Multidisciplinary Applied Research and Innovation Vol. 4 No. 4 (2023) 83-90
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MARI

Homepage: http://penerbit.uthm.edu.my/periodicals/index.php/mari
e-ISSN : 2773-4773

Performance of Waste Tire Activated Carbon on Methylene Blue

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DOI: https://doi.org/10.30880/mari.2023.04.04.012
Received 01 September 2023; Accepted 15 October 2023; Available online 01 December 2023

Abstract: Waste tire was chosen as a raw material to synthesize activated carbon through physical and chemical activation methods and to evaluate the performance of methylene blue treatment. Physical activation method was done by heating the waste tire in the furnace at 600˚C and the chemical activation was done by using phosphoric acid H_3PO_4. Prepared waste tire activated carbon (WTAC) was analyzed using Fourier-transform Infrared Spectroscopy (FTIR) to determine the presence of functional group and their composition. Methylene blue (MB) was chosen as a dye that can be considered as a major pollutant in textile industries' wastewater. It is a toxic and carcinogenic dye that is harmful to organisms that are exposed to or consumed. The adsorption method using WTAC was used to treat methylene blue due to its higher MB removal efficiency. In this research, methylene blue samples were treated using different dosages of WTAC (5g & 10g). Colour removal (CR%) analysis and COD analysis were done to evaluate the performance of WTAC on methylene blue treatment. 97.68% and 98.03% of colour removal (CR%) were achieved using WTAC dosages of 5g and 10g respectively. For COD analysis, 35.97% and 53.23% of COD removal (%) were achieved when 5g and 10g of WTAC dosages were used. Hence, adsorption of MB increases when the dosages of WTAC increases due to more exchangeable sites or surface areas present in the sample.

Keywords: Waste Tires, Activated Carbon, Methylene Blue, Adsorption, Colour Removal

1. Introduction

Wastewaters are released in large quantities on a daily basis from household and industrial sources all over the world, creating a range of obstacles such as a water crisis and environmental damage. As a possible answer to this challenge, the development of sustainable and energy-efficient wastewater treatment technologies is being pursued. Water pollutants and suspended particles in wastewater harm humans, animals, ecosystems, and the environment. Organic wastes from food processing, for example,

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such as protein, fat, human excrement, and vegetables, can have significant environmental effects. Bacteria breakdown biodegradable waste by consuming oxygen in the water, causing the water's oxygen content to drop. The lack of dissolved oxygen in water sources will gradually have an effect on aquatic organisms.

Dyes are extensively used in textile, paper and printing industries. In this case, Since a huge quantity of water is consumed in the dyeing and finishing processes, the textile industry is one of the greatest generators of wastewater. Textile effluents comprise both biodegradable and non-biodegradable compounds such as dyes, dispersants, levelling agents, and so on. These effluents are discharged into bodies of water, where they might alter the physical, chemical, and biological properties of the receiving bodies [1]. These coloured effluents not only obstacle light permeation and disturb the aquatic ecosystem but also exhibit toxicity towards living organisms [2]. Colorants that are water soluble or other specified substrates are referred to as industrial dyes [3]. In this case, methylene blue (MB) dye is a major contributor to waste discharged water. MB dyes are toxic and carcinogenic, exposing environmental quality at risk. Even in trace amounts, their presence in water is toxic to living organisms, impeding photosynthesis, lowering oxygen levels, and resulting in poor total oxygenation. The release of MB into waterbodies has significant aesthetic and ecological repercussions.

There are three types of dye industrial wastewater treatment processes such as biological, chemical, and physical treatments [4]. Furthermore, chemical dye removal technologies include advanced oxidation, electrochemical destruction, and Fenton reactions are less popular due to their high operating costs, toxic sludge formation, and complicated procedures [5]. These technologies use a lot of electricity to run the electrical equipment and reactors that extract the colour from the effluent. Biological dye removal methods can only remove a limited number of colours and are not suitable for all dyes [4]. Physical dye removal technology, such as adsorption, is chosen because it can efficiently remove any dye mixture from wastewater. In comparison to other biological and chemical dye removal procedures, the adsorption approach is very likely easy to operate and has a minimal operational expenses [6].

