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## Adsorption of COD in wastewater by Activated Carbon from Rice Husk

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**Abstract.** Organic pollution occurs due to the presence of organic compound that will causing oxygen depletion and gives potential adverse impacts on human health and the environment. Wastewater from the fish cracker industry typically has high chemical oxygen demand (COD) and it is needed to be treated before release to water bodies in order to reduce the organic pollution. The treatment of COD was done using rice husk activated carbon (RHAC) that produced from agricultural wastes that contributed to waste minimization and reduce the environmental pollution. Therefore, this study emphasized on the feasibility of rice husk activated carbon for removing the COD from fish cracker wastewater. It was also to identify the optimization factors (pH, flow rate and initial concentration of COD) that affecting adsorption by rice husk activated carbon. RHAC was produced by chemical activation using phosphoric acid, H<sub>3</sub>PO<sub>4</sub> and a real wastewater from fish cracker industry was collected, characterized, and treated by using continuous fixed bed column adsorption method. The result shows that the adsorption of COD by RHAC was optimum at pH 2, flow rate 5 ml/min and 5 % v/v initial COD concentration with the COD removal at 31.8% (150 mg/L) which comply with Standard B. Thus, RHAC could be used to develop an alternative method and suitable carbonaceous substances that was used to treat water and wastewater.

### 1. Introduction

Water is the most important element for life, and it is mainly used for domestic purpose such as food preparation, cleaning, personal hygiene, and other uses. Water pollution will decrease the total water availability due to the higher cost for treatment of the water that have been polluted [1].

Organic pollution is one of the water pollutions that caused by the release of organic compounds from the sources such as domestic sewage, urban runoff, industrial and agricultural wastes etc. into water bodies [2]. Wastewaters that had been discharged by many industrial consisted organic compounds which were toxic and would not suitable to direct biological treatment. Therefore, the wastewater should be treated before discharged into the river. Many conventional treatment methods had been used such as ion exchange, reverse osmosis and membrane filtration for treatment, but the cost involved in all these methods were very high [3]. The treatment of these pollutants also can be solved by using palladium catalyst and different support systems for enhanced catalytic activity [4]. However, the use of catalyst was also costly. Of the treatment methods existing, adsorption had been proven to be effective due to the lesser land requirement and flexibility in raw material use [5].

The large amount of agricultural or industrial materials will cause environmental pollution if do not disposed properly. Agricultural waste such as banana peel also has been proved to treat wastewater [6]. The utilization of agricultural or industrial materials to develop suitable carbonaceous substances will be useful to treat water and wastewater by adsorption process. One such product was activated carbon. Activated carbon was either commercial or the one synthesized from any renewable resource. Commercially activated carbon was very costly. Hence manufacturing activated carbon from low cost and easily available raw materials was an innovative idea. Activated carbon prepared from agricultural product such as kenaf core fiber [7], rubberwood sawdust [8], foxtail palm fruit [9] and palm kernel



shell [10] will reduce the cost of activated carbon production. In this study, rice husk, which was an agricultural waste, was used to produce activated carbon to reduce chemical oxygen demand (COD) in wastewater from fish cracker industry.

Fish cracker is one of popular snack in Malaysia market and it has great potential to be introduced internationally. Wastewater from the fish cracker industry typically has high COD that causes the organic pollution and brings harm to the environment, if it is released to the water bodies without treatment [11]. Currently, there is no treatment of COD for the wastewater from the fish cracker industry. The treatment of COD in wastewater could be done using adsorption by activated carbon that produced from agricultural wastes that were low in cost and easily available raw materials such as rice husk.

## **2. Materials and Methods**

### *2.1. Materials*

The raw material rice husk was obtained from the rice mill located in Tumpat, Kelantan. Whereas the wastewater samples were obtained from fish cracker industry at Tumpat, Kelantan. The wastewater samples were collected from different point of effluent discharged. Wastewater samples were collected in the clean plastic bottles, where the samples pH was adjusted using concentrated sulfuric acid to pH less than 2 in order to preserve the samples for later analysis. The preserved samples were kept at or below 6 °C for a maximum of 28 days.

### *2.2 Wastewater Characterization*

Wastewater from fish cracker industry was analysed through In-situ and Ex-situ analysis. For In-situ analysis, the wastewater samples were analysed and measured by using YSI Multiparameter instrument. While for Ex-situ analysis, the characterization of wastewater was analysed using the methods prescribed by American Public Health Association (APHA) method [12].

