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Cite as: AIP Conference Proceedings **2339**, 020092 (2021); <https://doi.org/10.1063/5.0044540>
Published Online: 03 May 2021

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Production of Activated Carbon from Rubber Wood Sawdust Using Microwave Processing for Removal of Textile Dye Effluent

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Abstract. The production of activated carbon using heat from microwave processing for activation time was successfully conducted and its performance in treating textile dye effluent was observed through various factors such as phosphoric acid loading used, contact time, dosage of activated carbon and pH. Activated carbon with phosphoric acid loading of 60% shown highest BET surface area that was 213.52 m²/g however, it obtained least thermal stability. The result indicated that about 97.11% of dye from textile effluent can be removed in only 90 minutes of contact time at pH 3 with the use of 2.0 g of produced activated carbon. The production of activated carbon using microwave processing can be an alternative way for activation process using conventional furnace heating. The used of rubber wood sawdust as the raw material for activated carbon production can solve the waste issue from sawmill as an example.

INTRODUCTION

Activated carbon has recently been utilized as an adsorbent because of its ability for adsorption process in removing gas, dye and heavy metal [1-3]. It also tremendously used in drinking water purification and wastewater treatment, particularly in dye. Dyes are widely used in industries such as textiles, leather, paper, and plastics to color their final products [4]. The presence of dyes even in small quantity can seriously disturb living aquatic [5] due to highly toxic, stable, hardly biodegradable and visibly seen as pollutant in the environment [1,6]. Besides, Malaysia is known for its *batik* textile industry that has contributed significantly to the country economy particularly to the state of Kelantan and Terengganu [6-7]. However, textiles industry consume a lot of dye (for colouring the textile) and water during its manufacturing especially during washing stage which later contributed to the textile wastewater containing variety of dyes and chemical addition that can pollute the environment [6]. Moreover, *batik* manufacturing industries in Kelantan recorded the lowest percentage of environmental compliance (62.50%) [7]. Hence, treatment action for textile effluent should be conducted before discarded to the rivers or lakes. Among technologies use for wastewater treatment such as chemical precipitation, solvent extraction, oxidation, reduction reverse osmosis, flocculation and sedimentation; adsorption is considered to be relatively superior, most convenient and effectively to other technologies. Nevertheless, the major challenges faced with adsorption by activated carbon were its cost production and effectiveness in term of raw material [4]. Thus, much attention has been devoted to obtaining activated carbon from timber production waste or sawmills; post-agricultural and post-industrial waste which later can significantly solve the waste management issues [1]. The second challenges were its production time

where carbonization and activation time in activated carbon production consuming a lot of time once using conventional furnace heating. In respond to this, microwave heating is an alternative way to produce activated carbon as it has capability to reach higher temperature faster, quickly reduce heat when the microwave is turn off, accurate and more efficient [8-9]. In this study, the microwave processing for heating process was used in the activation of the char only however, the carbonization of the rubber wood sawdust still using conventional furnace heating as the biomass (rubber wood sawdust) is a poor microwave absorber since it required a dielectric material such as carbon material to initiate the process of heating. Furthermore, the resulted char form carbonization should be impregnated with the activating agent such as $ZnCl_2$, $NaOH$, KOH and H_3PO_4 prior expose to microwave irradiation. Besides as oxidants and dehydrating agents, the chemical activating agents also serve as microwave absorbers because of their dielectric properties [9]. Thus, the aim of this study was to produce activated carbon from rubber wood sawdust using microwave processing and tested its application in treating textile dye effluent collected from local batik factory.

MATERIALS AND METHODS

The rubber wood sawdust waste was collected from local saw mill located in Kota Bharu, Kelantan, Malaysia. The sample was washed, dried in the oven for 24 hours and sieved in the range 0.5 – 1.0 mm. While the textile dye effluent was obtained from local batik factory located in Kota Bharu, Kelantan Malaysia as well. The dye was heated on a hot plate and the formed wax on top layer of the dye need to be removed once cold prior treated with the produced activated carbon.

Production of Activated Carbon

The rubber wood saw dust is carbonized using a furnace at $500^\circ C$ for an hour. The resulting chars are impregnated with different loading of H_3PO_4 (30%, 40%, 50% and 60%) for 24 hours soaking duration. The impregnated ratio was 1:4 (acid:char). After that the mixture is filtered using a vacuum pump and dried overnight in an oven $105^\circ C$. The mixture is activated in the microwave processing with the heat of 500W for 6 minutes of irradiation time along the nitrogen stream of 20 ml/min.