Adsorption is referred to as the separation of a material from a phase followed by its concentration on the other surface. The adsorbent is the adsorbing phase, while the adsorbate is the substance condensed or adsorbed on the surface of that phase. In this case, activated carbon is a non-toxic, solid adsorbent material that is often used to remove dissolved pollutants from water and process gas streams. The adsorption process is aided by the presence of micropores on the surface of activated carbon [4]. In wastewater treatment, activated carbon is a powerful adsorbent because it has a large surface area and pore volume, which allows the removal of liquid-phase contaminants, including organics pollutants, heavy metal ions and colors [6].

Activated carbon can be produced from waste rubber tires because it contain carbonaceous particles. Studies showed that worldwide annual production of scrap tires is about 1000 million [7]. It is now well known that tires are mixture of polymers such as natural rubber, butadiene rubber and styrene butadiene rubber, carbon black, sulphur and zinc oxide [8]. Majority of the tires consist of carbon black and this carbonaceous absorbent acts similar to activated carbon. The waste tires pose considerable risk to public health and environment and along with fire hazards, they provide good living conditions for mosquitoes, insects, rats, etc. Moreover, storage of waste tires requires large amount of space and disposal of these tires is a costly affair [2]. Therefore, use of waste tire as potential adsorbent for the removal of MB from aqueous solution is a thoughtful attempt. The aim of this study is to evaluate the performances of waste tire activated carbon on the adsorption of methylene blue from wastewater.

2. Materials and Methods

2.1 Materials

Waste tire was collected from a workshop at Pagoh Jaya, Johor. Phosphoric acid (H_3PO_4) was requested from the Shared Lab at University Tun Hussein Onn Malaysia (UTHM) while the sodium hydroxide (NaOH) was requested from Environmental Engineering Technology Lab of UTHM.

2.2 Equipments

- Drying Oven (DK-800)
- Laboratory Chamber Furnace (Carbolite Gero)
- Ultraviolet-Visible Spectroscopy (Hiatachi U-3900H)
- Fourier Transform Infrared Spectroscopy (Agilent Technologies)
- DRB 200 Reactor (Hach)
- DR6000 Spectrophotometer (Hach)

2.3 Flow Diagram of Research Activities

The overall research prosedur started from the preparation of activation of waste tire to analysis process were shown in **Figure 1** below.

