

Proceeding Paper

Reduction of Oil and Grease from Degreaser Discharged Tank via Adsorption Process Using Microwave-Carbonized Corn Cobs [†]

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Abstract: The effect of the rapid industrialization created an enormous burden on the part of the environment. The quality of the water source is drastically affected by the discharged coming from the industry and domestic usage. Nowadays, the treatment of wastewater involves the use of chemicals and powder activated carbon made from agricultural waste is commonly used. This study used waste corn cobs activated with sodium chloride in a 1:2.5 ratio and utilized microwave. The percent adsorption for powder activated carbon (PAC) from corn cob with microwave heating reached 93% removal of oil and grease for 10 g dosage while 87% for powder activated carbon without microwave heating. Freundlich and Langmuir isotherms both represent the behavior of PAC and the breakthrough time decreased as flow rate of contaminant increased in the continuous flow system. The characteristics of powder activated corn cobs contained a lot of grooves, crevices and cracks and the macropores deep inside the surface was highly developed, typical for an activated carbon that facilitate effective adsorption process. The pore volume was found to be 1.3 cm³/g for PAC with microwave heating and 1.5 cm³/g for those without microwave heating. The pore volume determined the adsorption capacity of PAC from corn cobs.

Keywords: adsorption; corn cobs; Freundlich and Langmuir isotherms; microwave heating; oil and grease; powder activated carbon

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1. Introduction

Setting up of wastewater treatment is a common standard part of the operational project of the plants whose line of manufacturing uses water in their industrial processing line. One of the industries that use water in their process line is the metal fabrication with painting section. Prior to painting, there is a degreaser tank whose main function is to eliminate oil and grease on the surface of the metal prior to application of paint. The degreaser contains a lot of contaminants aside from its chemical content which is mostly surfactant in nature. These contaminants include oil and grease, soluble and non-soluble particles, color, high level of chemical and biological oxygen demand which when not treated will affect the quality of the water like the river or lakes that serve as the catch basin of this water pollutants. Chemical treatment is so far one of the effective ways of treating the degreaser wastewater discharged and part of that treatment is the carbon adsorption section of the wastewater chemical treatment line.

Many researches on the use of activated carbon derived from different agricultural waste product had been conducted. In one study, walnut and almond shells was used as

raw materials for activated carbon and used to remove the methylene blue dye (Pragya et al., 2013), rice husk and corn cobs were used to remove the phosphates waste (Aloko et al., 2007), similarly, maize corn cob activated carbon was used to remove lead contaminant from wastewater (Okafor et al., 2015). Many studies used corn cobs as precursor in removing waste like iron (Azad, 2014), chromium (Youssef et al., 2008) and remazol brilliant blue R dye (Ahmad et al., 2011). Although interests on corn cobs had grown, much more needs to be explored.

Thus, it is the main aim of the present work to determine the adsorption performance of corn cobs. Specifically, this study (i) created a powder activated carbon from corn cobs via microwave-assisted and non-microwave-assisted (thermal) process with sodium chloride as the activating agent; (ii) characterized the microwave-assisted and thermally carbonized corn cob; and (iii) used batch test and continuous fixed flow in determining the breakthrough time, Freundlich and Langmuir Isotherms and percent adsorption.

2. Materials and Methods

2.1. Preparation of Corn Cobs

The corn cobs were obtained from the local market of Biñan and Sta. Rosa Laguna. Ten kg of corn cobs were bought and the kernels were manually removed. The corn cobs were washed 5 times using distilled water to remove any dirt or impurities. It was dried under the sun for 1 week and powdered using a milling machine. It was then sieved to get the particle size distribution. The moisture content was determined before it was placed in an air tight container.

2.2. Carbonization Process

Twelve crucibles were prepared and 2 g of powdered corn cobs was placed in each crucible. The furnace was set to 4 different temperatures (150, 200, 250 and 300 °C) with 3 different time interval (1, 2, and 3 h) for each temperature. Each crucible was placed on a specific temperature and time interval. The results were recorded for observation and comparison and one crucible was chosen based on its appearance qualitatively. This served as the basis for best temperature and time in the production of a large amount of carbonized corn cobs. The temperature at 300 °C and the time of 1 h were considered as the best condition to carbonize the corn cobs.

