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Studies on removal of methylene blue from aqueous solution using activated carbon derived from wood apple waste biomass by microwave irradiation

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Abstract: *The purpose of this study is preparation and characterization of activated carbon using wood apple rind waste biomass by microwave irradiation using two different acids like sulphuric acid and phosphoric acid. A systematic investigation of the effect of dose, pH and initial concentration was done. The FT-IR analysis of the activated carbon depicts the presence of a variety of functional groups on it. The pH of the dye solution plays a major role in the adsorption process and it is the most important factor compared to all other factors that affect the adsorption process.*

Keywords: *Methylene blue, Microwave oven, wood apple rind, Langmuir isotherm*

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I. Introduction

Water pollution due to toxic metals and organic compounds remains a serious environmental public problem for past few decades. Heavy metal ions, aromatic compounds including phenolic derivatives, polycyclic aromatic compounds and dyes are often found in the environment as a result of their wide industrial uses. They are common contaminants in waste water and many of them are known to be toxic. Today more than 3000 dyes are in use worldwide[1]. Many methods are available for the removal of dyes from wastewater such as coagulation, solvent extraction, catalytic degradation and chemical oxidation[2,3]. But these methods have some drawbacks. Among these methods, adsorption technology is one of the attractive methods due to its salient features such as cost effectiveness, efficiency, eco-friendliness and ease of recovery[4]. Many industries are employing adsorption technique and prefer to use activated carbon for the removal of various pollutants from effluents. This is because of the high adsorption control, highly porous nature of the material and high surface area. The activated carbon can be prepared using chemical activating agents such as H_3PO_4, NaOH, KOH, H_2SO_4 or $ZnCl_2$. Activated carbon has been used for various industrial applications like adsorption of H_2S[5], dyes[6,7].Activated carbon is an excellent adsorbent that is used to remove different organic and inorganic pollutant from many aqueous solutions. Various agriculture waste or cellulose materials such as cotton stalk, date stones, rice husks, apricot stones, olive stones, and almond shells are used to prepare activated carbon[8,9]. The selection of activation method and raw materials are important in controlling the physical and chemical characterization of the prepared activated carbon[10,11]. The use of the thermal heating is to enhance the surface chemistry and structure of pores[12,13]. Microwave processing of material is a technology that can provide a new, powerful, and significantly different tool to process materials or to improve the performance characteristics of existing materials.[14,15] Compared with conventional methods, this heating offer many advantages including shorter synthesis time, rapid heating, fast reaction, easy reproducibility, narrow particle distribution, high yield, high purity, efficient energy transformation. Electricity consumption can be reduced by more than half by using microwave assisted sintering[16]. In recent years, microwave has emerged as a promising alternative energy source for the heating materials.

II. Materials And Methods

The waste biomasses obtained from wood apple rind was crushed into small pieces and soaked with phosphoric acid (H_3PO_4) in 1: 1 ratio (w/v) for 48 hours to yield a black carbon product. Then it was kept in a microwave oven maintained at 850 watts for 4 minutes. It was then washed thoroughly with distilled water until the pH of the wash water becomes the pH of the distilled water. The wet product obtained was dried at 105 ± 5°C. The carbonaceous adsorbent is ground and sieved. The adsorbent obtained after phosphoric acid treatment was designated as MPWAC. The above process is repeated with same quantity of sulphuric acid and the carbon obtained was designated as MSWAC. A stock solution of 1000mg/L of Methylene blue dye was prepared using

distilled water. Batch mode experiments were conducted in screw cap bottles of 200 mL capacity. Hundred milliliter of the solution containing predetermined concentration of the MB dye under investigation was taken in the bottles. The pH of the dye solution was adjusted by using digital pH meter ELIGO digital model. After the addition of known amount of adsorbent, the bottles were equilibrated for a predetermined period of time in a mechanical shaker at 120 rpm. At the end of the equilibration period, using micro filters, the dye solution is taken and the concentration of residual MB in solution was determined. The functional groups available on the surface of MPWAC and MSWAC were detected using FT-IR analysis. The surface morphology of MPWAC and MSWAC was characterized using Scanning Electron Microscope. The effect of initial dye concentration was studied by varying the initial MB dye concentrations from 10 ppm to 80 ppm for the optimized adsorbent dosage (0.1g).

