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RESEARCH ARTICLE

STUDY ON ADSORPTION OF METHYLENE BLUE DYE ON LOW COST ADSORBENT DERIVED FROM WILD WEED *LANTANA CAMARA* IMPREGNATED WITH PHOSPHORIC ACID AND POTASSIUM HYDROXIDE

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ABSTRACT

Adsorption is a surface phenomenon that is used to remove both organic and inorganic pollutants. Chemical indicators, dyes, and biological dyes use methylene blue a lot. The printing and dyeing processes generate a significant amount of organic dye wastewater. The development of an adsorbent for the removal of methylene blue dye was the focus of the study, which sought to determine how various activators affected this process. H_3PO_4 and KOH activations were used to conduct the optimal batch adsorption studies of MB dye on the adsorbent. The effectiveness of MB removal was examined in relation to a number of process parameters, including the initial MB dye concentration, adsorbent dose, and contact time. AC-KOH performed good result than AC-PA in terms of MB dye removal when the adsorbent dose was 1.25 g/L. According to the contact time studies, methylene blue removal was found to be 100% at 70 minutes. When using methylene blue, the percentage of MB that is removed begins to decrease in proportion to the MB concentration. The data that was obtained during the experiment can be used with either model. Nevertheless, Langmuir Isotherm is more precise. The pseudo first order model fits well in the kinetic study.

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INTRODUCTION

Adsorption is a surface phenomenon that is used to remove both organic and inorganic pollutants. Liquid–solid intermolecular forces of attraction cause some of the solution's solute molecules to be concentrated or deposited at the solid surface when it comes into contact with a solid with a highly porous surface structure. Adsorbate refers to the solute that is retained in adsorption processes, while adsorbent refers to the solid on which it is retained (Pereira Sales *et al*., 2019). Adsorption refers to the accumulation of adsorbate on the adsorbent's surface. Adsorption technology is based on the creation of an adsorbed phase with a composition distinct from the bulk fluid phase (Nageeb, 2013). The textile, paper, pulp mill, leather, dye synthesis, printing, food, and plastics industries all make use of dyes frequently. ave the potential to cause cancer, making the water contaminated with dyes harmful to aquatic life. Dye-laden wastewater is produced in large quantities by the textile industry (Alexandro *et al*., 2011). Chemical indicators, dyes, and biological dyes all make use of methylene blue (C16H18ClN3S, MB). The printing and dyeing processes generate a significant amount of organic dye wastewater. According to Kuang *et al*., the dye wastewater has characteristics like a large discharge, high chromaticity, high organic matter concentration, and poor biodegradability. It also has a significant impact on the health of the water body and the photosynthesis of microorganisms in the water environment. 2020). Adsorption methods for the removal of dyes from wastewater have recently shown promise due to their simplicity, low cost, and efficiency.

Activated carbon is the most widely used commercial adsorbent for treating waste water; however, its production processes result in a high cost (Thang *et al*., 2021). The low-cost raw materials could be used to make activated carbon, as previous research demonstrated. Additionally, the products' characteristics significantly increase their effectiveness in adsorbing pollutants (Wang *et al*., 2022; Gecgel and other, 2013). Therefore, this study aims to establish a new activated carbon source with different pyrolysis methods from wild weed *Lantana camara* that is the non hazardous medicinal wild waste source.

MATERIALS AND METHODS

Stems of *Lantana camara* were collected from the forest area of Kolar dam, Bhopal, India. The leaves free stems were air-dried for 15 days and made into fine powder.

Preparation of Activated Carbon: In the current investigation, phosphoric acid and potassium hydroxide was used to activate *Lantana camara*. The impregnated powdered sample was placed in a 4 hour hot air oven at 100°C after being impregnated with conc. phosphoric acid and 1% potassium hydroxide at a ratio of 1:1. The sample that had been dried in the oven was held in a muffle furnace for 6 to 8 hour at 700°C activation temperature.

The obtained activated carbon washed several times with distilled water and then dried in an oven at 120 °C for eight hours.

Preparation of stock solution: A stock solution of Methylene Blue MB (ƛ max =600) (HiMedia, Mumbai, India) was prepared by dissolving 100 mg of dye in distilled water and then making up the volume to 1000 ml with distilled water. It was reserved in a volumetric flask for creating more dilute solutions. The stock solution was further diluted with distilled water to desired concentration for obtaining the test solutions. All chemicals used were of analytical grades.