2.3. Activation Process

Two 1.5 L beakers were prepared and 250 g of laboratory grade NaCl was added each to the two beakers. Then 1.2 L of distilled water was added and mixed until the salt was dissolved. 100 g of carbonized corn was added to each of the salt solutions and was slowly stirred for 8 h using the stirring plate and was allowed to stand for 24 h. It was transferred in another beaker and placed in a hot plate and was heated to 120 °C until it became paste in form. The content of the first beaker was placed in 20 crucible porcelain with cover and was placed in a furnace with a temperature set at 300 °C for 1 h. It was allowed to cool and was placed inside the filter cloth bin. It was washed with a running 18 L distilled water to ensure that no more salt is coming out from the filter cloth. The ion concentration of the filtrate was checked using a portable conductivity meter and after it reached the reading of 5 ppm or less, the washing was stopped. The solid particles were removed from the cloth and were placed in a pan and dried in an oven at 105 °C for 12 h. It was placed in a clean air tight container and was labeled with activated carbon without microwave heating.

The content of the second beaker was placed in a microwavable pan and placed inside the microwave oven and was set at 500 W and 20 min time. It was allowed to cool and was placed inside the filter cloth bin. The content was washed with a running 18 L distilled water to ensure that no more salt was coming out from the filter cloth. The ion concentration of the filtrate was checked using a portable conductivity meter and after it

reached the reading of 5 ppm or less, the washing was stopped. The solid particles were removed from the cloth and were placed in a pan and dried in an oven at 105 °C for 12 h. It was placed in a clean air tight container and was labeled with activated carbon with microwave heating.

2.4. Characterization

Ten grams each of carbonized charcoal, activated carbon with microwave heating and without microwave heating was sent to UP Diliman, College of Chemical Engineering and Chemistry for Scanning Electron Microscope analysis to test the surface morphology using 10 microns magnification. Another 10 g each of activated carbon with microwave heating and without microwave heating was sent to the Department of Science and Technology, Standard and Testing Division to test the percent carbon, hydrogen, and nitrogen content of the sample using the combustion method.

2.5. Batch Testing

The batch testing was conducted for the purpose of assessing the adsorption performance of the prepared activated carbon from corn cobs. It was also use to obtain the plots to model the appropriate isotherm/s. The parameter used to test the adsorption performance is the percent adsorption of oil and grease. The first set of samples using 1, 5, and 10 g of activated carbon with and without microwave heating was sent to Mach Union Laboratory for oil and grease adsorption test using the partition gravimetric method. The second set of samples was assessed in Mapua University's Environmental Engineering Laboratory for oil and grease test using the isotherm modeling batch test.

2.6. Isotherm Modeling Batch Test

A mixture of oil and water was prepared by adding 1 g of oil in five 500 mL Erlenmeyer flasks. On the first flasks 1 g of activated carbon with microwave heating was added and on the second flask 2 g was added, on the third flask add 3 g was added, on the fourth flask add 4 g was added and on the fifth flask add 5 g was added. The flasks were agitated for 5 h using a magnetic stir plate and filtered using a Buchner filter set up. The oil and grease was analyzed using the gravimetric partition method. Equations (8) and (9) were used to calculate the x/m and C_e . The x is the difference between the initial mass of the oil before adsorption and the final mass of the oil after adsorption in the sample. The m is the mass of the adsorbent used. C_e is the concentration of the oil-water mixture after adsorption. The curve was plotted for the Freundlich and Langmuir Isotherm and the best fit was determined by means of coefficient of determination.

3. Results

3.1. Carbonization Results of Corn Cobs

Base on the different samples prepared for the optimization of the best time and temperature for carbonization of precursor corn cobs, among the temperature used: 150 °C, 200 °C, 250 °C, 300 °C and at different carbonization time of 1, 2 and 3 h per temperature, the optimum time and temperature selected was 1 h and 300 °C. The basis of selection was on the appearance of the carbonized corn cobs as shown in Figure 1.

