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Removal of Methyl Red using Chemical Impregnated Activated Carbon Prepared from *Parkia speciosa* Pod (Petai) as a Potential Adsorbent

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Abstract

Textile industry is one of the major contributors either in terms of employment or economies. This industry has provided variety of daily necessity such as sources of yarn and clothing. Extensively use of dyes in this textile industry has created water pollution. The serious problem happened when the daily water usage is from the untreated effluents which are discharged directly into water bodies. However, the disposed dyes into environment can be treated with adsorbents such as activated carbon via adsorption process. In this study, *Parkia speciosa* (petai) pods were chosen as the raw material from agricultural waste to produce activated carbon. Activated carbon was prepared from two different chemicals and application of four different carbonization time. Two parameters studied in the experiment are initial dye concentration and contact time. From the result, 100% of methyl red was removed by the activated carbon impregnated with zinc chloride solution at 1 hour carbonization time. The optimum time and initial concentration of dye was 30 minutes and 10 ppm respectively with the percentage removal of 100%. Thus, this result could contribute some knowledge on the use of alternative adsorbent from agricultural waste impregnation with specified chemicals in treating textile industrial wastewater.

1. Introduction

Wastewater from industrial activities mostly is being dispersed at the end, into the water bodies which are the main sources for human to do their daily activities for household and also agriculture. This wastewater is not only toxic to the biological world, but also contains colour, which blocks sunlight to reach into the water for photosynthesis (Abidin et al., 2016). For these reasons, it causes many problems to the ecosystem.

Adsorption is a physical method which removes dyes from the wastewater. Despites introduction of biological method such as biodegradation, this conventional biological treatment processes are not very efficient for the treatment of dyes, due to the low biodegradability of dyes (Rahman et al., 2012). Adsorption by activated carbon has a greater potential for the removal of dyes without availability of any impurities. Adsorption onto activated carbon is proven to be very effective in treating textile wastewater (Baseri et al., 2012). Recently, raw materials that are cheap and readily available such as agriculture waste have been experimented in order to reduce the activated carbon cost production. Due to their availability at low price, they were recognized as promising raw materials for the activated carbon production (Savova et al., 2001) Therefore, in this study, *Parkia speciosa* pods were chosen as a low cost adsorbent for the preparation of activated carbon.

Parkia speciosa is a tropical leguminous tree in the family of *Leguminosae* found in most of South East Asian country (Jamaludin & Mohamed, 1993). It is known in different countries with different names such as *petai* in Malaysia, *sataw* in Thailand, and *nejirefusamame* in Japan (Amarnath, 2004). It is known to have important chemical and medicinal compounds such as several cyclic polysulfides which are used for treatment of antibacterial activity on kidney, ureter and urinary bladder infections, thiazolidine-4-carboxylic acid for anticancer activity and have a hypoglycaernic effect due to synergistic action of sitosterol and stigmasterol (Mohd Azizi et al., 2008). This species has very stingy smell. Even though, the seeds are being cooked or boiled, the smell is more obvious to be stinking. The seeds are used as a main ingredient in cooking and have always been a popular local delicacy whereas the pods are usually disposed (Foo & Lee, 2010).

Despite making an effort in cost reduction on activated carbon production, there are numbers of

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contemporary research on potential of agriculture waste as adsorbents and sustainable solution in environmental disposal issue. Maiti et al. (2007) reported the potential of agriculture waste such as mustard stalk, jute stalk, sesame stalk, wheat straw, and bagasse and rice husk conversion to bio adsorbents in dye removal. Thus, in this study, *Parkia speciosa* pods were chosen as a potential adsorbent in removing methyl red in synthetic textile wastewater.

2. Materials and Methods

2.1. Materials

Parkia speciosa pods were obtained from a local market and the seeds were taken out from the pods. The pods were washed with distilled water to remove impurities before dried in oven at 100°C for 24 hours to reduce moisture content. Zinc chloride and phosphoric acid were prepared at 30% concentration solution for activation process. Reduction of methyl red was determined using UV-Vis spectrometer.

2.2. Preparation of Activated Carbon

The dried pods were ground with a grinder and sieved to 425 µm particle size using a siever. After that, 10 g of dried pods were added into four conical flasks containing 20 ml of 30% zinc chloride. The conical flasks were shaken about 5 minutes for homogeneity and were impregnated for 24 hours. Later, the samples were carbonized with different carbonization time (5, 10, 30 and 60 minutes) in furnace at 400°C. Finally, the activated carbons produced were rinsed with distilled water to obtain neutral pH 7. The final product of activated carbon was dried in oven at 50°C for 24 hours and kept in a sterile container with anhydrous sodium chloride. The procedures were repeated using 20 ml of 30% phosphoric acid. In the end, total of 8 samples were prepared for the dye removal study. The experiment was replicated three times and the average reading was taken.