### *2.3 Preparation of Rice Husk Activated Carbon (RHAC)*

Rice husk was washed thoroughly by distilled water to remove dust and other impurities and oven dried at  $100 \pm 5$  °C for 24 hours. After oven dried, the rice husk was sieved to smaller size (approximate to 2 mm) to ensure uniform impregnation with the activating agent. 100 g of rice husk was then treated with 1L of 3 M phosphoric acid (QRec) at 80 °C for 3 hours. The pre-treated rice husk was washed by distilled water until the pH value become 7. The sample was oven-dried at  $100 \pm 5$  °C for 24 hours. The pre-treated rice husk was carbonized at 450 °C for 2 hours by using muffle furnace (WiseTherm). Rice Husk Activated Carbon (RHAC) was obtained and stored in airtight containers for further use. The process was repeated until substantial amount of carbon is obtained.

### *2.4 Characterization of RHAC*

Properties of rice husk activated carbon such as surface morphology was studied by employing scanning electron microscopy (SEM). While Thermogravimetric (TGA) analysis spectrum was conducted to determine amount of weight change of a material over time with the increasing of temperature.

### *2.5 Column Adsorption Study*

Column studies was conducted in a plastic column with internal diameter of 3 cm and length of 15 cm. RHAC (30 g) was placed into the column to yield a desired bed height of the adsorbent at 10 cm. The upper part and the lower part of the bed were covered by cotton to prevent the loss of RHAC adsorbent and ensure the arrangement of the bed was closely packed.

Influent of fish cracker wastewater was flowing through column at a fixed and controlled discharge rate downward through the column. Before the wastewater was injected, the distilled water was flow through the column to improve the wetting characteristics of the granules in order to ensure the provision of high interfacial area. After the distilled water was drained out from the column, the wastewater sample was flow through the bed column. Concentration of COD for the wastewater was carried out using

HACH Method 8000, USEPA Reactor Digestion Method (HR) with DR 6000 Spectrophotometer. Removal efficiency of the COD was calculated using the Equation 1.

$$P = \frac{C_i - C_o}{C_i} \times 100\% \quad \text{Equation 1}$$

where,

P = Removal percentage of COD (%)

C<sub>o</sub> = Initial concentration of COD (mg/L).

C<sub>i</sub> = Final concentration of COD (mg/L)

### 2.6 Effect of pH

The experiment was carried out by flowing the wastewater solution through the column containing RHAC with different pH parameter 2, 4, 6, 6.97 (original pH), 8 and 10. At the same time, the other parameters (flow rate and initial COD concentration) were kept constant at 5 ml/min and 1 % v/v respectively at room temperature. The resulted optimize value for pH that obtained was used in another adsorption study.

### 2.7 Effect of Flow rate

The experiment was carried out by flowing the wastewater solution through the column containing RHAC. Different flow rate 1 ml/min, 3 ml/min, 5 ml/min and 10 ml/min was adjusted and used for the COD adsorption at constant optimized pH value. At the same time, the other parameters which was initial concentration was kept constant at 1 % v/v at room temperature. The resulted optimize value for flowrate together with optimized pH were used in another adsorption study.

### 2.8 Effect of Initial COD concentration

The experiment was conducted by flowing the wastewater solution through the column containing RHAC. Different initial COD concentration 1 % v/v, 5 % v/v, 10 % v/v, 20 % v/v and 30 % v/v were used for adsorption by RHAC at constant optimized pH and flow rate. The experiment was conducted at constant room temperature and the result was recorded.

## 3. Results and Discussion

### 3.1 Wastewater Characterization

Table 1 indicated the wastewater characterization of fish cracker industry through In-situ and Ex-situ analysis.

