bamboo Activated Carbon for Removal of COD from Fish Cracker Industry Wastewater via Response Surface Methodology (RSM)

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Abstract. Pre-treatment for wastewater in industry were utilizing activated carbon are gradually recognized by the world. Hence, the objective of this study is to determine the performance of chemically activated carbon prepared from bamboo in removing Chemical Oxygen Demand (COD) from wastewater collected from fish cracker industry. The effects of initial concentration (10 % - 50 %), carbon dosage (0.1 g - 1.0 g), and contact time (3 h - 12 h) were examined. The adsorption experimental design was performed using the standard Response Surface Methodology design which was Box-Benhken design via Design-Expert software (version 7.0 Stat Ease, trial version). By increasing carbon dosage and lower initial concentration, the removal efficiency increased. According to Box-Benhken results, the activated carbon with zinc chloride activation was fitted to a quadratic equation and statistically significant. The R^2 for the model was 0.9780 and the adjusted R^2 was 0.9498. The optimum conditions suggested by the model for the process variable were 49.27 mg/l, 9.49 hours and 0.932 g for initial concentration, contact time and carbon dosage, respectively. The maximum removal obtained at these conditions was 70.579%.

1. Introduction

Food industry discharged huge load of wastewater into the environment. These load of consumed water were used in kitchen cleaning, material preparations and generally contain high organic concentration [1]. These lead to high biological and chemical oxygen demand value.

There were numbers of waste water abatement technology in the market, including precipitation, ion exchange, reverse osmosis and adsorption [2]. Adsorption is a process where an adsorbate attracted to surface of an adsorbent with Van der Waal's force [3]. Generally, activated carbon recognized for its high internal surface area for effective adsorption capacity is widely used as adsorbent. Despite expensive cost on production of activated carbon due to its raw material, research nowadays focus on producing activated carbon from agriculture residual to solve the cost issue as well as solution to waste abundance in nature [4]. In the case of food industry, utilization of adsorption process as pre-treatment stage in reducing the COD level of the effluent were proved to be effective. The simple operation design and cost effective option possessed made adsorption a wise decision.

RSM is a combination of mathematical and statistical techniques that could be used to approximate and optimize a system from several responses and different types of experimental runs [5]. Box-Benhken design is a popular experimental design in RSM that were used in may research work. The design is able to reduce the number of experimental trials required to study interactions of multiple parameters and responses [6].

In this study, application of Box-Benhken Design in Response Surface Methodology was used to perform an experimental design to investigate removal of COD using activated carbon prepared from bamboo. The effects of operating parameters such as initial concentration, contact time, and carbon dosage on the COD removal were analyzed.

2. Material and Methodology

2.1 Preparation of Activated Carbon

Old bamboos were collected from Agropark in UMK Jeli Campus. The old bamboo were cut into smaller pieces using machine cutter. First of all, the materials were washed with water to remove any physical impurities and were dried under the sun. The bamboo was further dried in an oven for a day at 105 $^\circ$C to ensure removal of moisture content on the materials. Next, the old bamboo was ground with a grinder. The ground bamboo was sieved to 1 mm - 2 mm particles size using a siever. The end material were kept in desiccator for next activation step. The bamboo were soaked with $ZnCl_2$ which act as activating agent in this chemical activation process in 1:3 ratio (material:activating agent). Next, the impregnated samples were carbonized in a furnace at 600 $^\circ$C with holding time of an hour. After the carbonization was completed, the activated carbons were washed using distilled water to remove excessive activating agent present on the carbons surface. Lastly, the activated carbons were dried in oven overnight at 105 $^\circ$C and were kept in desiccator.

2.2 Wastewater preparation

The wastewater were collected from fish cracker industry in Tumpat, Kelantan. The wastewater was characterized for pH, BOD, COD, An, TSS and the result was shown in Table 1. Characterization of COD for the wastewater was carried out using HACH Method 8000, USEPA Reactor Digestion Method (HR) with DR 6000 Spectrophotometer. Prior to the experiment, the wastewater was diluted to three different concentrations (10%, 30%, 50%) for the initial concentration parameter using dilution series. In this study, utilization of concentration in terms of percentage is justified by feasibility to dilute the strength of the wastewater as the wastewater do not have discrete numbers in term of ppm or mg/l unit.

Table 1. Characterization of wastewater

Parameter	Unit	Wastewater
BOD	mg/l	1384.5
COD	mg/l	9612
TSS	mg/l	1018
pH	-	6.71
AN	mg/l	65.6

2.3 Analytic Methods

The initial and final concentration for the prepared wastewater and treated solution were determined using spectrophotometer (HACH DR 6000). The percentage removal of COD was calculated using Eq. 1:

$$Percentage\,removal = \frac{(C_i - C_f)}{C_i} \times 100$$

Which,

   C_i = Initial concentration
   C_f = Final concentration

2.4 Optimization of adsorption experiment

Adsorption experiments were conducted in batch mode with initial concentration (10%, 30%, 50%), contact time (3h - 12h) and carbon dosage (0.1 g - 1.0 g). A series of flask containing 100 ml of wastewater sample with preset parameter condition were set in orbital shaker at 120 rpm.