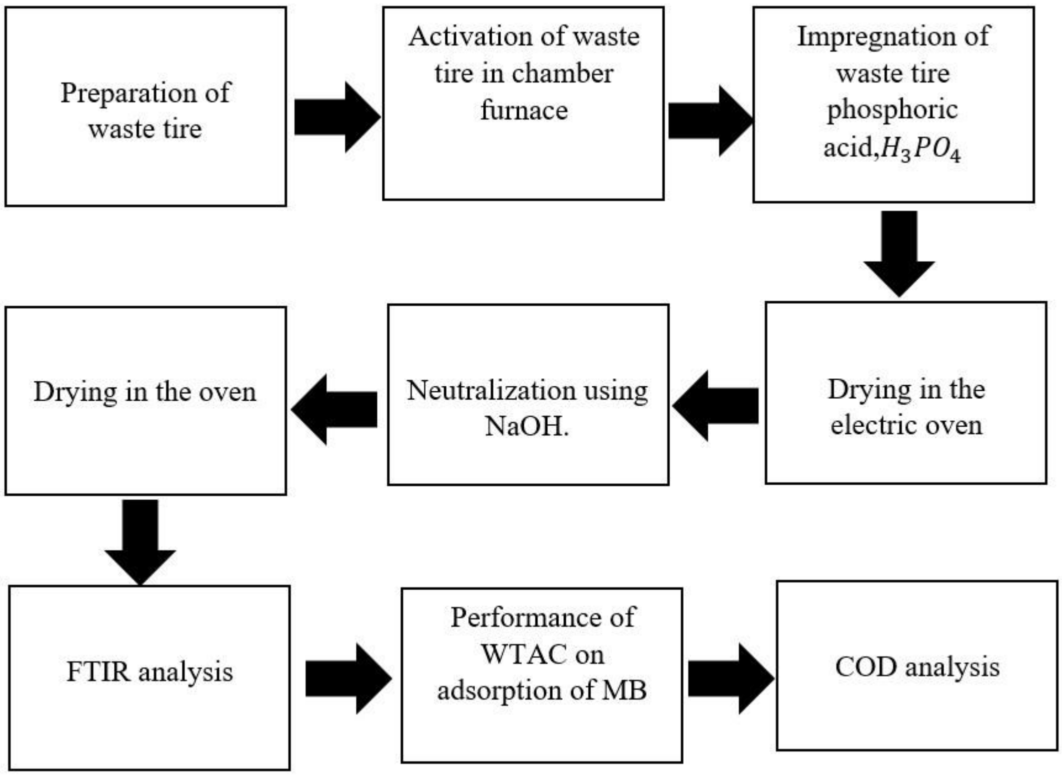

Figure 1: Flow Diagram of Research Activities

2.4 Preparation of waste tire

Firstly, shredded waste tires were washed using distilled water and fractionated to remove any residual dust or clay that may be suspended during tire miling. Then, 50g of the waste tire was dried overnight at 105˚C in the oven to remove moisture.

2.5 Synthesis of Waste Tire Activated Carbon

The dried samples were then cooked at 600˚C for 4 hours in a chamber furnace. Then, the furnace of activated carbon is left to cool to ambient temperature for about 30 minutes. After cooled, the samples were crushed using mortar and pestle until it become powder. 50g of the sample powder was mixed with 50 mL phosphoric acid (H_3PO_4) in 100 mL beaker for 8 hours. After that, the samples were filtered out using filter papers and dried overnight at 105˚C. The waste tire activated (WTAC) was neutralized using sodium hydroxide (NaOH) and the pH was measured using a pH meter. The WTAC was filtered again. Then, WTAC was dried overnight at 105˚C, cooled again and stored in a beaker to be ready for the testing of methylene blue adsorption.

2.6 Characterization of Activated Carbon

Waste tire activated carbon (WTAC) produced in the previous step undergoes characterization using Fourier-Transform Infrared Spectroscopy (FTIR) and UV-Vis Spectroscopy for a better understanding on the structure of the WTAC as well as the intensity of the light that passes through the sample with respect to the intensity of light through a reference sample or blank [9].

2.7 Colour Removal Analysis on the Adsorption of Methylene Blue

0.01g of methylene blue was dissolved in 10 mL of distilled water before being transferred to a 1000 mL volumetric flask. It was shaken and built up to the desired concentration of methylene blue solution, which will be 10 mg/L. For validation, the initial concentration of methylene blue was measured using ultraviolet-visible (UV-Vis) spectrophotometry. Various absorbent dosages (5g WTAC and 10g WTAC) were applied to 200 mL of methylene blue solution in each 200 mL duran bottle. The batch adsorption took 2 hours in an incubator shaker with a 120 rpm agitation speed at 30˚C [10]. Filtration process was carried out to separate the treated solution with the WTAC absorbent. UV-Vis was used to measure the final concentrations of each treated methylene blue solution (Ct).

2.8 Chemical Oxygen Demand (COD) Test

Chemical Oxygen Demand test was performed on wastewater treated with waste tire activated carbon (WTAC) to assess the adsorption of methylene blue using various WTAC dosages.

The digestion of the materials was carried out in batches (blank sample, pure methylene blue sample, solution treated with 5g WTAC and solution treated with 10g WTAC). The high-range (HR) vials were heated for 2 hours at 150˚C in a preheated DRB 200 reactor. The HR vials were cooled down to room temperature for 30 minutes. The HR vials were placed in the DR 6000 spectrophotometer and the COD of the treated solutions was measured.

2.9 Equation

$$CR\% = \frac{c_o - c_t}{c_o} \text{ x } 100\% \quad \textbf{Eq. 1}$$

The percentage of colour removal (CR%) from the MB sample was calculated using **Eq.1**. $c_o$is the initial concentration of methylene blue samples and c_t is the final concentration of treated methylene blue sample for each respective weight of WTAC added to the MB samples.