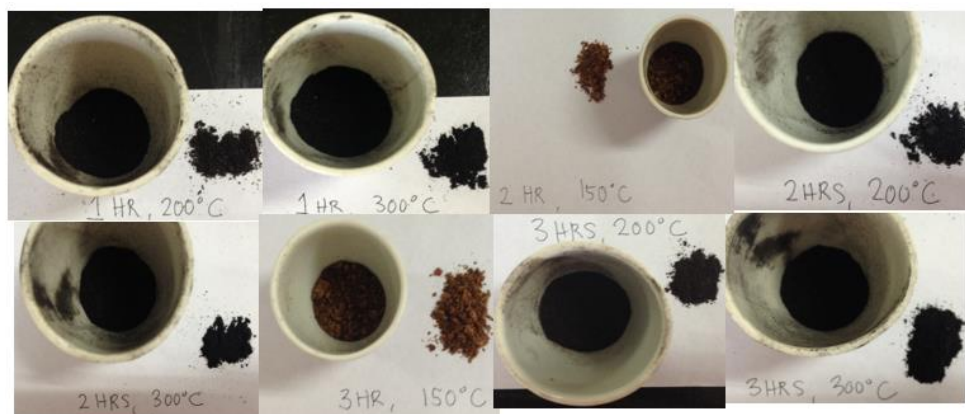


Figure 1. Carbonized Corn Cobs at Different Pyrolysis Temperature.

3.2. Characterization Results of PAC

Table 1 above show the characteristics of activated carbon from corn cobs use in this paper. It was compared with those found in related researches. There was only a slight difference on the values between the PAC with microwave and non microwave heating in comparison to the data from other researches in all aspects of the parameters which shows that the prepared activated carbon from maize corn cobs was expected to perform effectively in terms of contaminants removal in wastewater specifically the oil and grease via adsorption process. In some literature, the C content is as high as 78% (Aloko, 2007) and 72% (Azikiwe, 2012). Most of the activated carbon derived from agricultural materials has the values range between 41.23–84.50% C, 4.63–6.26% H and 0.7–4.10% N (Salam and Buhari, 2014). The moisture content was slightly higher as compared to the literature in the table but in some studies, percent moisture was reported as 15.47% (Salam and Buhari, 2014) and 13.2% (Raffiea et al., 2012) which are double in value in comparison to this paper. The pore volume for PAC corn cobs with and without microwave heating was a little bit higher than was found in the literature but it was still within the values of ASTM range of 0.68–2.80 cm³/g (Shah, 2007). Microwave created a direct heating of the particle interior as compared to conventional heating where the surface of the particle was heated before their interiors, inducing a thermal gradient between the surface and the core of each particle (Niya et al., 2012). A 500W power with an activation time of 20 min was used and in some literature, the power is increase from 550 W to 700 w up to 1000 W. A sign of decrease in adsorption was notice at 1000 W because excessive microwave energy could burn the carbon destroying the pore structure (Daud, 2012).

Table 1. Physical Characteristics of Powder Activated Carbon (PAC) from corn cobs.

Parameters	PAC with Microwave Heating	PAC without Microwave Heating	Literature	Reference
pH	7.7	7.4	6.4	Siva et al. (2012)
Density	0.4 g/ml	0.33 g/ml	0.47 g/ml	Siva et al. (2012)
% Moisture	7.4	7	5	Siva et al. (2012)
Iodine No.	550 mg/g	540 mg/g	453 mg/g	Rasayan (2012)
Surface Area *	900 mg/g	900 mg/g	504 mg/g	Rasayan (2012)
Ash Analysis	3.4%	4%	8.9%	Kumar (2009)
Pore Volume	1.3	1.5	1.1 cm ³ /g	Aloko(2007)
% C	64.7	55.5	52.31	Buhari (2014)
% N	4.55	4.84	3.38	Buhari (2014)
% H	0.915	1.16	1.02	Buhari (2014)

* Estimated from the work of Mopoung et al. (2015) based on iodine number.

The SEM result of PAC corn cob with microwave heating is shown in Figure 2a. It contains a lot of grooves, crevices and cracks. The macropores deep inside the surface were highly developed. Macropores served as a transport channels for the adsorbate into the micropores and mesopores (Bansal and Goyal, 2005). The high iodine value of 550 mg/g which gives an estimate amount of micropores (Srisa-ard, 2014) explain the reason for high percent adsorption (96%) since 95% of the the total surface area is covered by the specific area of micropores (Bansal and Goyal, 2005). The same analysis is given to PAC corn cobs without microwave heating as shown in Figure 2b magnified at 10 microns. Groves, crevices and cracks are also visible. Macropores are highly developed inside the surface and high iodine value of 540 mg/g is due to the macropores branching into mesopores and mesopores branching into micropores (Bansal and Goyal, 2005) giving a high adsorption capacity (96%).