**Table 1.** In-situ and Ex-situ Analysis for Fish Cracker's Wastewater

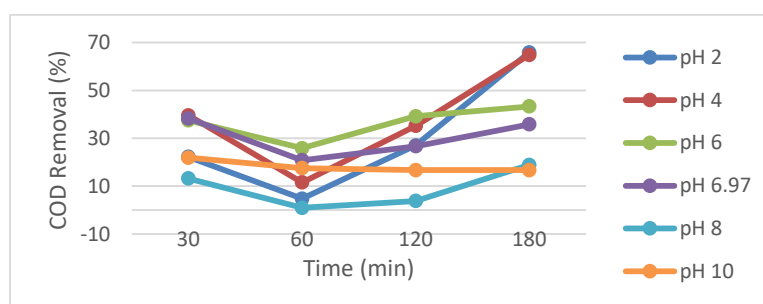
Physio-chemical Parameter	Average Reading	EQA Standard B
DO (%)	17.75	-
pH	6.97	5.5-9.0
SS (mg/L)	327	100
AN (mg/L)	14.6	20
COD (mg/L)	3415	200
BOD <sub>5</sub>	37.27	40

Among the physio-chemical parameter, the value of SS and COD were higher where the value were 327 mg/L and 3415 mg/L respectively. Both had exceeded the limit of discharge of industrial effluent for Standard B. The Environmental Quality Act (EQA) 1974 specifies two standards for effluent discharge: Standard A for discharge upstream of any raw water intake, and Standard B for discharge downstream of any raw water intake. The value for other parameter such as pH, AN and BOD<sub>5</sub> were pH 6.97, 14.6 mg/L and 37.27 mg/L which were still in the acceptable range for discharge. Thus, the fish cracker's wastewater was not suitable to be discharged into the river without treatment because it can cause the water pollution.

### 3.2 Column Adsorption Study

#### 3.2.1 Effect of pH

Figure 1 shows the result of COD removal that affected by the effect of pH parameter. The optimum pH was determined from the experiment conducted with different pH which were pH 2, 4, 6, 6.97, 8 and 10. The pH of solution is important in affecting the properties of adsorbate and adsorbent as well as the adsorption process in aqueous solutions [13].



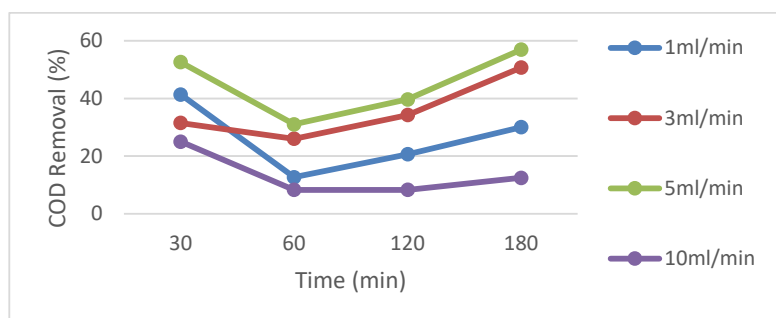
**Figure 1.** Effect of pH

From Figure 1, COD removal decreased from 30 to 60 min. The COD removal higher at the first 30 min due to the presence of water that still contained inside the RHAC. This was because before running the experiment the distilled water was used to flow through the column filled with RHAC which may affect the COD removal. From 60 to 180 min, it indicated that the COD removal increases with time where the COD removal for each pH parameter achieve highest efficiency after 180 min.

The percentage of COD removal for pH 2, 4, 6, 6.97, 8, 10 were 65.87%, 64.84%, 43.33%, 35.83%, 18.87% and 16.67% respectively. This shown that the COD removal efficiency was higher in acidic condition and it was attributable to the electrostatic interaction between the adsorbed compounds and the adsorbent surface [14]. From these, it can be concluded that pH 2 was the optimum pH for the COD removal. However, research conducted by Ratnoji & Mukundan [15] shown their optimized pH parameter was pH 6 where the COD removal was 92.37%, which was still in acidic condition but less acidic compared to the finding. The difference between the optimized pH for COD removal might due to presence of different elements in fish cracker wastewater and sewage wastewater that might affect the optimized pH factor [16]

#### 3.2.2 Effect of Flow rate

Figure 2 shows the result of COD removal that affected by the effect of flowrate parameter. The experiment was conducted by using different flowrate parameter which were 1 ml/min, 3 ml/min, 5 ml/min and 10 ml/min. From the figure, the COD removal higher at the first 30 min and became lower at 60 min. This was because at 30 min, the distilled water which was used to flow through the column RHAC was still present. From 60 to 180 min, it shown an increase trend of COD removal for each flow rate parameter.