Batch equilibrium method: The batch adsorptions were carried out at room temperature. The effect of contact time, adsorbent doses and the initial concentration of dye was studied. A predetermined amount of adsorbent was added to 50 ml of the dye solutions with an initial concentration of 50 to 250 mg/L. The mixture was shaken thoroughly using a mechanical shaker rotating with a speed of 120 rpm. The mixture was then filtered at predetermined time intervals and the residual concentration of the dye and metals was measured by UV-Visible spectrophotometer (Dula *et al*., 2014). The percentage removal of dye was calculated as follows:

% Removal = co – ce / co ×100

Co= initial absorbance and Ce = absorbance after adsorption

Adsorption isotherm: Two adsorption isotherm models were tested for the batch reactions which included the Langmuir and the Freundlich models. The Langmuir model assumes that the adsorbed molecules form a monolayer and that adsorption can occur at a fixed number of adsorption sites and all of them being equivalent in adsorption abilities. The Freundlich model, which supports multilayer adsorption, agrees with the Langmuir model over moderate ranges of concentrations but differs at low and high concentrations (Ayoub *et al*., 2019).

The linear form of Langmuir expression:

$$C\ e/Q\ e = 1/Qmb + Ce\ /Qm$$

The linear form of Freundlich model expression:

$$\log Qe = \log K\ f + 1/n \log Ce$$

Adsorption kinetics: Pseudo-first-order and pseudo-second-order models were used in modeling adsorption kinetics. The respective linear forms of the model equations are presented by the following equations (Manjuladevi *et al*., 2018):

$$\ln (qe-qt) = \ln qe - kf\ t$$

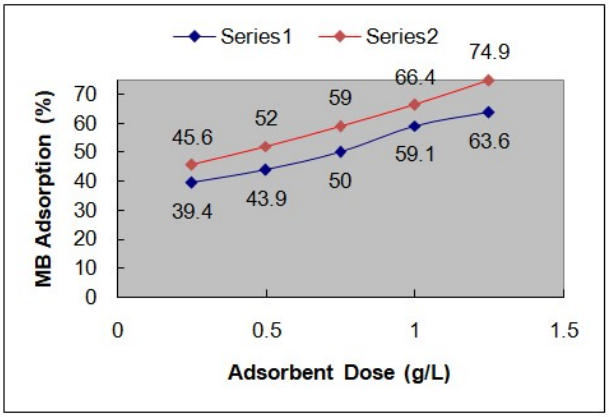
$$\frac{t}{Qt} = \frac{1}{(k_s q_e)^2} + \frac{1}{(qe)}\ t$$

where q_t is the amount of ions adsorbed at time t (mg g^{-1}), qe is the concentration of dye k_f and k_s are the pseudo-first-order and pseudo-second-order model rate constants expressed in min^{-1} and g mg min^{-1}, respectively.

RESULTS AND DISCUSSION

Figure 1 depicts how activated carbon treated with phosphoric acid and KOH influenced the adsorption of methylene blue dye. Adsorbent doses of 0.25 g to 1.25 g/L were considered, and other process parameters (pH 5.3, concentration 100.0 mg/L, contact time 30 min, and temperature 30oC) remained constant. AC-KOH was found to have the highest percentage of MB dye removed when compared to AC-PA. For both AC-PA and AC-KOH, adsorbent doses of 1.25 g/L were sufficient to achieve maximum MB adsorption within 30 minutes.

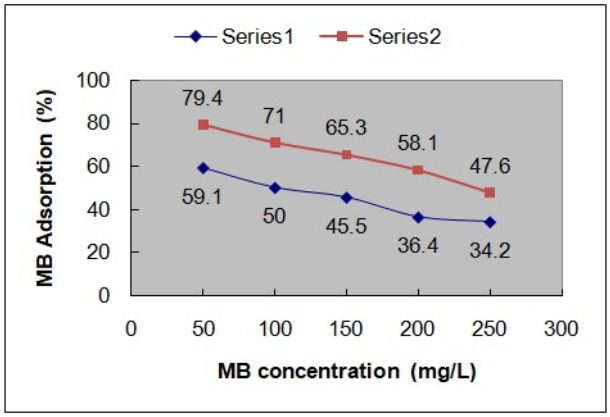
All of the adsorbents have a limited number of active sites that would have reached saturation above a certain adsorbate concentration, which accounts for the low adsorption percentage.