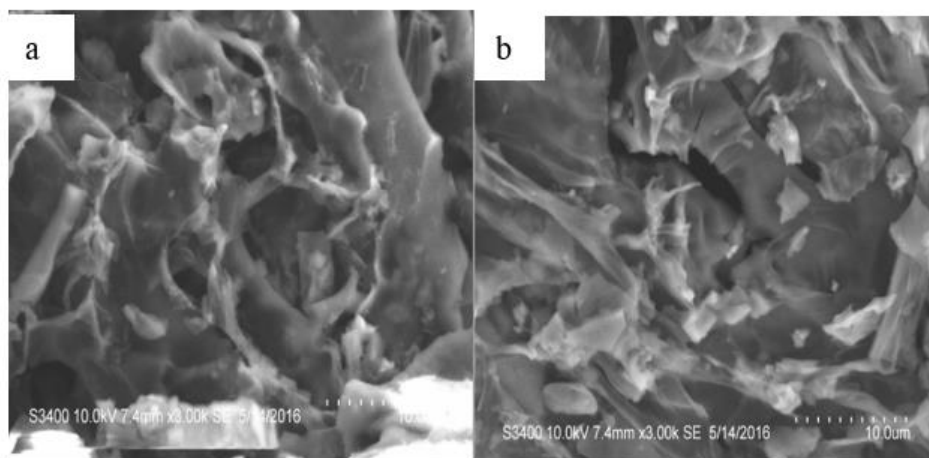


Figure 2. (a) SEM PAC corn cobs with microwave heating; (b) SEM PAC corn cobs without microwave heating.

3.3. Equilibrium Batch Test of Oil and Grease Using In-House Laboratory Testing

The results of the batch test for oil and grease using 500 mg of oil spike in 250 mL water and applying 5 g of PAC corn cobs with and without microwave heating is shown below:

The graph in Figure 3a shows that at time equal to 15 min, the concentration of the oil and grease in the adsorption solution reach it equilibrium value. The percent adsorption is 96.3% and the adsorption capacity is 27.24 mg/g. Figure 3b on the other hand reach equilibrium time after 25 min with percent adsorption equal to 96% adsorption. Both types of PAC corn cobs exhibit the same amount of adsorption at 5 g dosage.

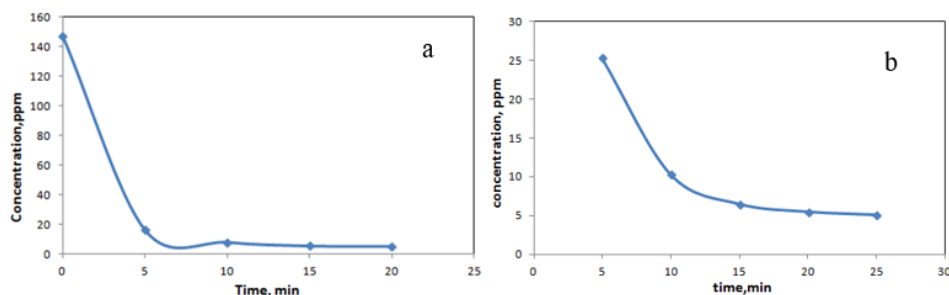


Figure 3. Concentration versus time graph of batch equilibrium test using PAC corn cobs with (a) and without (b) microwave heating; ◆ Data from this work.

The high percent adsorption which was 96.3% for powder activated carbon with microwave heating and 96% adsorption for powder activated carbon without microwave heating was related to the high carbon content after carbonization as shown in Table 7. The reason for this is because during carbonization, element such as Oxygen, Hydrogen, Nitrogen, and Sulfur are removed as volatile gaseous products. This leaves the residual elementary carbon atoms to form stacks of aromatic sheets that are good foundation for the creation of pores which are enhanced during activation process (Bansal and Goyal, 2005).

