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Efficient Abatement of Methylene Blue Dye by Use of Newly Derived Adsorbent from the Bark of Pongamia Pinnata

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Abstract:
Methylene blue (MB) dye is a harmful pollutant commonly found in industrial wastewater and poses a significant threat to the environment and human health. Adsorption using activated carbon is encouraging approach to eliminate Methylene Blue dye from contaminated water. The aim of this work is to explore of the removal of MB dye by utilizing newly prepared activated carbon derived from *Pongamia pinnata* bark. The activated carbon was prepared through carbonization method and characterized using FTIR spectroscopy, XRD, TGA and SEM studies. The adsorption experiments were conducted by changing parameters viz. contact time, initial dye concentration, pH, and adsorbent dose. The optimum removal of 94.5% was found at an optimal pH of 6.5, contact time of 110 min, and adsorbent dose 4.0 at room temperature. The adsorption data fitted well to the Freundlich isotherm model, indicating a multilayer adsorption process. The findings suggest that the newly developed activated carbon is a highly effectual and sustainable adsorbent for the elimination of methylene blue dye from wastewater. This study highlights the future of activated carbon derived from bark of *Pongamia pinnata* for the treatment of dye-contaminated water.

Keywords: Methylene blue dye, Activated Carbon, Adsorption, Bark of Pongamia pinnata

Introduction:

“Water contamination is substantially caused by the discharge of domestic, urban and industrial wastes including textile industry which is considered to be one among the topmost polluters. Dyes are organic compounds widely used in the textile, paper, food, plastics, leather, pharmacology and printing industries and one of the major toxic chemicals discharged from these industries”[1-3]. “Textile wastewater are colourful because of mixing of unfixed dyes and these dyes must be isolated from the wastewater due to less than 1mg/L can be visually detected”[4]. “The nonstop disposed of dyes into submarine sources cause of severe health issue which destroys the ecosystem. These dyes are exceptionally toxic, mutagenic, and cancer causing in nature”[5–8]. “Amongst the different types of dyes, methylene blue (MB) is a cationic type dye used in the textile. MB dye used in the silk, wool, paper, and acrylic industries”[9,10]. The deliverance of MB dye in the environment cause aesthetic issues and diverse effects on aquatic life [11] Although medicinal applications are common, the use of this substance can lead to various health complications like eye damage, vomiting, gastritis, breathing difficulties, nausea, mental confusion, skin cancer etc [12]. Methylene blue is also largely carcinogenic in living organisms. It is important to eliminate from waste water before squeezing off into the environment. There are various standard procedure have been studied for removal of MB dye from water. All of these methods are adsorption [13-17], classical and chemical oxidation [18-22], membrane separation [23,24] and coagulation [25-27]. Amongst all these methods, adsorption method is efficient method for removal of dyes, organic compounds and oils from water and wastewater [28]. Most of the industries generally used activated carbon as adsorbent for removal of dye. Lot of studies have been directing to development of activated carbon materials from low cost and easily available waste materials such as barks, coconut shell, lignite or coal, but for the development of carbon adsorbent, carbonaceous material almost used.[29,30]. A broad range of carbon have been synthesized from agricultural waste such as sugarcane pulp [31,32,33],corn cob and fly ash[34-37], maize cob[34,38], rice hulls, fruit stones and nutshells [39], sawdust [40], coir pith [41,42], banana pith [43,44], rice husk [45-47] for removal of various types of dyes. ***Pongamia pinnata***, the scientific name for karanj tree, and found in several area of forest in Chandrapur district. The bark of tree used for cooking purpose. Burning waste bark is discarded as waste into the environment without considering its significance. Activated Carbon from these natural sources will be the most economical, and it can then be applied to the ever-present issue of water pollution. The aim of this work is to use the adsorption method to remove MB dye from the wastewater. Nitric acid-activated carbon was used as an adsorbent, termed PPAC. Bark of ***Pongamia pinnata*** was collected from local forest area of Chandrapur district. This specific adsorbent was chosen due to its cost-effectiveness, eco-friendliness, abundant availability, and sustainability compared to conventional activated carbon and other biomass sources. The novelty of this work is to use of the waste materials into novelty product to eradicate water pollutants for the benefit of environmental conservation. This work discusses in detail the adsorption ability of the PPAC to eliminate MB dye from the aqueous solution where batch experiment studies are performed to effective adsorption parameters. Various characterization techniques, including FTIR, SEM, XRD, TGA characterized nature adsorbent. Adsorption kinetics, Isotherm, and thermodynamic studies were also carried out. The regeneration efficiency of the adsorbent was also discussed in detail. According to our knowledge and from the literature review, it is the first attempt to use PPAC to effectively remove MB dye by Nitric Acid (HNO_3) activation .

2. Materials and Methods

2.1 Chemical

For the preparation standard solution of MB, analytical grade Methylene blue were used. For the experimental study, 1000 mg/L of stock solution was prepared and the desired MB dye concentrations could then be synthesized from the stock solution via consecutive dilutions. Potassium hydroxide and sodium hydroxide were procured from Gloabal Marketing, Nagpur.

2.2 Synthesis and activation of Carbon Material (PPAC)

***Pongamia pinnata* bark** was assembled from the native forest. The bark was broken into pieces and rinsed with water again and again to eliminate the sandy material & dried in sunlight for 5-6 days. The dried material put to pyrolysis technique for carbonization under Muffle Furness at 750-800^{0}C for 7-8 hrs in order that volatile matter had been taken out and residuo was transferred right into a char form . The Char term as Carbon. The char was then subjected to activation by using acid treatment. The acid treated activated carbon was prepared by adding 100gm of carbon powder into 100 ml of 0.5M of nitric acid in 500 ml beaker for 24 hour. The content in the beaker were filtered through whatmann filter paper no.41 and residue carbon was then washed with several time distilled water so that no more acid was left and dried in oven at 90^{o}C for one day and store in bottle.