Treatment of Textile Dye Effluent using Activated Carbon (Adsorption Study)

The volume of textile dye effluent used was 50 ml treated with activated carbon where the effect of H_3PO_4 loading (30%, 40%, 50% and 60%), contact time (30, 60, 90 and 120 minutes), activated carbon dosage (0.4, 0.6, 0.8, 1.0, 1.4, 1.6, 1.8 and 2.0 g) and initial pH of dye effluent (3,5, 7, 9 and 11.2 (original pH)) were investigated.

Characterization

The thermal behavior and stability of the produced activated carbon was evaluated using thermogravimetry analyzer (Mettler Toledo, model; TGA/DSC 2) with heating rate of $10^\circ C$ under nitrogen atmosphere and temperature range from room temperature to $900^\circ C$. The BET surface area of the activated carbon was calculated from N_2 adsorption isotherms. The concentration of the textile dye before and after adsorption treatment was determined by HACH DR6000.

RESULTS AND DISCUSSIONS

Effect of Phosphoric Acid Loading

The production of activated carbon from rubber wood sawdust using microwave processing showed in the Fig. 1 indicated that the higher the phosphoric acid (H_3PO_4) loading used in the activation process the lower the percentage yield of the activated carbon production. Even the use of microwave have shorten the heating process time however, the role of H_3PO_4 had stimulated the gasification of char thus increased the weight loss of the activated carbon resulting in the low yield. However, as the loading of H_3PO_4 increases, it is expected for the more potential sites penetration which benefited the following pore-opening and widening the processes. Nevertheless, insulating layer

might be formed if excessive H_3PO_4 load because it could not promote further activation of the char [10]. Generation of more pore especially micropore area ($93.45 \text{ m}^2/\text{g}$) in the produced activated carbon with 60% loading H_3PO_4 is recorded through BET surface area analysis which shown in Table 1.

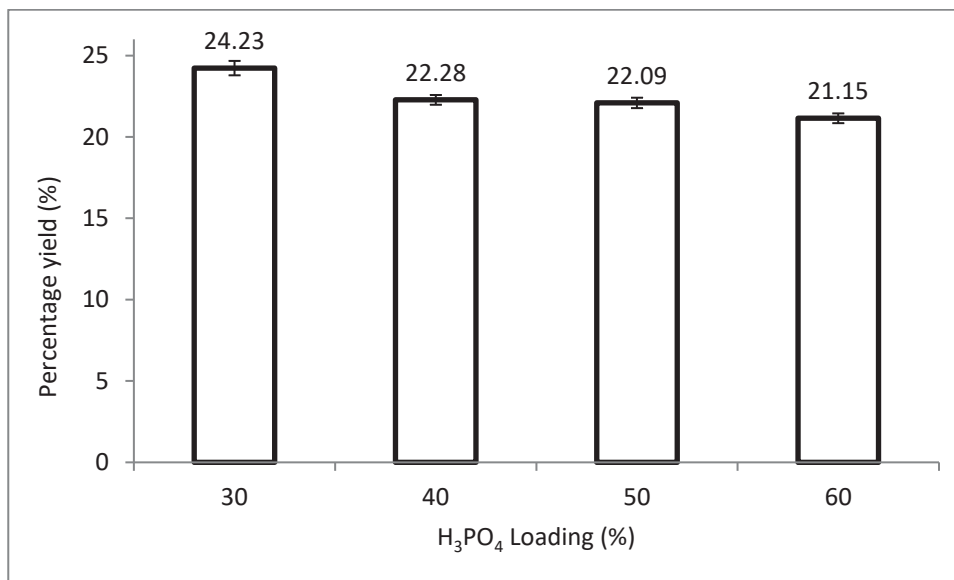


FIGURE 1. Percentage yield of the produced activated carbon using microwave processing.

TABLE 1. Micropore area and volume of the activated carbon using microwave processing with various H_3PO_4 loading.