Series 1. KOH treated AC and Series 2: phosphoric acid treated AC

Figure 1: Effect of adsorbent dose on methylene blue dye adsorption

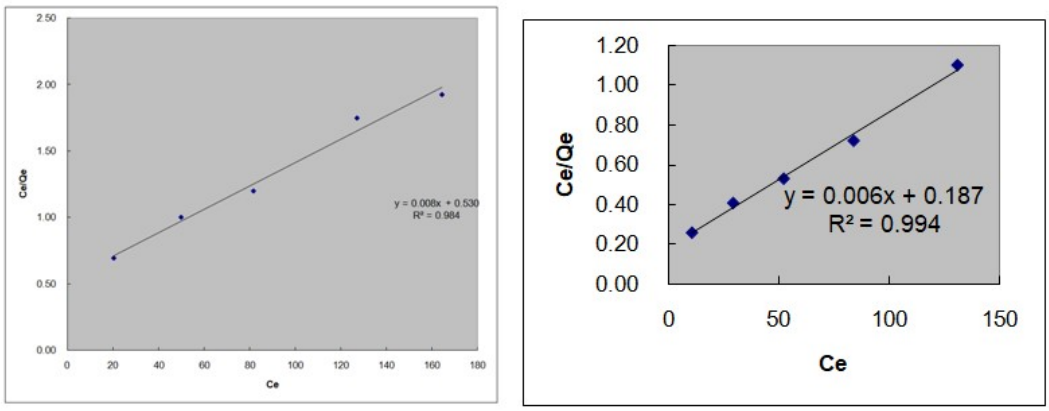
The initial concentration of MB dye in the solution is an important parameter as the dye concentration changes over a broad range in industrial effluents. Changes in the adsorption patterns of ACs are shown in Figure 2 as a function of the initial concentration of adsorbate in the solution and under specific experimental conditions. The adsorption behavior in the figure clearly shows that as the initial MB dye concentration increased from 50 mg/L to 250 mg/L, MB dye adsorption on AC-PA decreased from 59.1% to 34.2 %. The same trend was also seen for AC-KOH, which decreased from 71% to 47.6% as the dye concentration increased from 50 mg/L to 250 mg/L. However, the lack of binding sites on carbon caused a further decrease in dye adsorption. AC-KOH exhibits greater adsorption than AC-PA.



Series 1. KOH treated AC and Series 2: phosphoric acid treated AC

Figure 2: Effect of Methylene blue dye concentration on adsorption

Figure 3 shows that MB dye uptake was found to increase with increase in contact time and maximum percentage of adsorption occurred at 70 min where-after, it became constant. The initial rapid rate of adsorption may be due to the availability of the vacant active surfaces of the adsorbents for MB dye in the solution. The later slow adsorption rate could be due to the electrostatic hindrance caused by already adsorbed adsorbate species and the slow pore diffusion. In general, the initial rate of adsorption is fast, and then a slower adsorption would follow as the available adsorption sites are slowly decreased. This is due to the fact that a large number of unoccupied surface sites are available for adsorption during the initial stage and after ascend of time the remaining unoccupied surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases.

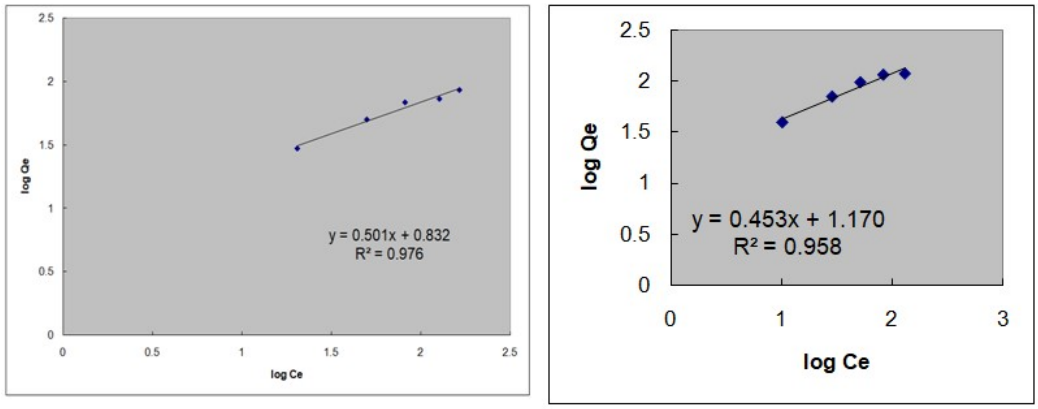


(A) (B)

