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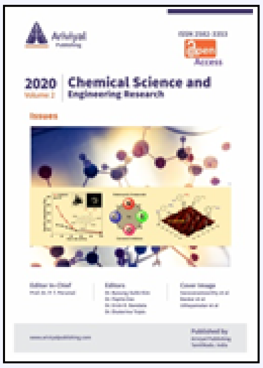

Adsorbent Activity of Activated Carbon Obtained from Dried Leaves of *Millettia pinnata*: A Colorimetric Approach

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Abstract: Water decontamination using adsorbent materials has been studied extensively and reported in the literature. Readily available and less expensive material can be used to prepare efficient adsorbents. Dried leaves of *Millettia pinnata* are not only readily available but also rich in hydrocarbon content. This stimulated us to prepare activated carbon from *Millettia pinnata* and investigate its adsorbent activity using colorimetry. In this present study, the adsorption efficiency of methylene blue on commercial and test carbon using batch adsorption process is reported. Effect of adsorbent dose and contact time are analyzed and Langmuir's adsorption isotherms constructed. It is observed that as the concentration and contact time of adsorbent increases, there is a marked enhancement in the percent adsorption of methylene blue. This is confirmed in colorimetric spectra of both commercial and test carbon.

Keywords: Adsorbent; *Millettia pinnata*; Adsorbent dose; Contact time; Langmuir isotherm; Methylene blue

1. Introduction

Water, air, fire, and earth are the classic elements gifted to humans for survival. But as a result of evolution in mankind, these elements are being affected one way or the other. The balance of our environment is getting disturbed causing many ecological hazards. Water pollution is one of them.

A Central Pollution Control Board (CPCB) report of 2015 brought out the fact that 61,948 million litres of urban sewage are generated daily in India. Assessment of water quality and streams revealed that the aquatic resources are polluted due to the discharge of treated and untreated wastewater from municipal and industrial resources.[1,2]

The wastewater may contain heavy metals, chemicals, dyes, oils, microbes, and many other harmful substances. Among these contaminants, the presence of a small amount of dye in water is highly visible and undesirable, as they have complex non-biodegradable organic structures causing serious effects on the ecosystem.[3] Decontamination of water became the priority to many countries, including India, and research strategies adopting various methods to remove dyes from wastewater such as adsorption, ion exchange, reverse osmosis, ultrafiltration, and chemical degradation are in practice.[4-6] Adsorption is a very popular and widely applied technique for removing dye contaminants using various adsorbents like activated carbon, bentonite, sepiolite, zeolite, kaolin, coal, wood, flyash, etc.[7-9] Among all adsorbents activated carbon exhibits good adsorption due to its large surface area and microporous nature.[10] Though adsorption with activated carbon is a very efficient technique for the removal of a wide range of dyes from industrial effluents, the disadvantage is the cost of adsorbent preparation which makes it economically infeasible.[11,12] To overcome this financial hurdle, usage of various novel materials as adsorbents to replace commercial activated carbon with relatively cheaper alternatives like plant materials are being used, for example, peels of Citrus aurantium, Citrus lemoni, Achras sapota, Musa paradisiaca, etc.[13-22]

Millettia pinnata, also known as Pongamia pinnata, is widely distributed throughout the world. Almost all parts of this plant are used in Ayurvedha and herbal medicines. The leaves are rich in alkaloids, carbohydrates, phytosterols, saponins, tannins, and flavonoids. Besides its medicinal properties, the plant is familiar for its biodiesel and eco cleansing activity. It is well known for its large surface area with 31% carbon content.[23-27] Literature survey, all facts about *Millettia pinnata* and in continuation of our earlier works based on adsorption, present study which focuses on emphasizing the adsorption efficiency of carbon prepared from dried leaves of *Millettia pinnata* was carried out.

2. Experimental Section

2.1. Preparation of Test Carbon

Fresh leaves of *Millettia pinnata* were collected, shade dried for 6-7 days, crushed into small pieces. The impurities from the leaves were picked out. Crushed leaves were kept in the microwave oven and charred at 90-150^{o}C until the leaves turned into charcoal. The prepared charcoal was passed through a sieve (No-44) and made into a fine powder using mortar and pestle. Then it was stored in an airtight plastic container.