3. Results and Discussion

3.1 FTIR Results

The compounds of WTAC were identified in FTIR analysis. The transmittance of the compound for WTAC started from the range of 895.024 cm^{-1} to 3195.949 cm^{-1} as shown in **Figure 2.** FTIR was used to determine the spectral characteristics of activated carbon and the composition of functional groups on the surface of activated carbon samples [9]. FTIR analysis was used to study the properties of the WTAC. 5 types of carbon were analysed [9]. The transmission spectrum between 800 – 3800 cm^{-}

[1] was recorded as shown in **Figure 2**. The peak around 3195 cm^{-1} shows compound class of carboxylic acid, with functional group of O-H. The peak with 2328 cm^{-1} shows the presence of carbon dioxide in WTAC, with functional group of O=C=O with 2 double bonds. Next, the peak around 2113 cm^{-1} shows the presence of alkyne with active functional group of C≡C. Alkynes are nonpolar, unsaturated hydrocarbons with physical properties similar to alkanes and alkenes. Alkynes adsorp more strongly to catalytic surfaces and occupy reactive sites on catalyst, which indicates that its presence in WTAC will improve the adsorption of MB from wastewater [9]. Moving on to the next peak which is around 1989 cm^{-1} shows that there is presence of functional group C=C=C which is called as allene. Next, the peak around 1819 cm^{-1} shows the presence of α, β - unsaturated ester with functional group of C=O double bond. Lastly, the peaks around 895 and 1636 cm^{-1} shows that the presence of alkene in WTAC.