Figure 4. Correlation graph for Langmuir isotherm for adsorbent impregnated with (A) phosphoric acid and (B) KOH

Table 1: Langmuir and Freundlich isotherm for the methylene blue dye adsorption of different adsorbent prepared by *Lantana camara*

Isotherm parameters	Adsorbent activated by phosphoric acid (AC-PA)	Adsorbent activated by potassium hydroxide (AC-KOH)
Langmuir		
Qm	114	167
b	0.016	0.032
R^2	0.984	0.994
RL	0.55	0.625
Freundlich		
Kf	0.83	1.17
1/n	0.5	0.45
R^2	0.976	0.958



(A) (B)

Figure 5. Correlation graph for Freundlich isotherm for adsorbent impregnated with (A) phosphoric acid and (B) KOH

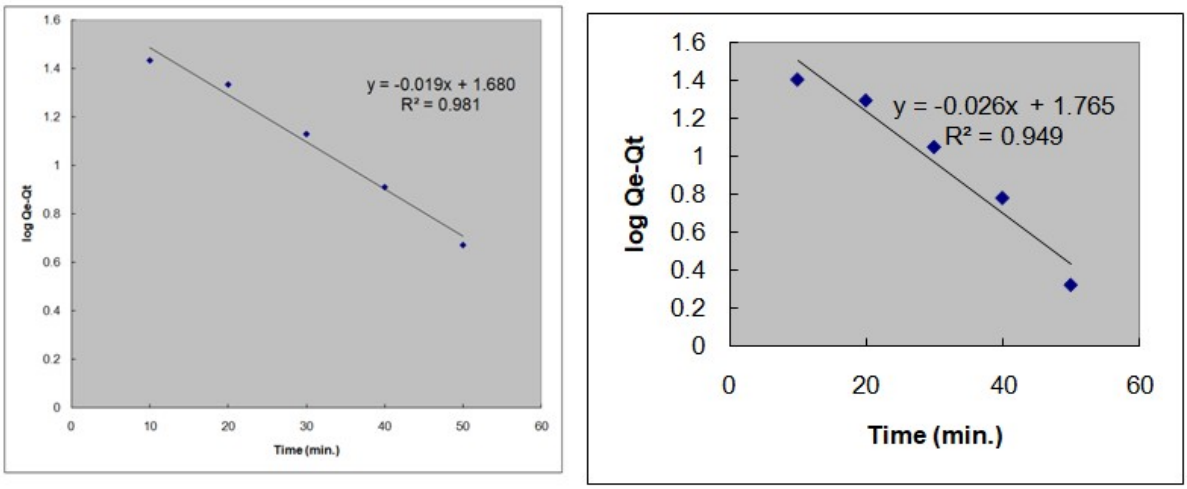


Figure 6. Fit of pseudo first order kinetics for MB dye adsorption on both (A) phosphoric acid and (B) KOH based activated carbon