Fig.1 *Pongamia pinnata* tree

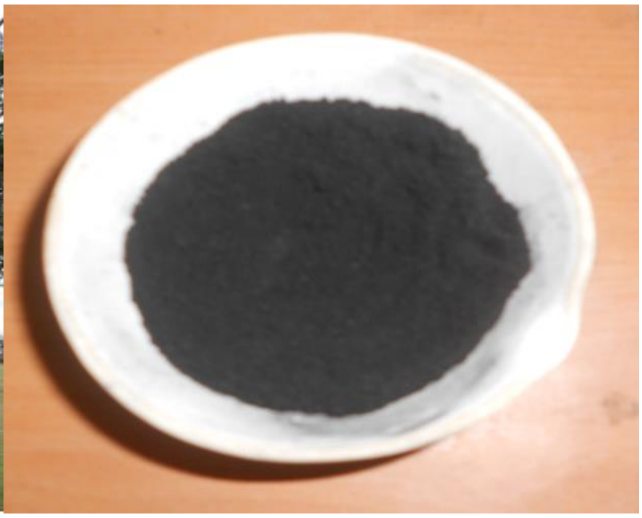


Fig.2 Activated Carbon of PPAC

2.3 Characterization of Adsorbent:

The Surface of PPAC material were analysed using scanning electron microscope (model JEOL 6390LV) for SEM analysis ; Fourier Transform infrared spectrometer (FTIR)(model : Thermo Nicolet Avatar 370) analysis was perform to display the various functional group associated with adsorbent and X-ray Diffractometer was used for X-ray powder diffractograms. FTIR analysis used for distinguish the different functional groups employing a spectrometer.

2.4 Adsorption Experiment

In the recent study, batch experiment were conducted for the adsorption process. Batch experiment was carried out by taking appropriate quantity of PPAC in 50 ml of the MB dye solution (100 mg/L) in conical flask. The pH of solution was maintained by use 0.1M Hydrochloric acid & 0.1 M sodium hydroxide. The solution was put on rotatory shaker for shaking purpose for 1 hr. Then after solution was filtered and the remaining concentration of MB were calculated using AAS. The experiments were carried out to find effect of different parameters on MB removal like, solution pH (2.0-10.0), contact time (10-150 min), initial MB dye concentration (10-100 mg/L) and adsorbent dosage (0.5- 6.00g). The adsorption capacity q_t (mg/g) was calculated using equation (1) and the percent removal was calculated using equation (2) equation :

$$q_t = \frac{(C_0 - C_e)\,V}{W} \quad \text{----------------------------------A}$$

$$R_e = \left(\frac{C_0 - C_e}{C_0}\right) X\, 100 \quad \text{--------------------------------------B}$$

where q_t (mg/g) is the adsorption capacity of adsorbent for MB at time t (min) and C_0 and C_e (mg/L) are the initial and equilibrium dye concentration in solution respectively. V and W are the volume of the solution and the weight of PPAC used respectively .

2.5 Adsorption isotherms

There are different adsorption isotherms namely: Langmuir, Freundlich, BET, Temkin, Dubinin-Radushkevitch, Hill, Redlich-Peterson and Sips [48]. But on this study, we hired handiest two, Langmuir (equation C) and Freundlich (equation E) isotherms as they may be the maximum typically used theoretical adsorption isotherm models. These isotherms expose the effect of initial concentration of dye and temperature at the adsorption process. We reckoned the adsorption capacities at equilibrium (q_e) from Eq. (D) and (F) for Langmuir and Freundlich isotherms, respectively. "To acquire this, we used 100 mL beakers to prepare 50 mL volume of MB solutions of varying concentrations, the maximum initial concentration of 100mg/L. The pH of the dye solution was maintain at 6.0 by using 0.1 M HCl and NaOH 5 gm of PPAC added to its and stirring for 5 hr. When equilibrium attained, the solution had been then filtered. The residual MB dye concentration in the filtrate was measured using AAS. By using eq. (D) and eq. (F), result were calculated to good fit of both adsorption isotherm model." [49]

Langmuir Isotherm equations

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \text{ --- C}$$

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \text{ -----------------------------------D}$$

Freundlich Isotherm equations

$$q_e = K_f \, C_e^{1/n} \text{ -----------------------------------E}$$

$$ln \, q_e = \ln K_f + \frac{1}{n} C_e \text{----------------------------F}$$

Equilibrium relationship equation

$$q_e = \frac{(C_0 - C_e)V}{W} \text{-------------------------------- G}$$

In the equations(G), q_e indicated the adsorbed dye per unit mass of PPAC (mg/g); q_m is the maximum adsorbed dye molecule per unit PPAC (mg/g),; b represents the Langmuir adsorption equilibrium constant and is also known as K_L the equilibrium constant (L/mg); K_F is the adsorption capacity (L/mg); 1/n is the adsorption intensity (g/L)

3. Result and Discussion

3.1 Characteristics of PPAC

SEM Analysis of PPAC: SEM photographs of PPAC adsorbent have been shown in **Fig.3.1**. These SEM micrographs have been obtained by applying an accelerating voltage of 10.0 kV at x500, x2000, x4000 magnifications. From the images, regularly porous and rough surface of the adsorbent can be understood which is suggestive of very high surface area. The meso-porous dimension is found to be 8.00 to 8.30µm. The presence of holes and cavities between the layers within the structure of PPAC shown at high magnification. The surface structure of PPAC appears to have irregular crystalline form. The surface with small numerous pores provides ease for accumulation of metal ions which may prove it has better adsorbent. The surface of PPAC is peculiar having linear accumulation pattern and exterior edges on the surface with oval pores pattern.