2.3. Preparation of Methyl Red as Synthetic Textile Wastewater

The dye stock solution was prepared by dissolving 1 g of methyl red ($C_{15}H_{15}N_3O_2$) into a reagent bottle and diluted up with 1000 ml of distilled water to make a stock solution of 1000 mg/L (Abdullah et al., 2005). Then, for the preparation of calibration curve, dilution for definite volumes of stock solution was carried out to obtain desired concentration of 2, 4, 6, 8, 10 and 12 ppm. For absorbance reading, a UV-Vis spectrometer (Genesys Spec-20) was used with wavelength setting for methyl red of 410 nm. Then, the percentage of dye removal was calculated using following formula from the reading recorded.

$$\%\,removal = \frac{initial\ reading - final\ reading}{initial\ reading} \times 100\%$$

2.4. Selection of Best Adsorbent from different Carbonization Time and Impregnation Chemicals

All analysis results were subjected to statistical treatment to determine mean and standard deviation using the latest statistical software.

2.5. Effect of Contact Times

5 g of activated carbon selected from dye removal study was added into three conical flasks containing 25 ml of 10 ppm of methyl red solution. The solutions were shaken using orbital shaker for 30 minutes, 1 hour and 2 hours at 100 rpm to determine effect of contact time. Then, the dye solution in each of the conical flasks was filtered to separate the solution and activated carbon. The filtered dye solutions were pipette into cuvettes and the reading were recorded using UV-Vis spectrometer. The contact time that recorded highest percentage removal of dye was selected for the subsequent experiment.

2.6. Initial Dye Concentration

5 g of activated carbons were added into conical flasks containing different concentration of methyl red solution (10, 20, 50 ppm) to study effect of initial dye concentration on dye removal. The conical flasks were shaken using orbital shaker at 100 rpm for 30 minutes. Then, the dye solution in each of the conical flask was filtered to separate the solution and activated carbon. The filtered dye solutions were pipette into cuvettes and the reading were recorded using UV-Vis spectrometer.

3. Results and Discussion

3.1. Production of Activated Carbon form *Parkia Speciosa* Pods

Total of 8 activated carbon produced were labelled as shown in Table 1. The category of activated carbon was divided based on the chemicals used and carbonization time. The weight was recorded after carbonization and washing process to track weight loss after production. Carbon yield for each activated carbon was calculated using these data.

Table 1: Mass of activated carbon (Carbon yield %)

Types Of Activated Carbon	Chemical Agent	Carbonization time (minutes)	Mass of AC before wash (gram)	Mass of AC after wash (gram)	Carbon Yield (%)
A	$ZnCl_2$	5	6.94	4.27	42.7
B		10	7.49	4.54	45.4
C		30	6.81	3.77	37.7
D		60	6.55	3.40	34.0
E	H_3PO_4	5	9.94	3.67	3.67
F		10	10.43	1.97	19.7
G		30	11.01	2.54	25.4
H		60	9.47	3.01	30.1

From Table 1, highest carbon yield recorded was achieved by activated carbon B which is 45.4%. Activated carbon E recorded the lowest carbon yield which is

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3.67%. The carbon yield displayed different trend line for different chemical. Phosphoric acid showed increased carbon yield with increased concentration while carbon yield for zinc chloride showed decreasing trend line. According to Omotosho & Amori (2016), close value in percentage carbon yield shows that the base materials used for the activation process undergone close transformation effect. This phenomenon can be seen equally in both chemicals impregnation.

3.2. Comparative Adsorption

3.2.1. Activating Agent

Figure 1 shows the effect of different carbonization time and impregnation chemicals for the removal of methyl red in aqueous solution. Based on Figure 1, maximum percentage removal of methyl red was 97.97% using activated carbon D. Activated carbon B and F have lowest percentage removals with 86.8% and 89.34% respectively. Other activated carbon exceeded 95% of percentage removal for methyl red as shown in Figure 1. Chemical activation contributes to better pores development that enhance adsorption capability of the activated carbon produced. According to Timur et al. (2006), previous study showed that increase in activation time has same impact as increase in activation temperature. In this study, 10 minutes of activation time is not sufficient for pore developments.

Ahmad et al. (2014) claims that after activation process, generation of pores started to develop but insufficient activation time gives out incomplete pores development. Prolonged activation time give sufficient time for pores generation and in this study 60 minutes of activation is sufficient for the pores generation. According to Timur et al. (2006), zinc chloride treated activated carbon displays higher micropore volume compared to phosphoric acid activation. It proclaims that activated carbon treated with zinc chloride is significantly microporous and suitable for activated carbon production. Ahiduzzaman and Abul, (2016) also report that zinc chloride has higher surface area with higher micro and nano pores developed. This evidence supports activated carbon D for its highest dye removal percentage properties. Therefore, activated carbon D was selected for subsequent experiment.