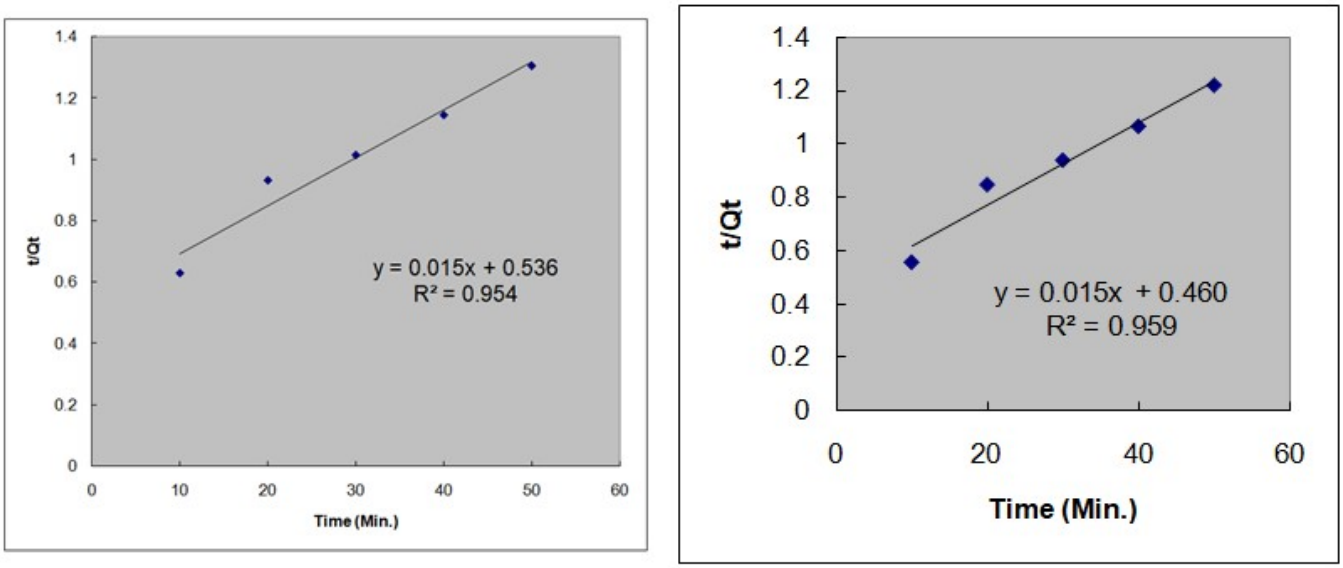


Figure 7. Fit of pseudo second order kinetics for MB dye adsorption on both (A) phosphoric acid and (B) KOH based activated carbon

Then from the results obtained the optimum contact time for adsorption onto adsorbents was fixed to be 70 min since the adsorption removal became constant after this contact time. 1 Activated carbon prepared with KOH shows highest (88.3%). Adsorption equilibrium is reached when the amount of solute adsorbed onto the adsorbent is the same as the amount desorbed. The adsorption isotherm is important for the design of the adsorption process because it explains the interaction between the adsorbate and the adsorbent. The models most frequently utilized to describe the adsorption experimental data are the Langmuir and Freundlich isotherms. These two isotherms were used to examine the MB dye's adsorption process on prepared ACs under various process parameters in this study. Reversible adsorption is described by the Freundlich isotherm, not just monolayer formation. Correlation coefficients that ranged from 0.958 to 0.976 demonstrated that the isotherms were linear. The adsorption data in this study were better matched by the Langmuir isotherm than by the Freundlich isotherm, indicating that the ACs are heterogeneous. Table 1 provides a summary of the Langmuir and Freundlich adsorption isotherm constants that were determined for each AC. The present study attempted to analyze the above mentioned isotherm parameters at 25 °C and the correlation coefficient R2 were calculated by fitting the experimental equilibrium data for MB dye on all adsorbents using both Langmuir and Freundlich isotherms, which are presented in Table 1. The results clearly showed that the adsorption of MB on adsorbents fits the Langmuir model best. The fact that the Langmuir model is a good fit suggests that physical adsorption as well as a heterogeneous distribution of active sites on the adsorbents surface exists. To evaluate the effluent adsorption method's mechanism and effectiveness, a study of adsorption kinetics is essential (Senthilkumaar *et al*., 2005). This study used pseudo-first order and pseudo-second order kinetics models to explain how the MB dye adsorbs on activated carbon samples. Figures 6 and 7 show the rate constant studies for various initial dye concentrations carried out with the pseudo-first-order and second-order models. The calculated equilibrium adsorption capacity and correlation coefficient of the pseudo-first-order adsorption model are both high. The calculated values match the results of the real-world experiments, assuming that a pseudo-first-order model fits the MB adsorption on carbon materials that have already been prepared. The pseudo second order model rate constant (k2) that was determined by the pseudo second order model exhibited the following order in terms of the type of the activator: KOH is prevails over phosphoric acid. This trend suggested that carbon materials activated with KOH for MB removal had a higher rate of adsorption. Consequently, it is reasonable to assume that MB adsorption is controlled by the pseudo-first-order kinetic model.

CONCLUSION

The current investigation came to the conclusion that, in comparison to acid, the adsorbent activated with KOH exhibited superior MB dye adsorption. According to the contact time studies, the percentage of methylene blue removed is good after 70 minutes and eventually stays the same after a very long time. This pattern can be explained by the fact that the adsorbate-adsorbent interaction gets stronger the longer the contact time, resulting in more adsorption and a higher percentage of MB removed per contact time. It was observed that the percentage of methylene blue removed rises with adsorbent dosage. This trend can be attributed to increased dosage; as the surface area exposed to dye solutions increases, more dye can be adsorbed into the adsorbent's pores, resulting in a higher percentage of MB removal. When using methylene blue, the percentage of MB that is removed begins to decrease in proportion to the MB concentration. The data that was obtained during the experiment can be used with either model. Nevertheless, Langmuir Isotherm is more precise.

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