2.2. Preparation of Dye Solution

A stock solution of 1000 µg/ml was prepared by dissolving 1.0 g of methylene blue of analytical grade in 1000 ml volumetric flask and made up to mark with distilled water. Different concentrations were prepared by diluting the stock solution with suitable volume of distilled water.

2.2.1. Preparation of Calibration Curve

Various concentrations in the range of 2-10µg/ml were prepared from a stock solution in 10 ml volumes with distilled water as the solvent. Prepared aliquots absorbance was measured using UV-Visible spectrophotometer (Shimadzu UV-1800; UV Probe 2.34). The concentration of dye was estimated using the line equation obtained in the calibration curve.

2.3. Batch Adsorption Study

To determine the effect of adsorbent dose on adsorption, variable amounts (2-10g) of prepared carbon (adsorbent) was mixed with 100ml of 10µg/ml dye solution in conical flasks. This mixture was stirred continuously on a magnetic stirrer for pre-determined time intervals (10-60 min) at a constant speed (2500 rpm). The filtered solution was subjected to colorimetry and concentration measured by using the calibration curve. Similarly, the effect of contact time (10-60 min) with a fixed concentration of dye and prepared carbon adsorption efficiency was investigated. Finally, the percentage of adsorbed was calculated using the Equation 1.

$$\% \ Adsorbed = Ao - At/ \ Ao \qquad (1)$$

Where, Ao = initial absorbance of the dye solution

At = absorbance of the solution after the adsorption

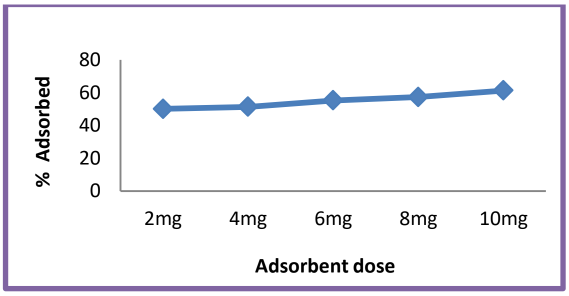

Fig. 1. Effect of Adsorbent Dose on Adsorption

Table 1. % Adsorption of Methylene Blue Dye at Various Doses of Adsorbent with Contact Time 30 Min and Dye Conc 10 µg/ml

Concentration of charcoal	2mg	4mg	6mg	8mg	10mg
% adsorption	50.1	51.4	55.3	57.3	61.2

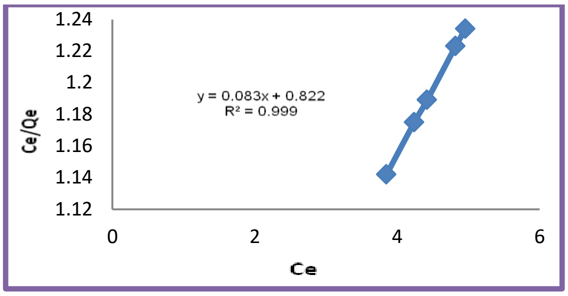

Fig. 2. Langmuir Model for Effect of Dose on Adsorption

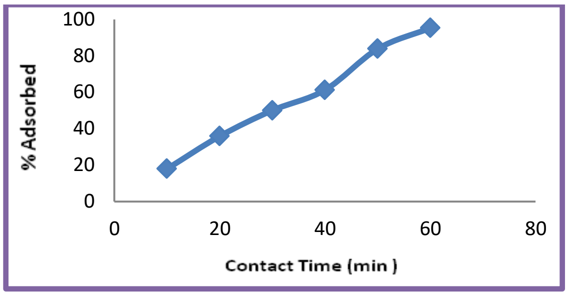

Fig.3. Effect of Contact Time on Adsorption

2.4. Adsorption Equilibrium

Adsorption equilibrium was described by Langmuir isotherm using Equation 2.