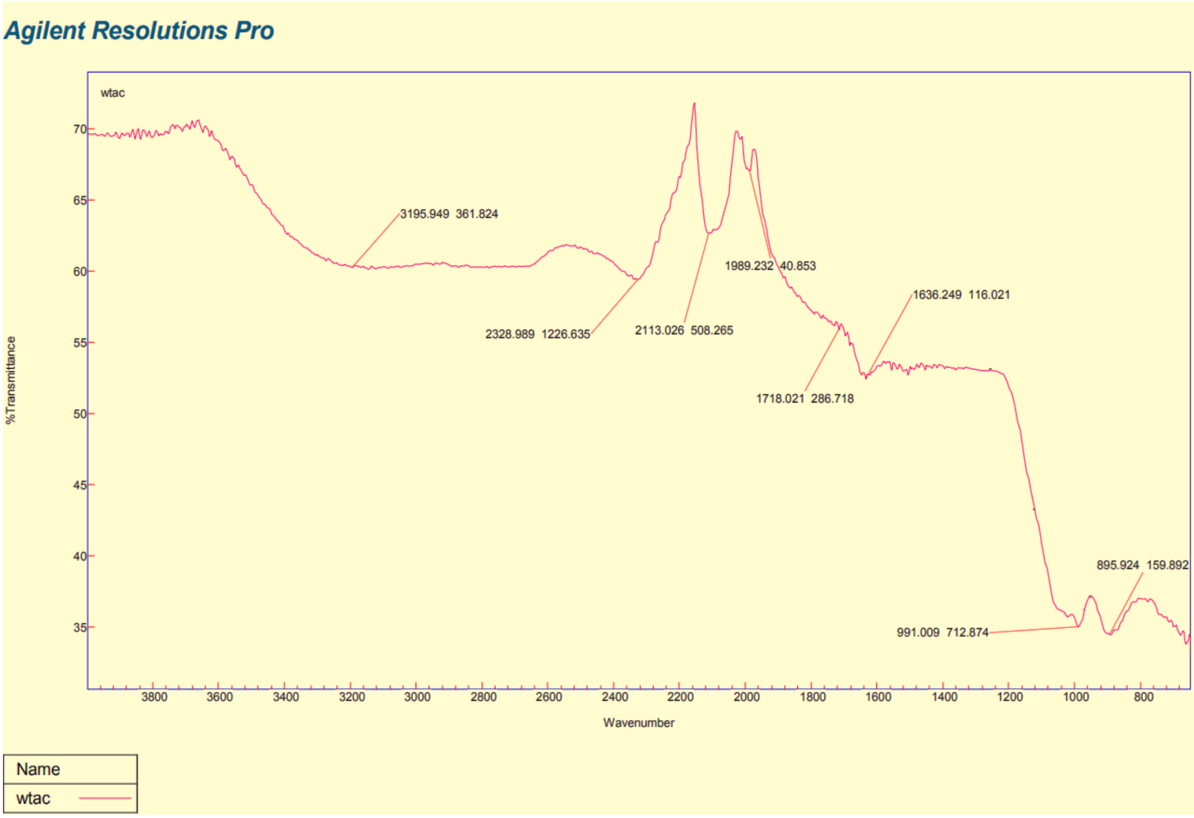

Figure 2: FTIR Spectrum for Waste Tire Activated Carbon

Table 1: Peak of FTIR Spectroscopy indicates Functional Group

Product	Highest Peak (Transmittance)	Standard Peak Reference	Functional Group	Compound Class
Waste Tire Activated Carbon	3195.949	3300-2500	O-H	Carboxylic acid
	2328.989	~ 2349	O=C=O	Carbon dioxide
	2113.026	2140-2100	C≡C	Alkyne
	1989.232	2000-1900	C=C=C	Allene
	1718.021	1730-1715	C=O	α, β – unsaturated ester
	1636.249	1650-1600	C=C	Conjugated alkene
	991.009	995-985	C=C	Alkene(monosubstituted)
	895.924	895-885	C=C	Alkene(vinylidene)

Waste tires have a high carbon content and have the potential to be processed to produce higher value and more efficient activated carbon [11]. This is because rubber carbon black compound has a higher specific surface area and adsorption number compared to the standard black [11-12]. The detailed functional group and compound in WTAC with correspond to the highest peak transmittance is shown in **Table 1**.

3.2 Color Removal Results (UV-Vis)

The determination of color removal percentage of sample in this study is very important to identify the adsorbent's capacity for a given initial concentration of MB sample and binding site available for adsorption [8]. The effect of adsorbent dosage on the removal of MB from an aqueous solution was investigated using WTAC in amounts ranging 5 to 10g. There were only 2 different dosages of WTAC available to carry on our study after the impregnation of WTAC because approximately 31g of WTAC was lost during chemical activation process. **Figure 3** shows the total percentage of color removal from MB solutions with 5g and 10g of WTAC added respectively. The percentage of color removal (CR%) for sample with 5g and 10g is 97.68% and 98.03% respectively. This test was carried out by using UV-vis spectroscopy in which the absorbance value of the light was determined. The absorbance value of light for 5g WTAC is 0.232 meanwhile for 10g WTAC is 0.197. The total percentage of the color removed was calculated using **Eq. 1**. As the weight of the WTAC increases in the MB sample, the percentage of color removal also increases. This is because when the amount of WTAC in sample increases, the adsorption process of the MB dye from the sample also increases due to increase in the presence of exchangeable sites or surface area presence of binding sites available for adsorption [7].