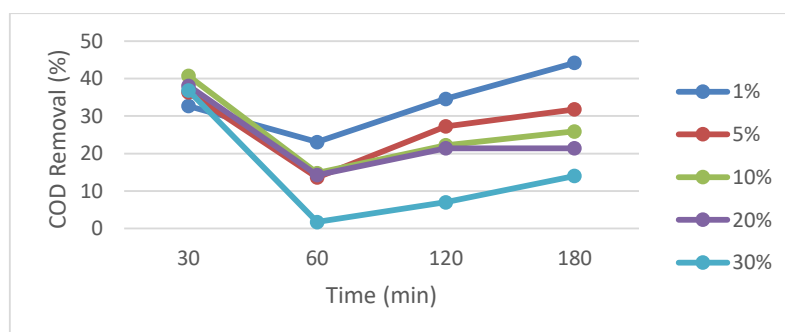


**Figure 2.** Effect of Flow rate

The percentage of COD removal for 1, 3, 5 and 10 ml/min flow rate after 180 min were 30.00%, 50.68%, 56.90% and 12.50% where the column was found to perform better at a lower flow rate compared to higher flow rate. This was because at higher flow rate, the adsorption capacity was lower due to insufficient residence time of the solute in the column and diffused into the pores of the adsorbent, and therefore, the solute left the column before equilibrium occurred [16]. However, the COD removal at flowrate 1 ml/min was lower than 3 ml/min and 5 ml/min. Unfortunately, there is no research to show the adsorption is less efficiency at lower flow rate. The variation of the result was due to the control of flow rate using burette which might cause a small error when the experiment was conducted. As indicate in these figures, the highest percentage of COD removal was at the flow rate of 5 ml/min and it was considered as the optimum flowrate for column adsorption for COD. The result was same as the research conducted by Patel & Vashi [16], where they had using activated Neem Leaf Powder as adsorbent to remove COD. The optimized flowrate parameter in their research was 5 ml/min where the adsorption was more efficiency at lower flow rate.

### 3.2.3 Effect of Initial Concentration

Figure 3 shows result of COD removal against initial COD concentration parameter. This experiment was conducted by increasing the concentration of COD from 1% v/v, 5% v/v, 10% v/v, 20% v/v and 30% v/v. As mentioned before, the removal at 30 min for all initial COD concentration was higher because it was affected by the present of distilled water which was still left in the column. The percentage of COD removal for all initial COD concentration was kept increased from 60 to 180 min.



**Figure 3.** Effect of Initial COD Concentration

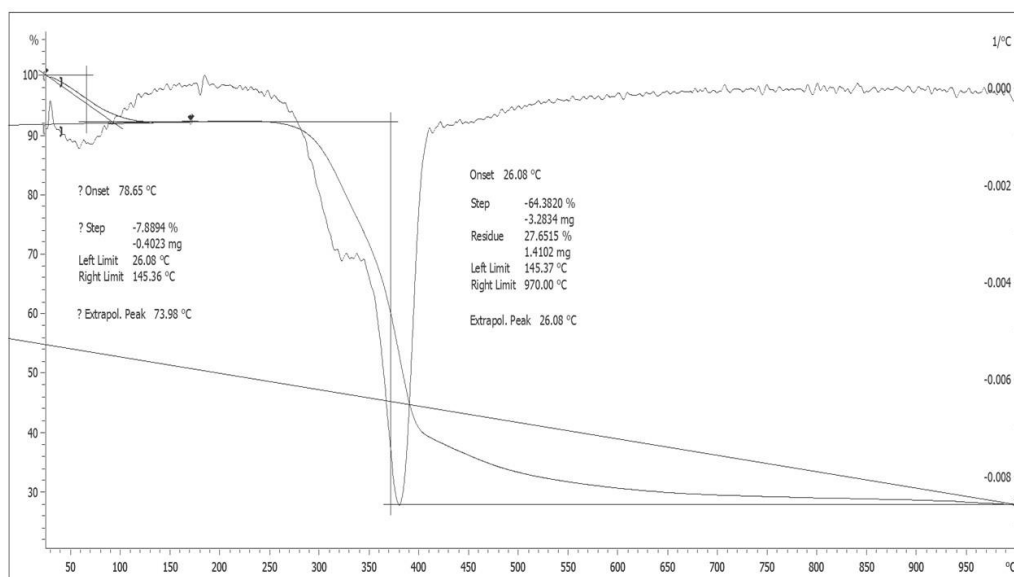
The percentage of COD removal efficiency for 1, 5, 10, 20 and 30% v/v as initial COD concentration were 44.23%, 31.8%, 25.93%, 21.43% and 14.04%. It was clearly seen that the removal of COD became lower as the initial COD concentration increase. This was because as the wastewater continued to flow, the concentration of initial COD increased, the RHAC's bed became more saturated and it became more difficult for the RHAC to adsorb the COD contaminants [17].