Samples	H_3PO_4 loading (%)	BET Surface area (m^2/g)	Micropore Area (m^2/g)	External surface area (m^2/g)	Micropore volume (cm^3/g)
AC30	30	124.67	62.19	62.51	0.0064 cm^3/g
AC40	40	132.73	66.18	66.56	0.0085 cm^3/g
AC50	50	137.45	58.59	78.86	0.0044 cm^3/g
AC60	60	213.52	93.45	120.07	0.0279 cm^3/g

As activated carbon being produced using microwave processing, it is then characterized using thermogravimetric analysis (TGA) for thermal stability analysis. The least thermal stable shown in Table 2 was AC60, as its total mass loss was 78.85%. The highest thermal stability is obtained by AC30 with lower H_3PO_4 loading.

TABLE 2. Thermoanalytical result for activated carbon activated with various H_3PO_4 loading.

Samples	H_3PO_4 loading (%)	DTG Peak at 1 st mass loss ($^{\circ}\text{C}$)	DTG Peak at 2 nd mass loss ($^{\circ}\text{C}$)	Total mass loss (%)
AC30	30	56.7	581	75.77
AC40	40	56.5	589	77.72
AC50	50	47.7	589	77.91
AC60	60	48.3	581.3	78.85

Although AC 60 has high micropore area which reflected to the good characteristic of activated carbon however, it is least thermally stable due to high mass loss production. As AC 60 obtained high surface area hence it is selected to be used for removing the dye in textile effluent.

Effect of Contact Time on The Removal of Dye in Textile Wastewater

The removal of dye in 50 ml of textile wastewater is conducted using 0.4 g of AC60 with four different contact times: 30, 60, 90 and 120 minutes. The pH of the textile wastewater was 11.2 without further pH modification unless for pH study. The result in Fig. 2 shown that dye removal is increased in time from 30 to 90 minutes (60.0%) and stated to decrease when the contact time at 120 minutes. The increment of dye removal at the initial stage of contact time was due to fast adsorption rate on the availability of uncovered surface area in AC60 [6]. Hence 90 minutes of contact time is selected for further study for the effect of AC60 dosage.

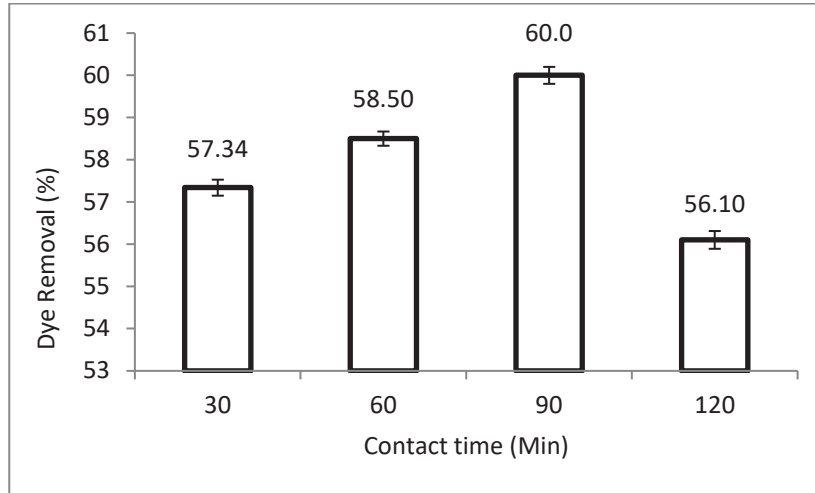


FIGURE 2. Effect of contact time on the percentage removal of dye in textile effluent.

Effect of AC60 Dosage on The Removal of Dye in Textile Wastewater

The removal of dye is extended to be studied under various AC60 dosages where the amount studied were 0.4, 0.6, 0.8, 1.0, 1.4, 1.6, 1.8 and 2.0 g. According to the Fig. 3, the percentage dye removal in 90 minutes of contact time is increased as the AC60 dosage is increased. This happened due to more surface site of activated carbon available with the fix volume of the textile effluent to be treated [6, 11]. The highest dye removal recorded was 70.89% at the dosage of 2.0 g of AC60. Hence, 2.0 g of AC60 is used for the subsequent experiment.