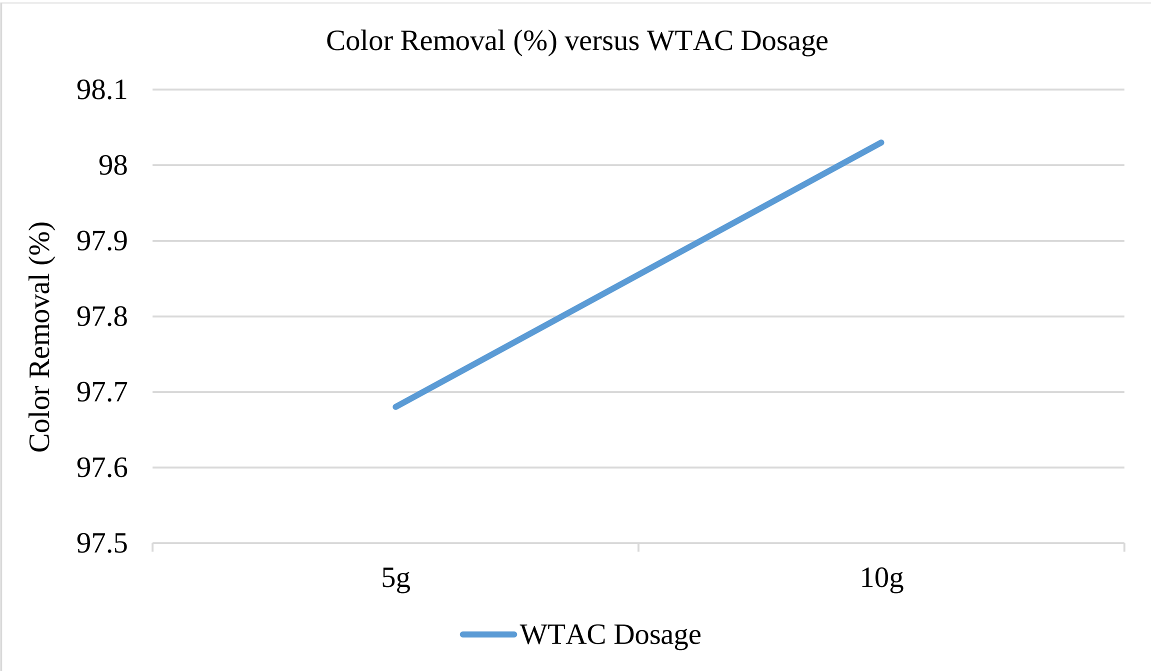

Figure 3: Percentage of Color removed from the MB Sample with respect to the Weight of WTAC added to the Samples

3.3 Chemical Oxygen Demand (COD)

Table 2 shows the Chemical Oxygen Demand (COD) values and COD removal (%) for WTAC dosage of 5g and 10g. The value of chemical oxygen demand in the treated methylene blue dye were analyzed. Distilled water was used as our reference solution which have COD value of 0 mg/L. The COD values of different dosages of WTAC were determined in this test. The COD test was carried out for high range (HR).

Table 2: Chemical Oxygen Demand (COD) of MB Samples

Item	Sample	Average COD value (mg/L)	COD Removal (%)
1	Blank (MB sample)	62.0	100.00
2	5g WTAC	39.7	35.97
3	10g WTAC	29.0	53.23
4	Distilled water	0.0	0.00

Table 2 shows the blank solution of MB sample which has 0g of WTAC, recorded as the highest value of average COD, 62.0 mg/L. Meanwhile, the MB dye which has 5g of WTAC recorded average COD value of 39.7 mg/L. The lowest COD value was recorded for MB sample which has 10g of WTAC, which is 29.0 mg/L. This shows that as the amount of WTAC increases in the MB sample, the adsorption process of the WTAC towards MB dye also increases. Since the value of COD is low when WTAC is added to MB sample, the oxygen needed to oxidize the MB dye in the sample is also become low. Therefore, when there is presence of WTAC in the sample, the COD value of the sample decreases. This is because the increasing dosages of WTAC will increase the surface area of adsorbent in sample. There will be more active exchangeable adsorption sites available to facilitate the adsorption process. A higher concentration of adsorbate which is MB dye in the sample can be adsorbed by active sites of the adsorbent, WTAC during adsorption process. During the COD test, a less amount of strong oxidizing agent was required to oxidize MB in the sample after the adsorption process. Hence, the COD value of the sample decreases when the WTAC dosage added to the sample increases.

4. Conclusion

In conclusion, activated carbon was successfully synthesized from waste tire by both physical and chemical activation method. The usage of the WTAC in removing the MB dye from samples were determined from different physical and chemical tests which are color removal analysis for physical, COD analysis for chemical test to indicate the adsorption ability of WTAC in removing MB dye which can be used in textile wastewater treatment. WTAC which was produced from waste tire was characterized using FTIR. The analytical methods of color removal and COD analysis that used in this study shows that adsorption process of MB using WTAC increase with the increasing of WTAC dosages (5g and 10g) due to more exchangeable sites or surface areas present.

The adsorption process in wastewater treatment for color removal can be improved by increasing the dosages of WTAC such as 15g, 20g, 25g, and so on. Since the production of activated carbon from waste tire is considered cheaper than normal production method, this study needs further research to increase the production of WTAC. Futhermore, Scanning Electron Microscope (SEM) can be used to characterize WTAC. Through the analysis, the morphology of WTAC before and after adsorption of MB can be observed more detailly [11]. Hence, using waste tire as an alternative to save environment is a good idea because we can prevent the annual increase of waste tire.

Acknowledgement

Author would like to thank Centre for Diploma Studies, Universiti Tun Hussein Onn Malaysia for the support and guidance.

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