Fig 3.2 is FTIR of PPAC. The appearance of broad absorption peak at 3438.57cm^{-1} connected to –OH stretching vibration of water and -OH and NH-stretching vibration of free amino groups. The broad peak is due mixing of bands of –OH and –NH group. FTIR peak of PPAC shows specific signal at 1612.03 cm^{-1} (strong absorption) and 2284.47cm^{-1} (weak absorption), which may be assigned to the acylamino group and S-H vibration respectively. The peak at 1314.26cm^{-1} indicate C-H and O-H deformation vibrations. Another absorption band at 1230cm^{-1} could be attributed to C-OH stretching. A peak at 779.13cm^{-1} represent -CH_2 rocking .

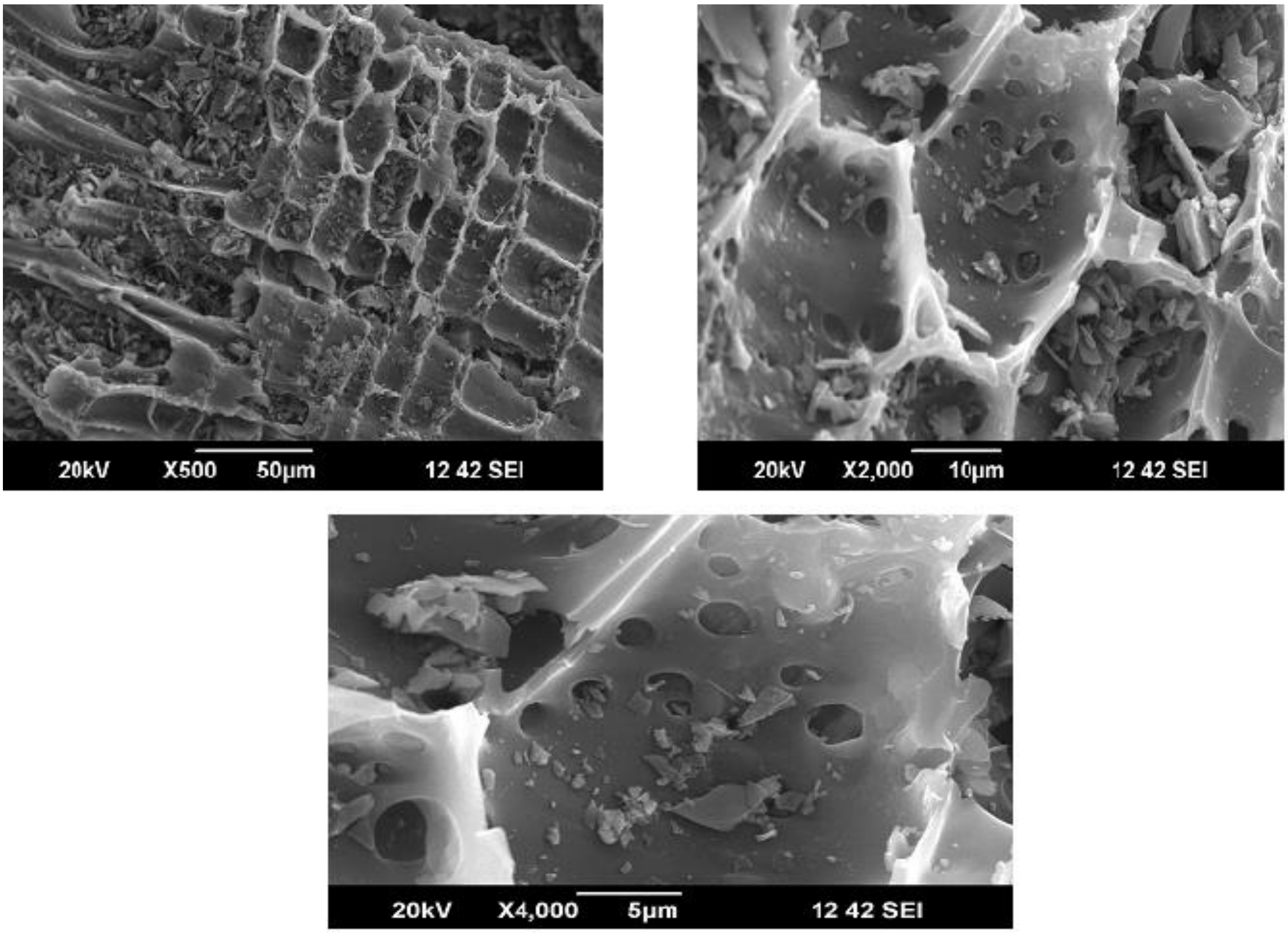


Fig.3.1 SEM of Activated Carbon of PPAC

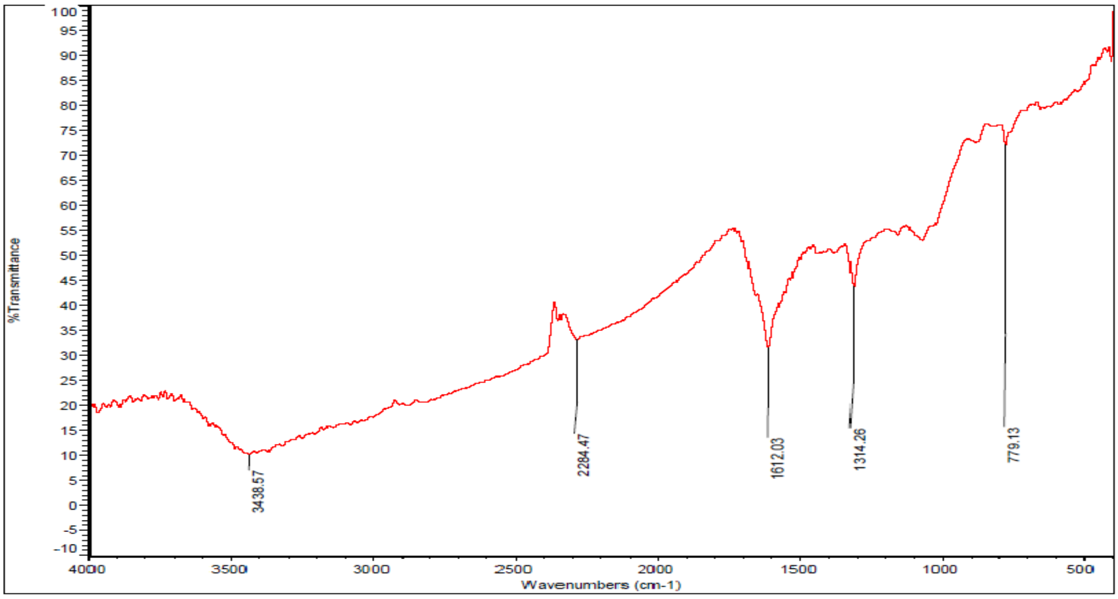


Fig.3.2 FTIR Spectrum of Activated Carbon *Pongamia pinnata* (PPAC)

XRD analysis of PPAC: Fig 3.3 show X-ray diffraction of PPAC. XRD view of PPAC has two broad diffraction peaks at $2\theta = 29^0$ and 43^0 in spectrum corresponding to the diffraction of (002) and (100) respectively. The peak appearance at around 24^0 signifies regularity of crystalline structure. The peak around