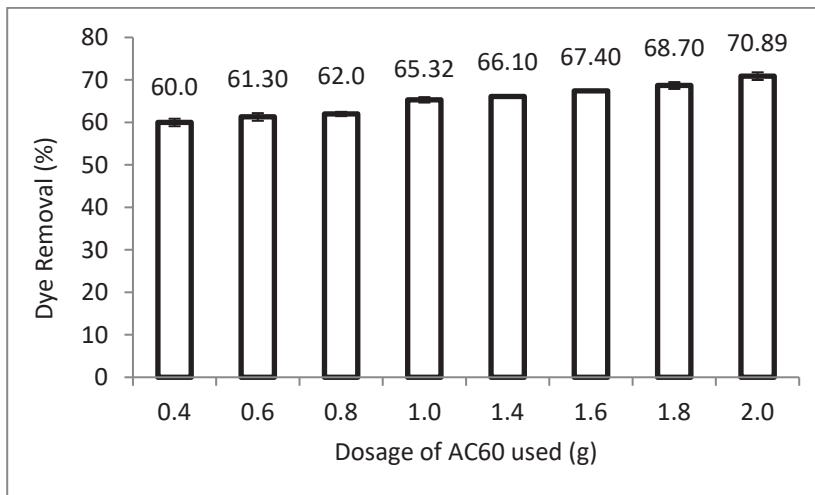


FIGURE 3. Effect of AC60 dosage on the percentage removal of dye in textile effluent.

Effect of Initial pH of Textile Effluent

The effect of the initial pH value of the textile effluent on the adsorption process using AC60 was investigated in conditions of 50 ml textile effluent, 2.0 g of AC60 in 90 minutes of contact time. The pH of textile effluent without modification was 11.2 and the varied pHs were 3, 5, 7 and 9 as can be seen on Fig. 4. At low pH values of 3, the surface of the AC60 is positively charged and the dye removal was found to be 97.11%. The dye removal percentage was decreases as the pH value is increased. This should be attributed to the presence of anionic dye type in the textile dye effluent where electrostatic attraction between AC60 surface and anionic dye molecule happened affected by the H^+ ions [4-12]. The faded dye that has been treated using AC60 is shown in Fig. 5.

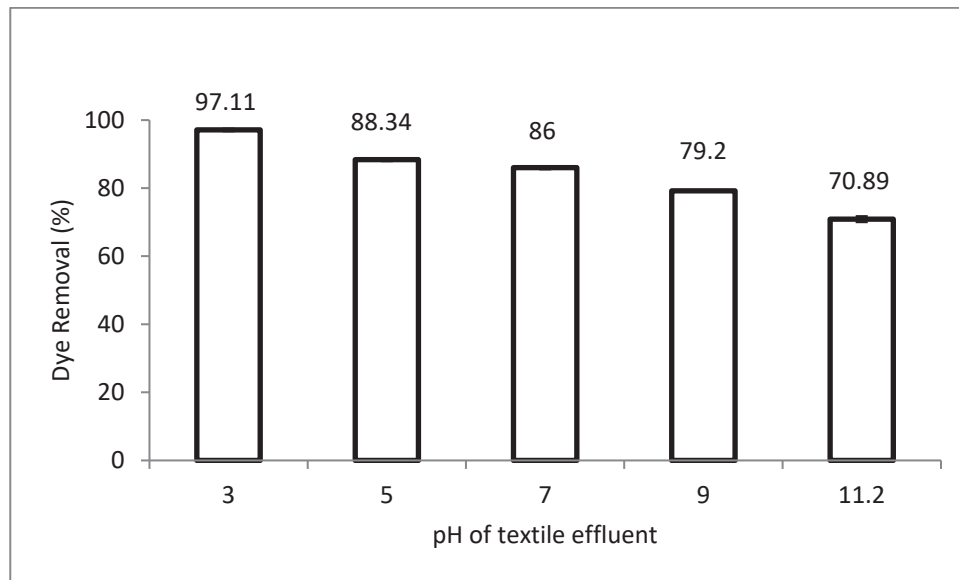


FIGURE 4. Dye removal using AC60 in various pH of textile effluent.



FIGURE 5. The treated dye effluent (modified pH from left: pH 3, 5, 7 and 9) with AC60.

CONCLUSIONS

This study revealed that rubber wood sawdust can be a potential raw material for producing activated carbon and microwave processing was able to shorten the activation time. The produced activated carbon also showed a potential application in treating dye with highest dye removal of 97.11% at pH 3 in 90 minute of contact time.

ACKNOWLEDGMENTS

The authors acknowledge the Ministry of Higher Education (MOHE) and Universiti Malaysia Kelantan (UMK) for funding the project through Research Acculturation Grant Scheme (R/RAGS/A08.00/00235A/001/2015/000198).

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