The results shows the highest removal of COD was at 1% v/v initial COD concentration, which was from 52 mg/L decrease to 29 mg/L but based on the EQA Standard B, the limit discharged of COD was 200 mg/L. Therefore, the maximum of the initial COD concentration that the RHAC can hold and treat to the COD concentration under limit discharged was 5% v/v COD concentration, that was from 220 mg/L to 150 mg/L. From these, the 5% v/v COD concentration of the wastewater was the optimum initial COD concentration that could be afford by RHAC. The COD removal was less efficient compared to the research conducted by Ademiluyi et al [18], where the activated carbon from waste Nigerian bamboo was able to remove 59.8 % of COD (from 378 to 152 mg/L) after three hours. This might be due to the saturation occur in the rice husk activated carbon due to the increase of concentration of contamination in the effluent as the wastewater continued to flow through the column [16].

### 3.3 Characterization of Adsorbent

#### 3.3.1 Thermogravimetric (TGA) Analysis

Figure 4 shows the result of TGA analysis for the raw rice husk. The TGA analysis was conducted to determine the weight change of raw rice husk over time. The thermogravimetric results for raw rice husk sample was plotted as a function of temperature as shown in Figure 4.

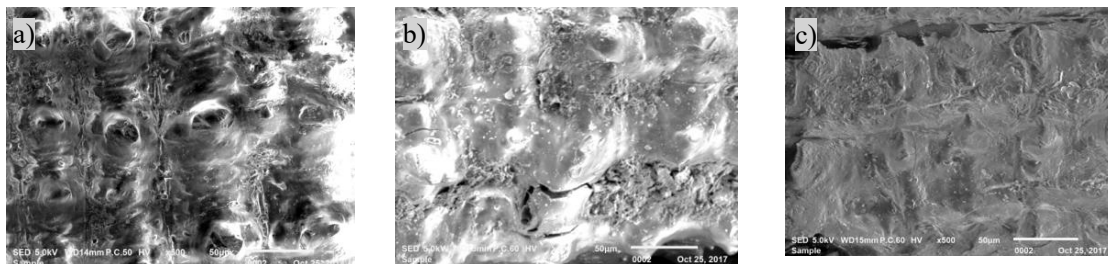


**Figure 4.** TGA analysis of Raw Rice Husk

It was observed that there were two phases in TGA analysis. For the first phase, the weight losses of raw rice husk were 7.8894% from the temperature 26.08 to 145.36 °C. The weight losses at the first phase TGA corresponded to the release of moisture from the biomass of raw rice husk [19]. The second phase of TGA analysis indicated there was significant 64.3820% weight loss in raw rice husk from temperature 145.37 to 970.00 °C. This was because the volatile matter had been pyrolysed and the others were converted into char and gases form [20].

#### 3.3.2 Scanning Electron Microscopy (SEM) Analysis

Figure 5 a, b and c show the surface morphology for raw rice husk, RHAC before adsorption and RHAC after adsorption for COD which were characterized by using SEM analysis with magnification 500x and 5kv.



**Figure 5.** SEM analysis for a) raw rice husk; b) RHAC before adsorption; c) RHAC after adsorption

From Figure 5a, there was many pores cavities could be seen clearly at the raw rice husk. After the raw rice husk was carbonized, the RHAC was produced where it contains pores cavities with some cracks and crevice that was highly possibility for COD adsorption (Figure 5b) [17]. In Figure 5c, it did not show any clear pore on the RHAC surface morphology. This was because during the adsorption, the pore cavities of the RHAC was filled with contaminants [20].

#### 4. Conclusion

Fish cracker industry was one of the sources of water pollution due to high COD concentration with other contaminants that was existing in the wastewater. From the result, the optimum pH, flowrate, and initial COD concentration were pH 2, 5 ml/min and 1% v/v where the COD removal was 31.8% (150 mg/L) which comply with Standard B. The use of activated carbon contributed for the utilization of several agricultural or industrial waste materials and helped to develop an alternative method that was used to treat water and wastewater.

#### Acknowledgement

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