$$Ce/Qe=1/(Q_{max}.KL) +Ce/Q_{max} \qquad (2)$$

Where,

Qe= content in the adsorbent at equilibrium

Ce=equilibrium concentration

KL=Langmuir constant

Q_{max}=maximum content in adsorbent

2.5. FT IR Spectroscopic Analysis of Millettia pinnata

FTIR spectrum helps in the identification of functional groups. FT IR spectra of prepared carbon from dried leaves of *Millettia pinnata* were recorded in KBr by FTIR spectrometer (Shimadzu IR Spirit T; Lab solutions IR).

3. Results and Discussions

3.1. Effect of Adsorbent Dosage

The optimum adsorbent dosage is one of the important parameters that affect the amount of adsorbed adsorbate. The surface area increases with increasing adsorbent dosage. Finding an optimal dosage is necessary to avoid excess consumption of adsorbent. The effect of adsorbent dosage for the adsorption of methylene blue by

Table 2. % Adsorption of Methylene Blue Dye at Various Contact Time Intervals with 2mg Adsorbent Dose and Dye Conc 1 µg/ml

Contact time	10min	20min	30min	40min	50min	60min
% adsorption	17.9	35.8	50	61.2	83.7	95.2

Table 3. Langmuir Adsorption Coefficient for Adsorption of Methylene Blue at Different Doses of Adsorbent Time Intervals with Contact Time 30 Min and Dye Conc 10 µg/ml

Concentration of charcoal	2mg	4mg	6mg	8mg	10mg
Ce	4.96	4.82	4.42	4.24	3.85
Qe(mg/g)	252	129.5	93	72	61.5
Ce/Qe	1.234	1.223	1.189	1.175	1.142

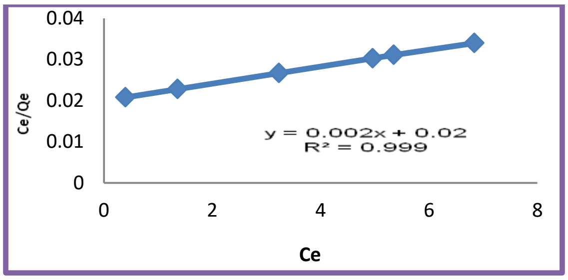

Fig. 4. Langmuir Model for Effect of Contact Time on Adsorption

activated carbon obtained from dried leaves of *Millettia pinnata* was found to increase by increasing the adsorbent dose. This is probably due to the increasing number of accessible active sites of the adsorbent. The adsorbent was found to have equilibrium concentration (50% of dye removal) at initial dye concentration i.e 2mg (Table 1, Fig. 1 and Fig. 5).

3.1.1. Application of Langmuir Isotherm for Effect of Adsorbent Dosage

The Langmuir isotherm takes an assumption that adsorption occurs at specific homogeneous sites within the adsorbent. The adsorption isotherm of methylene blue shows that the data fitted Langmuir isotherm. The Langmuir constants Qm and K are evaluated from the slope and intercept of the linear equation and R^2 value is less than 1 (Fig 2). This indicates that the surface of prepared charcoal is homogeneous in terms of energy and promotes monolayer adsorption.

3.2. Effect of Contact Time

Adsorption of methylene blue on activated carbon prepared from *Millettia pinnata* increases with increasing contact time (Table 2, Fig. 3, and Fig. 6). From this, it is evident that contact time plays a crucial role in the adsorption process as time increases from 10-60 min the percentage adsorption also increases 17.9 - 95.2. The adsorption rate was a little slow within the first 20 min then increases rapidly. Beyond 90% there was no significant increase in the adsorption rate. It is thought that the first step of adsorption involves surface adsorption and the second step intra particle transport from bulk fluid to the external surface of the carbon.