The low percent ash for powder activated carbon with microwave heating which was 3.4% and 4% in the case of powder activated carbon without microwave heating was related also to the high performance of the activated corn cobs since the lower the ash percent, the higher is the ability of the carbon to adsorb contaminant since ash constitutes the inorganic part of the carbon (Salam et al., 2014).

In comparison with the performance of activated carbon from corn cobs, Table 13 shows the other methods of removing oil and grease together with the percent removal

3.4. Isotherm Modeling

The model selected in terms of isotherm depends upon the value of the highest R and the n value for Freundlich. The n must be in unity because less than 1 value means there is poor adsorption characteristics (Bansal and Goyal, 2005). Below is the graph of Langmuir and Freundlich Isotherms for PAC corn cobs with and without microwave heating. The graphs shows the appropriate isotherm suitable for the PAC use in this paper and the corresponding isotherm equation.

Base on Figure 4a,b to Figure 4c,d and given the value of R², both isotherms indicates a strong evidence of adsorption of oil and grease to PAC corn cob with and without microwave heating. This indicates that the adsorption of oil and grease from the synthetic wastewater could be either monolayer or multilayer. This was the same finding conducted during the adsorption of Cr⁶⁺ using activated rice husk carbon (Yilleng et al., 2013). The high adsorption percentage during preliminary and equilibrium batch test is due primarily to high adsorptive capacity which is 1065 mg/mg for PAC with microwave heating and 578.1 mg/mg for PAC without microwave heating.

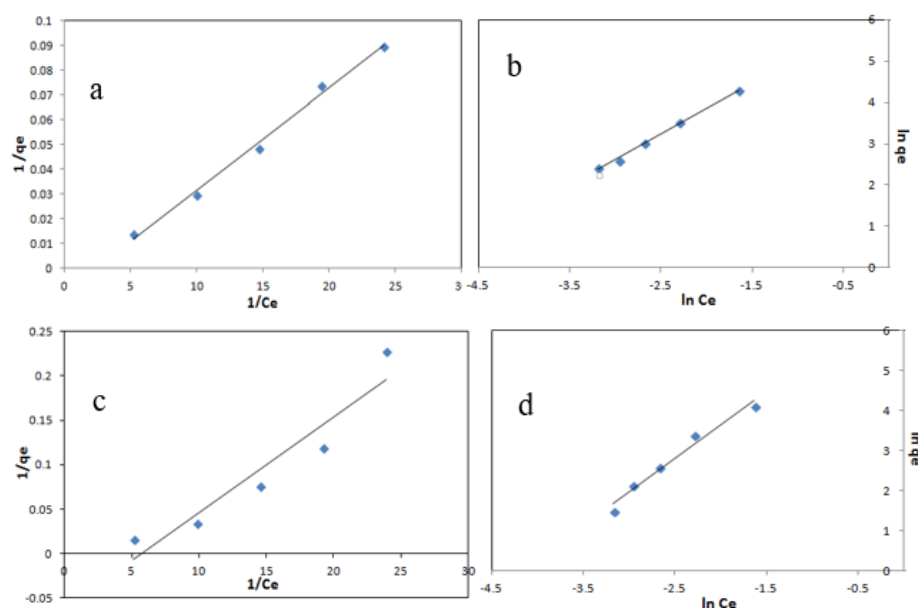


Figure 4. Isotherm model equation (a) Langmuir Isotherm (R² = 0.9933) (b) Freundlich Isotherm (R² = 0.9978) (c) Langmuir Isotherm (R² = 0.9055) (d) Freundlich Isotherm (R² = 0.9712) of PAC corn cobs without and with microwave heating respectively.

4. Conclusions

The study showed that the prepared PAC corn cobs are comparable in terms of those found in other literature. The SEM analysis reveals the cracks, grooves and crevices due to the reaction of sodium chloride with carbon creating those sites available for contaminants. The batch test confirmed the effectiveness of the prepared PAC from corn cobs both using microwave heating and without microwave heating by adsorbing or removing oil and grease by more than 85% of the initial value. Finally, the Freundlich isotherms and the Langmuir isotherms are both best fit for PAC corn cobs with and without microwave heating as R^2 are high for both types of activated carbon.

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