$2\theta = 30^0$ to 35^0 indicates the presence of silicon dioxide (minerals) as impurity entrapped in the adsorbent material. The sharp peak at 15^0 is again evidence towards high crystalinity.

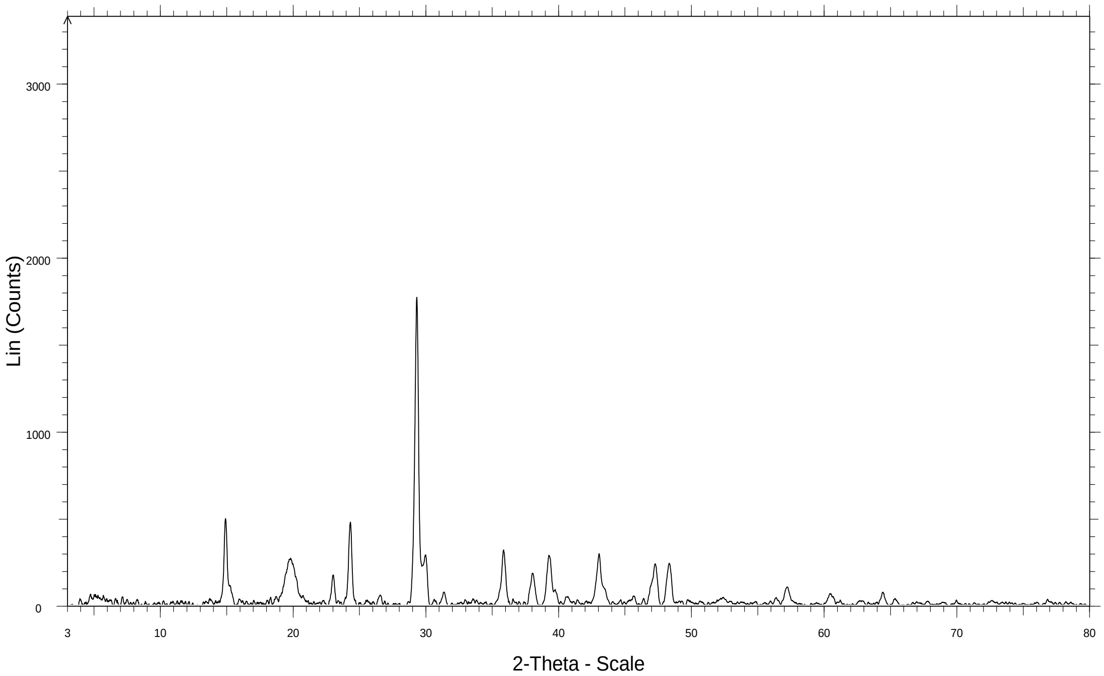


Fig.3.3 XRD View of *Pongamia pinnata* Activated Carbon (PPAC)

T.G. analysis of PPAC: Fig.3.4 shows TG curves of PPAC. There are three stages of thermal decomposition behaviour in TG curve of PPAC. The first derivative peak at 65.41^0C corresponds to 4.5% weight loss of the adsorbent, might be due to loss of some easily decomposable unknown matter entrapped in the cavities/pores of PPAC adsorbent. The material is thermally stable upto 200^0C and decomposition not seen stable and shows no decomposition. The second derivative peak at 301.26^0C indicates 10%weight loss. This loss of weight due to removal of volatile matter and other gases in the adsorbent. Huge weight loss i.e. 35% occurred at 600^0C, which is due to the breaking of polymeric bonds of cellulose, lignin, and organic molecules. After 600^0C, the TG curve almost flattens and suggests high thermal stability of the material.