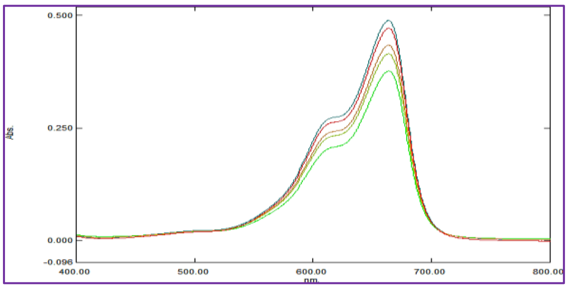

Fig. 5. Effect of Adsorbent Dose on Adsorption

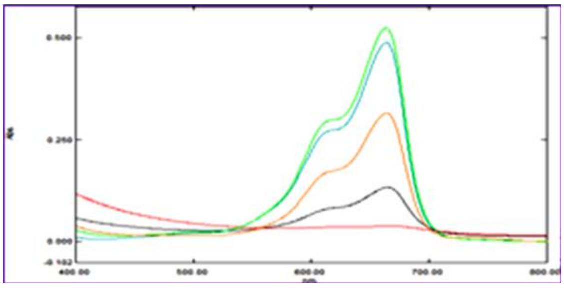

Fig. 6. Effect of Contact Time on Adsorption

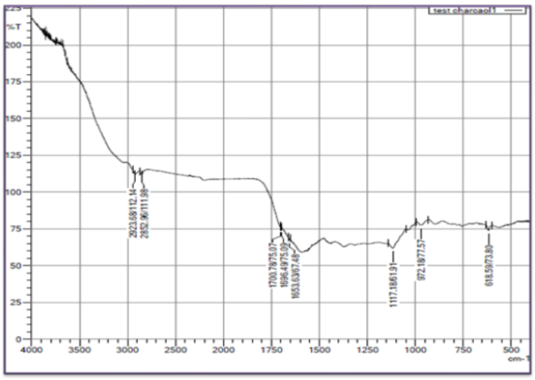

Fig. 7. FT IR Spectrum of Carbon Prepared from Dried Leaves of Millettia pinnata.

Table 4. Langmuir Adsorption Coefficient for Adsorption of Methylene Blue at Different Contact Time Intervals with Contact Time 30 Min and Dye Conc 10 µg/ml

Contact time	10min	20min	30min	40min	50min	60min
Ce	6.84	5.35	4.96	3.23	1.36	0.4
Qe(mg/g)	158	232.5	250	335	430	480
Ce/Qe	0.034	0.0311	0.0303	0.0267	0.0228	0.0208

3.2.1. Application of Langmuir Isotherm for Effect of contact time

The adsorption equilibrium was reached in 30 min with the isothermal data fitted better in Langmuir model. The R^2 value for Langmuir model for the effect of contact time in being less than 1 explains the monolayer distribution of dye on the surface of adsorbent at each time interval (Fig. 4).

3.3. FTIR analysis

FT IR provides information on the functional group constitution of a compound. FT IR spectra of prepared test carbon confirmed the presence of functional groups (Fig. 7). The absorption bands occurring at 2923cm^{-1}, 2852cm^{-1} corresponds to C-H alkane and aldehyde stretching. The bands observed at 1700 cm^{-1}, 1696 cm^{-1} signifies the presence of C=O group of ketone, amide and a weak band at 1653 cm^{-1} is due to C=C alkene stretching and absorption band at 1117 cm^{-1}, 972 cm^{-1}, 618 cm^{-1} may be due to presence of C-O, out plane bending of C-H bond and halogen (C-X) functional groups. These findings indicate the presence of alkane, alkene, aldehyde ketone, esters functional groups in small proportions does not affect the adsorption efficiency of test carbon.

4. Conclusions

The present study confirms the adsorption potential of activated carbon prepared from dried leaves of *Millettia pinnata*. The adsorbent dosage had a positive effect on the adsorption process of methylene blue. The adsorption equilibrium was reached in 30 min. The rate of adsorption is in harmonious with the experimental parameters like dose and contact time and Langmuir isotherms reveal that prepared carbon is homogenous providing monolayer adsorption.

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Conflicts of Interest

The authors declare no conflict of interest

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