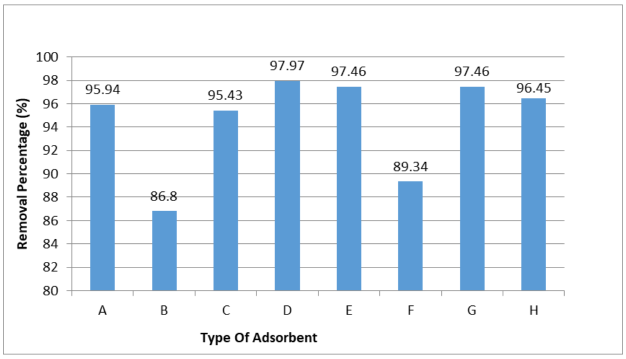

Figure 1: Effect of different type of adsorbent

3.2.2. Contact Time

The percentage removal of methyl red at different contact time was given in Figure 2. It showed that no significant difference in percentage removal with the increasing contact time. From Figure 2, 100% methyl red was removed from aqueous solution after 30 minutes and increasing contact time display constant slight reductions. The activated carbon is determined to have achieved its equilibrium state. Hameed et al. (2006) in a series of contact time experiments for methylene blue dye removal using bamboo activated carbon reported that there was a time where amount of adsorbed dye onto activated carbon achieved consistent value. This happened when the equal adsorbing and desorbing rate of the dye molecules with the activated carbon surface. Time needed to reach the state is known as equilibrium time. The dye removal percentage at the equilibrium time also proclaims the activated carbon maximum adsorption capacity under operating conditions. Therefore, 30 minutes contact time was chosen for the next experiment.

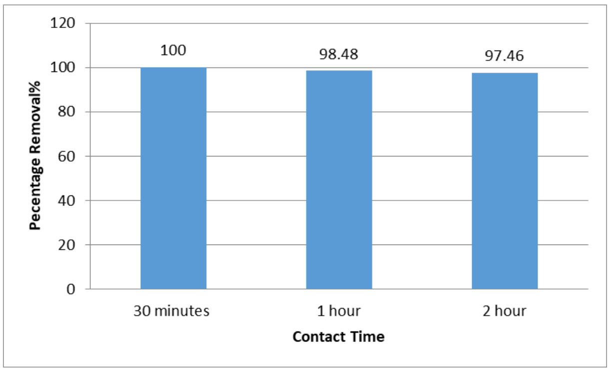

Figure 2: Effect of different contact time

3.2.3. Initial Concentration

Figure 3 showed that percentage removal of methyl red was inversely proportional to the initial dye concentration. Highest percentage dye removal was achieved in 10 ppm methyl red with 100% removal, followed by 88.32% in 25 ppm and 70% in 50 ppm solution. Research done by Mohammad et al. (2012) also showed that removal efficiency decrease with the increase of initial concentration. The activated carbon produced from rice husk has ability to achieve 99% removal using 2 ppm compared to 89.81% using 6 ppm (Mohammad et al., 2012). Similar result was reported in cationic dye removal using sugarcane bagasse activated carbon by Hazzaa and Hussein (2015). The percentage removal of dye adsorbed decreased from 88 to 55% using 10 ppm to 100 ppm. There will be unoccupied active sites on the adsorbents at low concentrations. But the active sites required for adsorption of dye molecules are not available as the initial concentration increases. Therefore, percentage removal decreases when the initial concentration increases.

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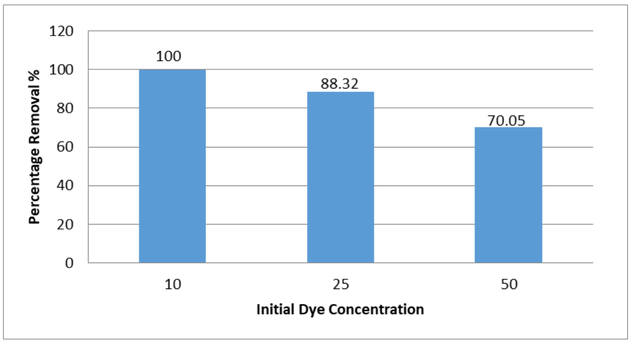

Figure 3: Effect of initial dye concentration

4. Conclusion

Based on this research, it can be concluded that activated carbon from *Parkia speciosa* pods can be used as a raw materials for the production of activated carbon with chemical impregnation. Activated carbon prepared from *Parkia speciosa* pods showed excellent efficiency in removing methyl red dye with percentage removal up to 100% at 30 minutes contact time and 10 ppm of methyl red dye solution.

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