Optimization experiments were designed using Box-Benhken design and the factors was evaluated as displayed in Table 2.

Table 2. Experimental design of COD removal on AC

Variables	Name	Unit	Low (-1)	High (+1)
A	Initial Concentrations	mg/l	10	50
B	Contact Time	hour	3	12
C	Carbon Dosage	g	0.1	1

3. Results and Discussion

3.1 Optimization Result

Table 3 refers to the corresponding analysis of variance (ANOVA) for Chemical Oxygen Demand (COD) adsorption capacity with initial concentration (A), contact time (B), carbon dosage (C). The model equation indicates that the high R^2 of 0.9901 showed that 99% of the variations in adsorption capacity can be explained by the independent variables. The high coefficient of determination between the predicted and experimental adsorption verified the quadratic polynomial model was sufficient to explain the interactions between variables and response [6]. The model also showed that R^2 is in reasonable agreement with adjusted R^2 value of 0.9774. The regression model for COD removal (%) in terms of the factors to be optimized is developed in Eq.2:

Removal of COD (%) = 70.26 - 3.52A* + 0.7813B* +12.95C* +1.23AB* +0.4550AC* - 0.1300BC* - 3.47A2* - 1.12B2* - 7.75C2*

The coefficients with one factor represent the effect of the particular factor, while the coefficients with two factors represent the interaction between the two factors. The positive sign in front of the terms indicates a synergistic effect, whereas a negative sign indicates an antagonistic effect. Therefore, increasing contact time and carbon dosage is expected to have better removal with lower initial concentration. This behavior is in agreement with the findings as discussed earlier [7].

Table 3. ANOVA results for the quadratic model of percentage removal of COD

Source	Sum of Squares	Degree of Freedom (DF)	Mean Square	F Value	P Value
Model	1782.58	9	198.06	77.76	< 0.0001
A	99.05	1	99.05	38.89	0.0004
B	4.88	1	4.88	1.92	0.2087
C	1342.14	1	1342.14	526.90	< 0.0001
AB	6.03	1	6.03	2.37	0.1679
AC	0.8281	1	0.8281	0.3251	0.5864
BC	0.0676	1	0.0676	0.0265	0.8752
A²	50.82	1	50.82	19.95	0.0029
B²	5.32	1	5.32	2.09	0.1916
C²	253.01	1	253.01	99.33	< 0.0001
Residual	17.83	7	2.55		
Lack of Fit	16.63	3	5.54	18.55	0.0082
Pure Error	1.20	4	0.2989		
Std Dev.		1.60	R^2		0.9901
Mean		64.44	Adjusted R^2		0.9774
			Predicted R^2		0.8511

The predicted color removal percentages versus the observed values are shown in Figure 1. The predicted values are calculated using the empirical model as shown in Eq.1.
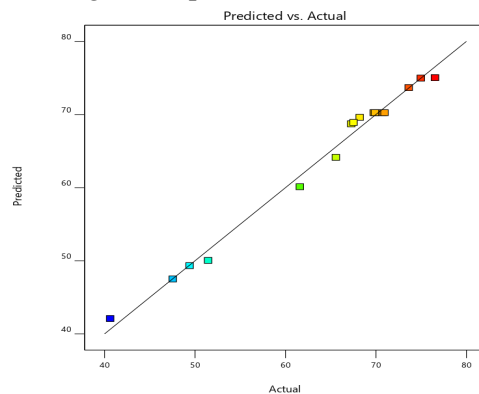
Figure 1. Experimental versus Predicted COD removal

Table 4 shows model verification for the study. Predicted value was estimated using Eq.2. The model predict achievement of 70% COD removal with 50% initial concentration, 0.9 g of carbon within 3 hour contact time and experimental value obtained under those conditions was in good agreement with predicted value.

**Table 4**. Experimental validation of empirical model

Parameter	Values
A, Initial Concentration	50%
B, Contact Time	3 hour
C, Carbon dosage	0.9 g
Predicted percentage removal of COD	70%
Experiment percentage removal of COD	71.63 %

The response surface plots for COD removal are shown in Figure 2. It could be seen from Figure 2a that removal was low at low carbon dosage and contact time. At lower carbon dosage of 0.1 g, the percentage removal were 47.54% for 3h and 49.4% for 12h. At higher carbon dosage of 1 g, the percentage removal were 73.62 for 3h and 74.96% for 12h. COD removal increased with both dose and contact time. This is expected as at higher dose, more active site available for adsorption and longer

times allow longer interaction between active site and adsorbate for removal occurrence. Another study on contact time and carbon dosage also supported this finding and stating that more adsorption site available as adsorbent dose was introduced and percentage removal is low with minimum contact time and carbon dosage [8].