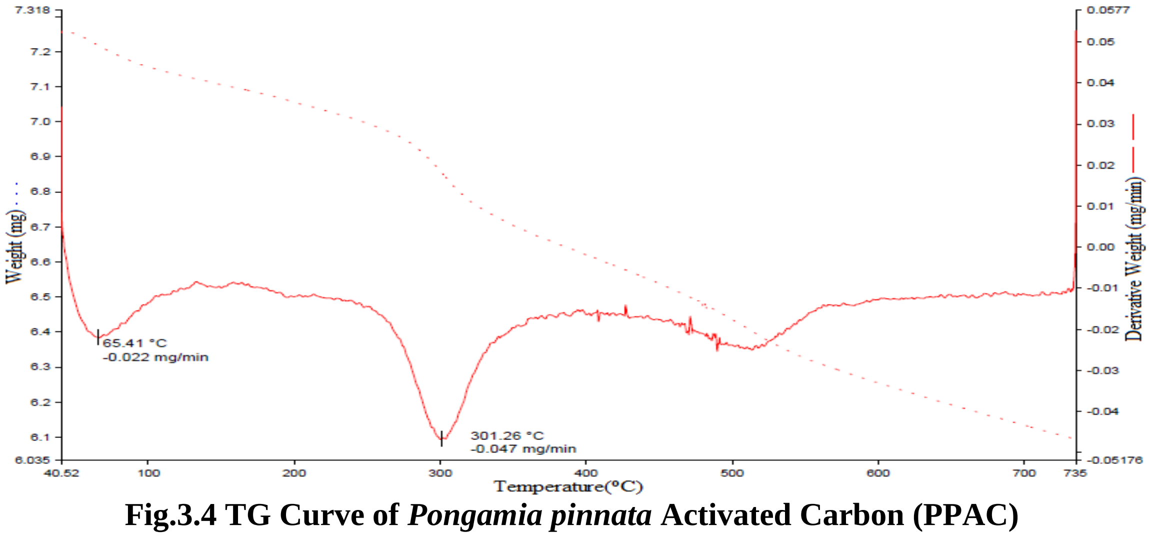


Fig.3.4 TG Curve of *Pongamia pinnata* Activated Carbon (PPAC)

Effect of pH: The pH significantly influences the removal of MB dye by PPAC. Under fixed conditions of 4.0g PPAC dose, 100mg/L MB concentration, and a 90-minute shaking period, the solution pH was varied from 2 to 10. Figure 3.5 illustrates the effect of pH on MB dye removal. The results demonstrate that the adsorption efficiency of PPAC increases from 35% to 93.5% as the pH rises from 2 to 5, beyond which it gradually declines up to pH 10. Consequently, the optimal pH of 5 was chosen for subsequent experiments. The enhanced MB dye removal at acidic pH can be attributed to pH-induced changes in the dye's surface charge and improved access to PPAC's active sites. The highest removal efficiency at lower pH levels likely results from increased protonation through neutralization at the adsorbent surface. "The preference of the dye for active pores chance to the diffusion process in the working solution" [50,51]. "While, in the alkaline conditions, protonation decreased and electrostatic repulsive force becomes active and therefore slowdown diffusion and adsorption process" [52]. A similar result pattern was noticed in the elimination of MB by using two kinds of hydro chars prepared from the orange peels and D-Glucose as feedstock [53]

Effect of Contact Time: "The most important parameter for the practical application of adsorption process is contact time. The effect of contact time for removal of MB Dye is graphically represented in Fig. 3.6 the contact time changing from 10 to 150 min with MB Dye concentration is 100 mg/L, pH of the adsorbate is set to 6.00 and adsorbent dose is 4g/L. For initially first 90 min, elimination of MB dye by PPAC was very fast i.e. 92.15% and after, the process is slower down and the equilibrium is attained after 120 min. The removal rate of MB dye molecules is higher in starting due to availability of the larger surface area of the adsorbent for the adsorption. After a certain period, only a very low increase in the dye uptake was observed because most of the pores and active sites on the adsorbent will be occupied by MB molecules, and only a few active site will be left. Once the equilibrium is achieved, this will significantly degrade the uptake of dyes. A similar observation was obtained in eliminating MB and Methyl Orange dye by oxidized chitosan" [54].

Effect of Adsorbent Dosage: The percentage of MB dye removal was studied by changing the amount of adsorbent from 1.0g to 10.00g and the concentration (100mg/L) , shaking time (100min) , temperature (room temperature) and pH (5) of the adsorbate keeping constant. From the Fig.3.7, The study found that the removal efficiency of MB dye improves with increasing adsorbent dosage until reaching maximum effectiveness. Beyond this point, further increases in the adsorbent dose do not enhance dye elimination. As shown in Fig. 3.7, the MB dye removal percentage rose from 45.5% to 93.5% as the PPAC dosage increased from 0.5g to 4.5g. The highest removal efficiency (93.5%) was achieved at a dosage of 4.5g of PPAC. MB dye removal remain constant after the 4.5g of adsorbent. Initially increases in efficiency with dose due to more availability of surfuce area and greater number of active sites. After 4.5g of dosage, adsorption will not occur because the system attained equilibrium. When extra amount of adsorbent was introduced into the dye soltion, the transfer of dye ions to the active site of adsorption will be confined as well, hence decreased in the adsorption effectiveness [55].