III. Results And Discussion

Characterization of adsorbents

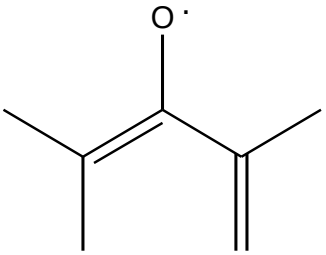
Table 1:

Parameter	MPWAC	MSWAC
pH	2.5	3.2
Moisture content(%)	3.6	6.6
Ash content(%)	2.8	1.8
Water soluble matter(%)	7.6	3.6
Acid soluble matter(%)	7.8	3.9
Iodine number(mg/g)	1208	2230
Decolourising power(mg/g)	30	50
Bulk Density(g/cc)	0.6006	0.6806
Zero Point Charge	11.9	10.1

FT-IR analysis:

The FT-IR spectroscopic study of MPWAC and MSWAC activated carbons are shown in Figure 1, 2. The spectrum showed four major absorption bands at 3400 cm^{-1}, 1600 cm^{-1}, 1400 cm^{-1} and 600cm^{-1} range. The band at 3400 cm^{-1} is due to the absorption of water molecules as result of an O-H stretching mode of hydroxyl groups and adsorbed water, while the band at 2800 is attributed to C-H interaction with the surface of the carbon. However, it must be indicated that the bands in the range of 3200-3650cm^{-1} have also been attributed to the hydrogen-bonded OH group of alcohols and phenols. The band at 600-800 cm^{-1} is attributed to C-Cl group.

The band at 1400-1600 cm^{-1} may also be attributed to the aromatic carbon-carbon stretching vibration. The intense band around 1600 cm^{-1}, may be due to asymmetric and symmetric stretching COO^{-} vibrations or to skeletal C=C aromatic vibrations. Bands appearing in the range of 1600 cm^{-1} were also attributed to vibrations in iono-radical structures.



The iono-radical structure

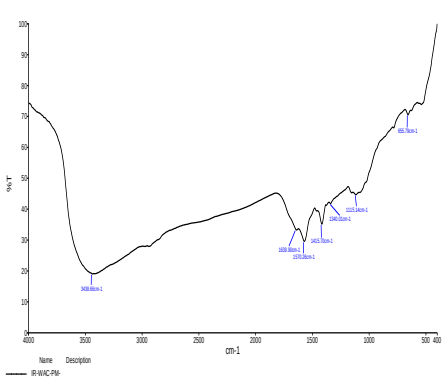


Fig 1: FT-IR spectrum of MPWAC

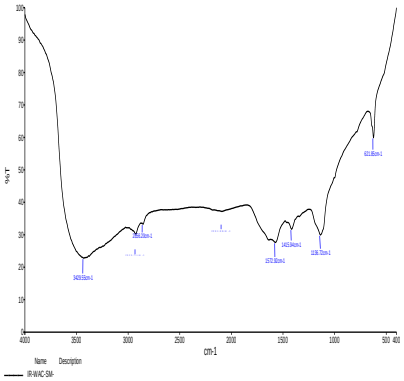


Fig 2: FTIR spectrum of MSWAC

SEM Analysis:

The scanning electron micrographs of plain MPWAC and MSWAC obtained are shown in Fig 3&4. The figures clearly depict the size, surface texture and porosity of carbons. A number of holes and small openings, called micro pores and widening of pore size at 100 microns are found on the surface of the carbons.

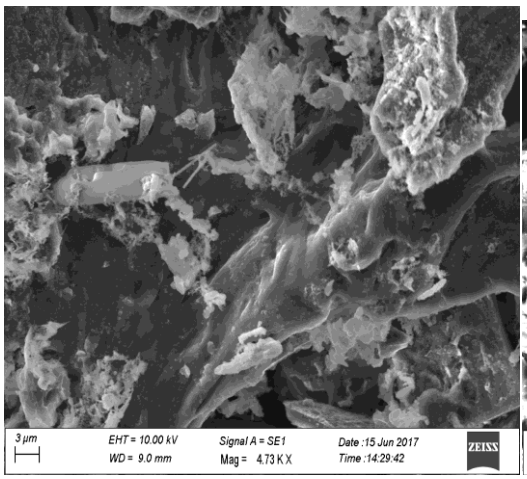


Fig 3: SEM images of MPWAC

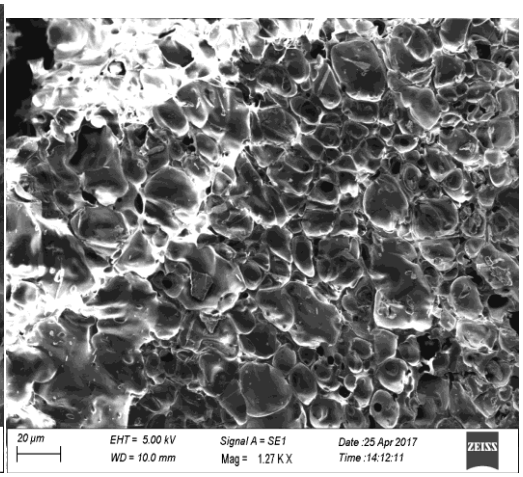


Fig 4: SEM images of MSWAC

Effect of carbon dosage:

The effect of adsorbent dosage was studied by varying the adsorbent dosage from 0.2g/L to 2.5g/L. From the fig 5&6 it was observed that the percentage adsorption of MB dye increased with an increase of the carbon dosage. The maximum % of adsorption of MB was found to be MPWAC is 93.6 % and MSWAC is 96.4% at a dosage of 2.5 g/L.

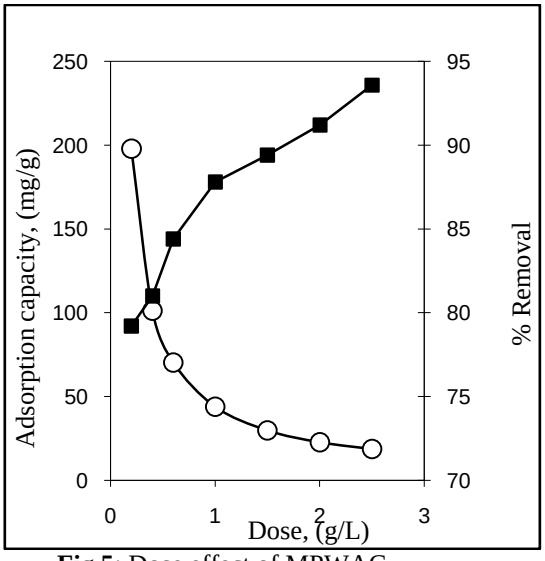


Fig 5: Dose effect of MPWAC

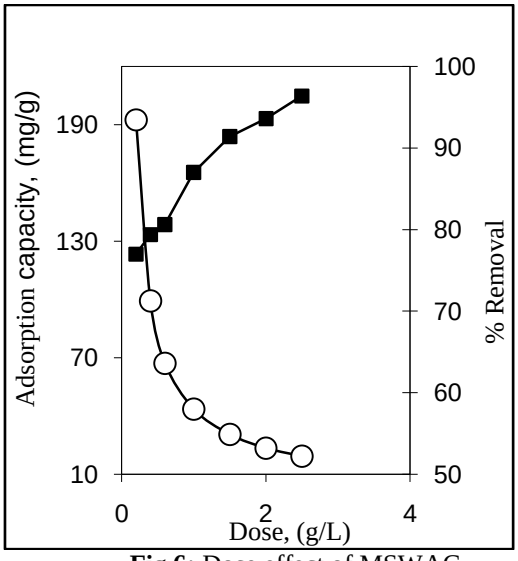


Fig 6: Dose effect of MSWAC

Effect of initial aqueous pH on adsorption

Initial aqueous phase pH plays an important role in the liquid-phase adsorption capacity. The pH of the solution is the most important factor compared to the all other factors that affect the adsorption process. In MSWAC-MB system, the increase of pH of the solution increases the adsorption of MB dye from solution. Initial rate of adsorption is mainly decided by the protons release from carbon surface due to ion exchange process which is confirmed by the measurement of pH of the solution after the adsorption process. (The pH of the solution decreased to the pH in the range of 2.5 -3.0). At acidic pH, the adsorption of the dye is low and upto pH 7 and then the adsorption capacity of MSWAC is found to be higher at pH 9 at higher pH the protons are neutralized with OH ions so the adsorption process increases.