   Figure 2b displays influence of initial concentration and contact time. At concentration of 10%, it was found that the percentage removal of COD at 3h and 12h produces 68.2% and 67.27% respectively. At concentration of 50%, the percentage removal for 3h is 61.59% and for 12h is 65.57%. With concentration of 30% with contact time of 7.5h, the percentage removal obtained result within range of 69% to 71%. There were no significant difference observed in low and high initial concentration using 3h and 12h contact time. However, in medium concentration with 7.5h contact time, higher percentage removal was achieved. According to study by Alkhatib, contact time is crucial in influencing adsorption equilibrium [7]. High percentage of COD was adsorbed in early phase due to larger surface area available and gradually only little removal occurred when approaching equilibrium phase. This finding supported by Thani claiming contact time is less important near equilibrium [6].

   Figure 2c shows initial concentration and carbon dosage relationship. At lower concentration of 10%, the obtained removal for 0.1 g and 1 g were 51.45% and 76.53%. At higher concentration of 50%, the results were 40.62% for 0.1 g and 67.52% for 1 g. Higher removal achieved when lower initial concentration with higher carbon dosage. This is because higher active site available to the lower number of particles to be adsorbed [9].

(a)

(b)

(c)
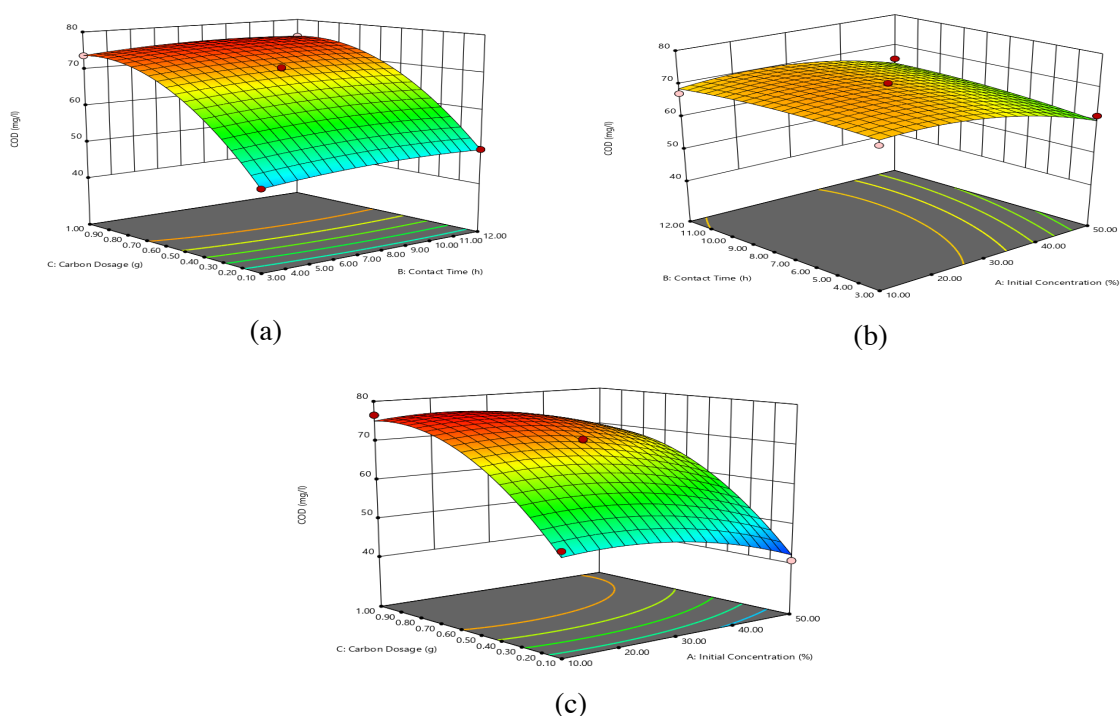
Figure 2. COD percentage removal as a function of a) carbon dosage and contact time b) contact time and initial concentration c) carbon dosage and initial concentration

4. Conclusion
Removal of COD from fish cracker industry effluent was studied using RSM. The removal was related to the batch experiment parameters by quadratic model. The model was able to predict the experimental data with R^2 of 0.9771. The COD removal was affected by initial concentration, carbon dosage and contact time. The optimum condition for highest removal (71.63%) was achieved with 50% initial concentration, 3 hour of contact time and 0.9 g of carbon dosage in agreement with 70% predicted

percentage removal. Therefore, chemically activated carbon prepared from bamboo was able to be utilized as good adsorbent.

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