Effect of MB Dye Concentration: The adsorption process of the MB dye on PPAC was studied by changing the concentration of dye from 10-100 mg/L and keeping pH(6.0), contact time (100 min) and dose of adsorbent (5g/L) remain constant. The result obtained is shown in Fig.3.8 and showed that MB dye removal decrease with the raised of concentration of adsorbate. With the increasing of dye concentration from 10 to 70 mg/L, the decreased of removal of dye from 92% to 62.8%. Initially percentage removal of MG dye is very high at lower concentration due to the greater available surface area and pores on the sorbent. But after 70mg/L concentration of adsorbate, there will be less space for the other molecules of adsorbate (dye molecule) on the adsorbent (PPAC) surface.

Adsorption Isotherm: Adsorption isotherm is a significant tool for calculating removal capability of the adsorbent and giving the details regarding the surface interaction between the pollutants and the adsorbent [56,57].

Langmuir Adsorption Isotherm: This models is valid only for monolayer adsorption. The Langmuir isotherm data was obtained and is given in Table 1. The plots of C_e/Q_e Vs C_e for the PPAC suggests the significant of the Langmuir isotherms represented in Figure 3.9. The 'Q_m' and 'K_L' were calculated from slope and the intercept of the plots. The coefficient of correlation (R^2) were found to be 0.9795 which shows the best fitting of Langmuir isotherm. The adsorption efficiency'Q_m'value was found to be 21.45 mg/g for PPAC. The value of adsorption energy 'K_L ' PPAC was found to be 0.617 . Hence, it is conclude that the adsorption process represented a saturated monolayer of adsorbate on the surface of adsorbent. The adsorption energy values remains constant indicate that there is no transfer of the adsorbate molecules in the absorbent surface. The favourability of the adsorption process, the separation factor 'R_L' was found in between 0 to 1 which suggest confirmation regarding approving adsorption process .

Freundlich Adsorption Isotherm: The Freundlich adsorption isotherm is an empirical equation which elucidate physicochemical multilayer adsorption process with interaction between molecules adsorbed on heterogeneous surfaces featuring non-identical sites and varied adsorption energies. In the present study, the Freundlich equation is utilized for the MB dye adsorption on the surface of adsorbents i.e. PPAC and equilibrium data were well fitted in the linear plots of log Q_e versus log C_e as shown in Figure 3.10 The values of adsorption capacity 'k_F' and intensity of adsorption 'n' were calculated at room temperature for PPAC . The value of 'k_F'for PPAC was found to be 14.885 mg/g. Quite higher values 'k_F' suggestive of accumulation number of adsorbate molecules (liquid phase) found in larger surface area of adsorbent (solid phase). The values of 'n' which provides idea about intensity of adsorption were found to be 2.85 for PPAC. The values of 'n' obtained indicate a favorable adsorption process. The coefficient of correlation (R^2) values was found to be 0.9427 which indicate the fitting of Freundlich isotherm. The determined values given in the table no 1. It is suggest that Freundlich equation best applicable than Langmuir equation. The K_F value indicated moderate attraction for MB dye. Freundlich model is again characterized by the heterogeneity factor 1/n i.e. 0.1 < 1/n < 1.0 shows that adsorption process is carried out in a heterogeneous nature with a multilayer surface. The maximum multilayer adsorption capacity of PPAC is 14.885mg/g which shows PPAC effectiveness in the adsorption process.

Table 1: Isotherm parameters for MB Dye Removal by PPAC

Adsorbent	Freundlich Isotherm				Langmuir			
	n	1/n	K_F (mg/g)	R^2	Q_M	K_L	R^2	R_L
PPAC	2.85	0.350	14.885	0.9427	21.45	0.617	0.9795	0.185

Conclusion:

In this study, activated carbon was successfully prepared from the bark ***Pongamia pinnata*** and used for uptake of Methylene blue dye from water by batch adsorption experiment. The characterization of newly developed activated carbon PPAC was done using FTIR, SEM, TGA and XRD techniques. The parameter of pH of MB dye, contact time, sorbent dose of sorbent and initial MB dye concentration as adsorption of MB dyes studied. The experimental result show that the evacuation of MB dye by PPAC is dependent on pH and adsorbent dosage and maximum removal i.e. 93.5% of MB dye can be achieved at pH 5 and 4g PPAC. The contact time for maximum removal of MB dye were found at 90 min. The PPAC adsorbent maximum adsorption ability was determined as 21.45 mg/g.

Fig. 3.5 Effect of pH

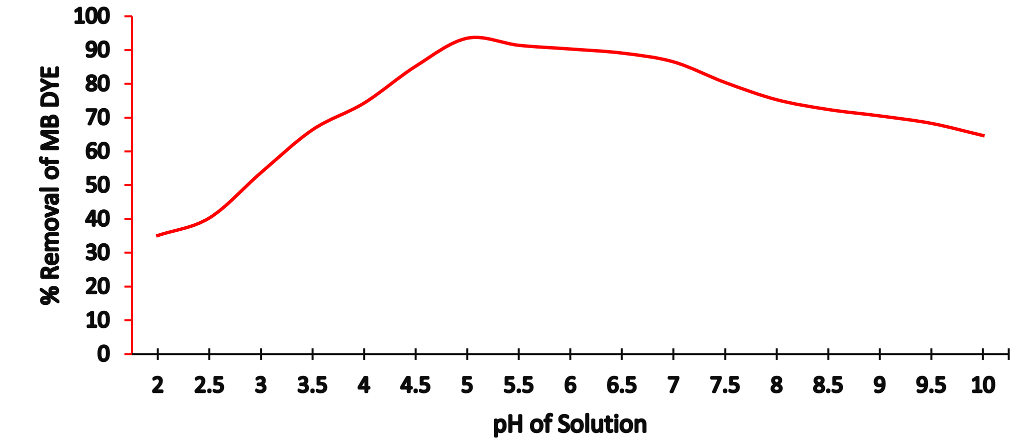


Fig. 3.6 Effect of Contact Time

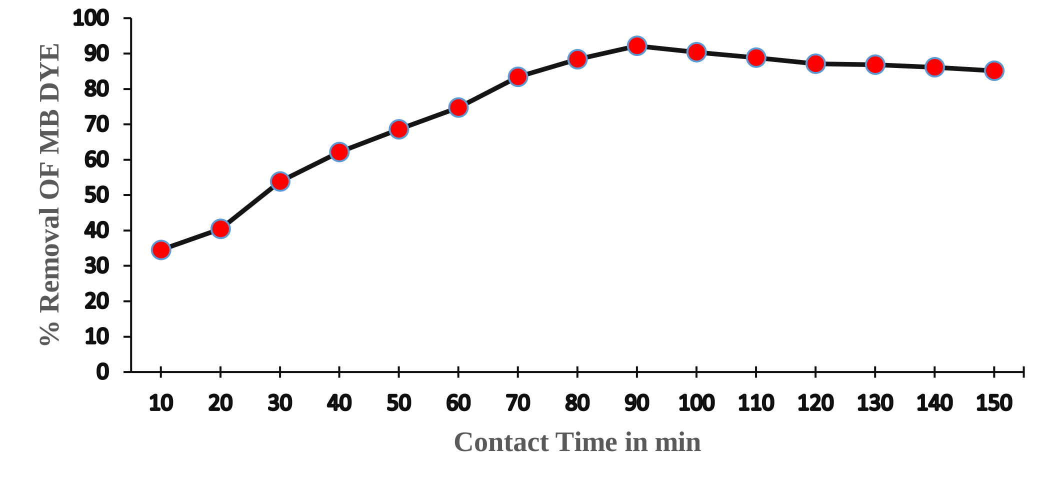
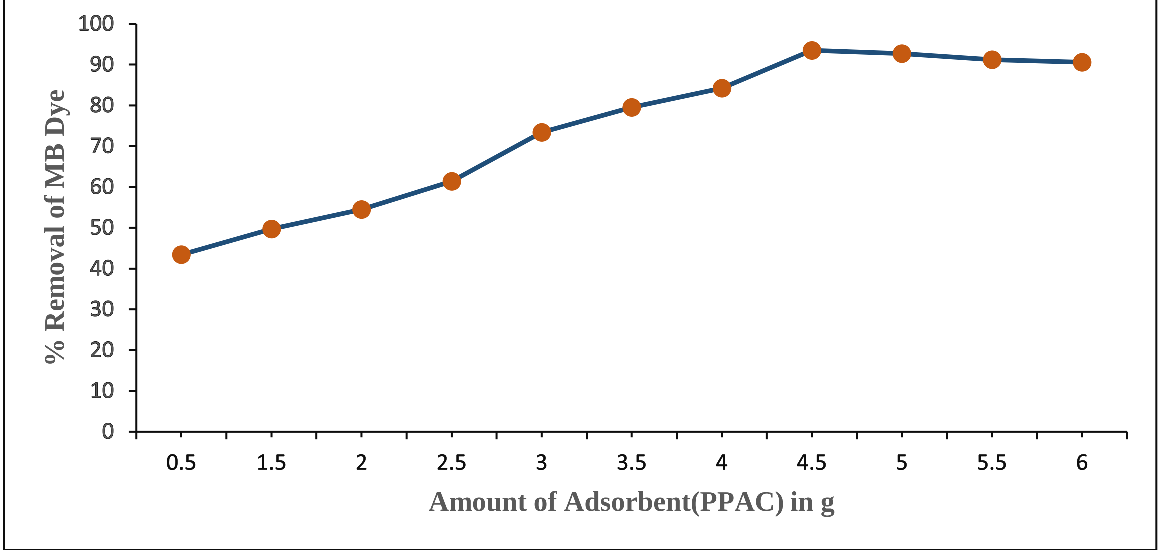


Fig.3.7 Effect of Adsorbent Dosage



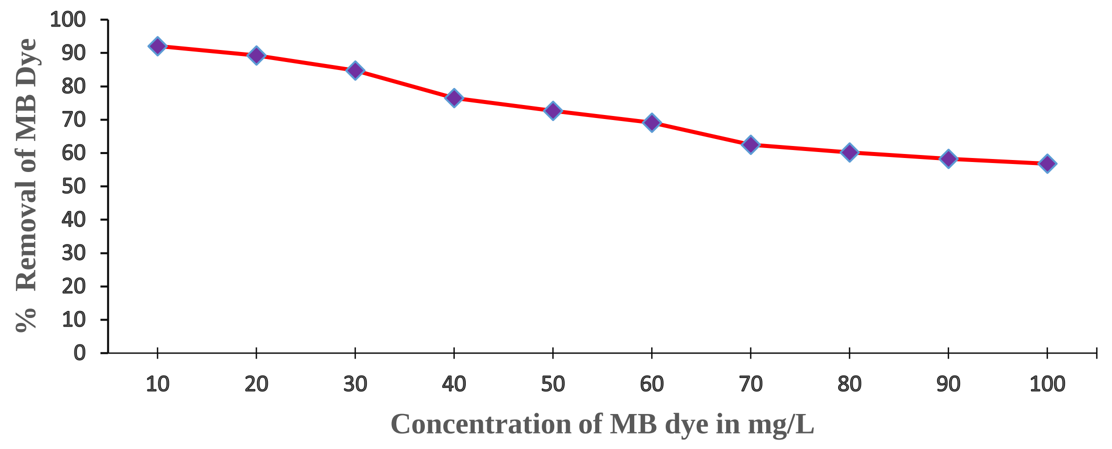


Fig. 3.8 Effect of Concentration of MB Dye

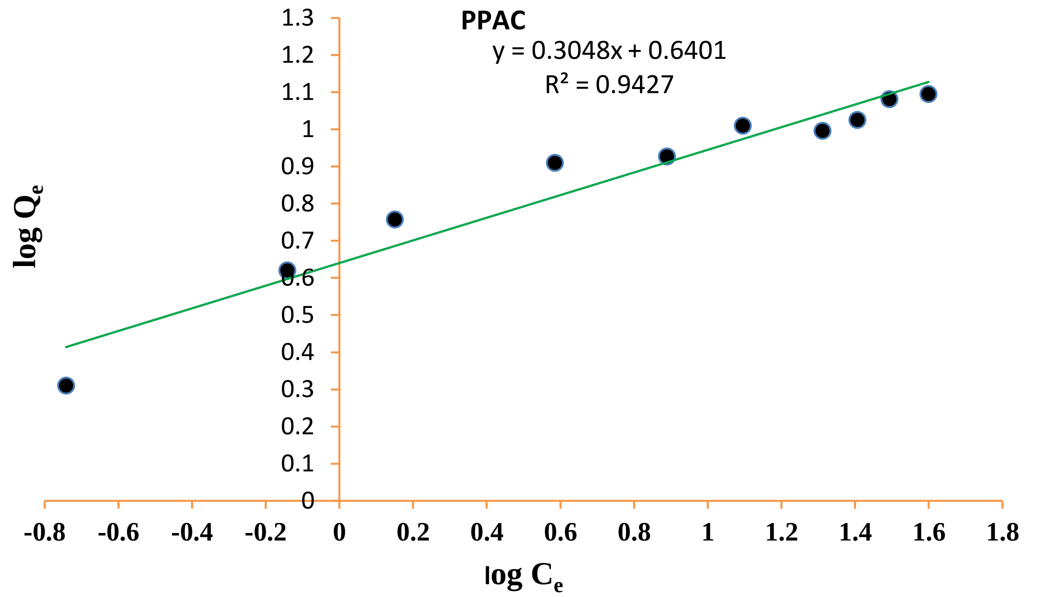
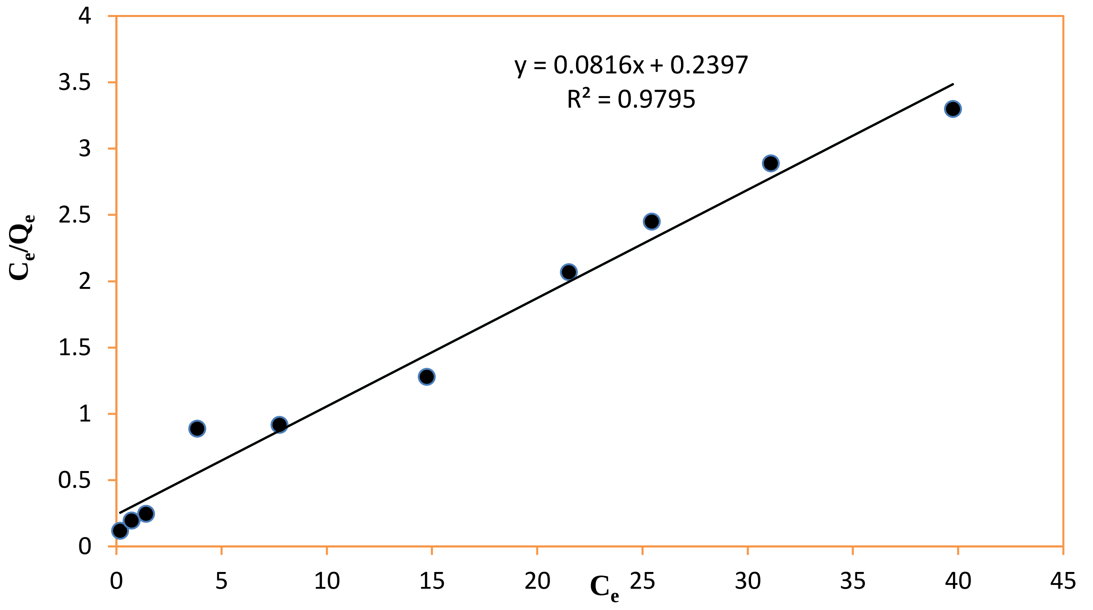


Fig. 3.10 Freundlich Isotherm for the adsorption Of MB dye on PPAC

Fig. 3.9 Langmuir Isotherm for the adsorption of MB Dye on PPAC



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