But in MPWAC-MB system, the % removal of the dye increased from 77% to 86% (upto 7.5) then after pH 7.5 adsorption of the dye is decreased. Lower amount of MB dye was adsorbed onto the prepared

adsorbents at acidic pH and then increased sharply upto pH 7 then it decreases. The amount of MB adsorbed is decreased after pH 7, the reduction in adsorbed amount of MB after pH 7 may be ascribed to the increasing repulsive forces between surface functional groups of adsorbent and cationic MB dye that mainly exists as anion form [17]. Basically, methylene blue and other cationic dyes produce an intense molecular cation (C^+)

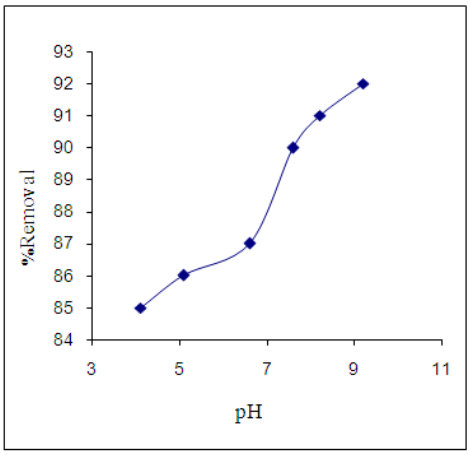


Fig 7: Effect of MSWAC

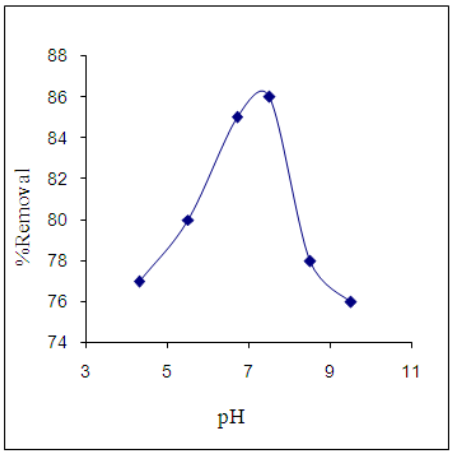


Fig 8: Effect of MPWAC

Adsorption Isotherm

The effect of initial dye concentration was studied by varying the initial MB dye concentrations from 10 ppm to 80 ppm for the optimized adsorbent dosage (0.1g). This study was further extended to calculate equilibrium parameters using linear form of isotherm models such as Langmuir and Freundlich isotherms

Langmuir isotherm

The Langmuir equation is applicable to homogeneous adsorption and it is based upon the assumption of monolayer adsorption, where the adsorption of each adsorbate molecule onto the surface has equal sorption activation energy. The linear form of this isotherm is represented by the expression[18]

$$C_e / q_e = 1/ q_0 b + C_e / q_e$$

Where, qe (mg/g) and Ce (mg/L) are the amount of adsorbed adsorbate per unit weight of adsorbent and unadsorbed adsorbate concentration in solution at equilibrium respectively. K_a (L/mg) is the Langmuir equilibrium constant and the maximum monolayer adsorption capacity (q_m) was found to be MPWAC is 63 mg/g (equilibrium time=3 hrs).and MSWAC is 66 mg/g(equilibrium time=2 hrs).

Freundlich isotherm

The most important multisite adsorption isotherm for the non-ideal adsorption on heterogeneous surfaces is the Freundlich adsorption isotherm and the linear form of this isotherm is expressed as[19]

$$\log q_e = \log K_F + \log C_e^{1/n}$$

Freundlich constant K_f indicates the adsorption capacity also represents the strength of the adsorptive bond and n is the heterogeneity factor which also represents the bond distribution.

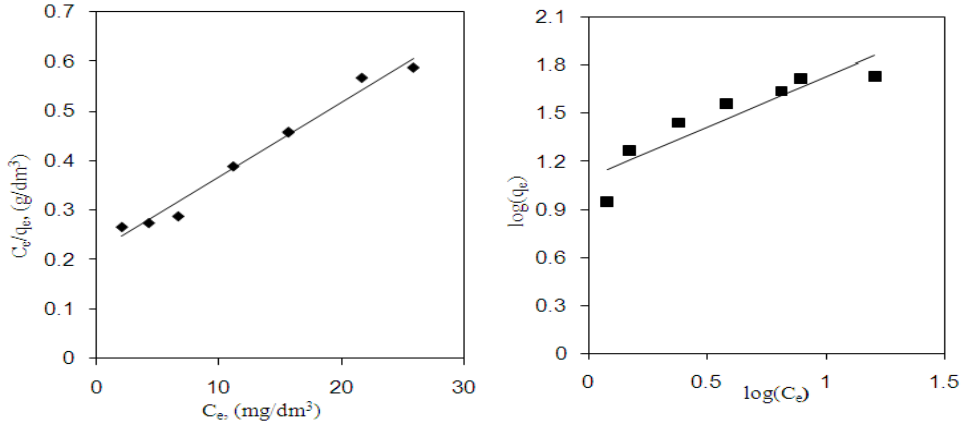


Fig 9&10: Langmuir & Freundlich isotherm model MSWAC

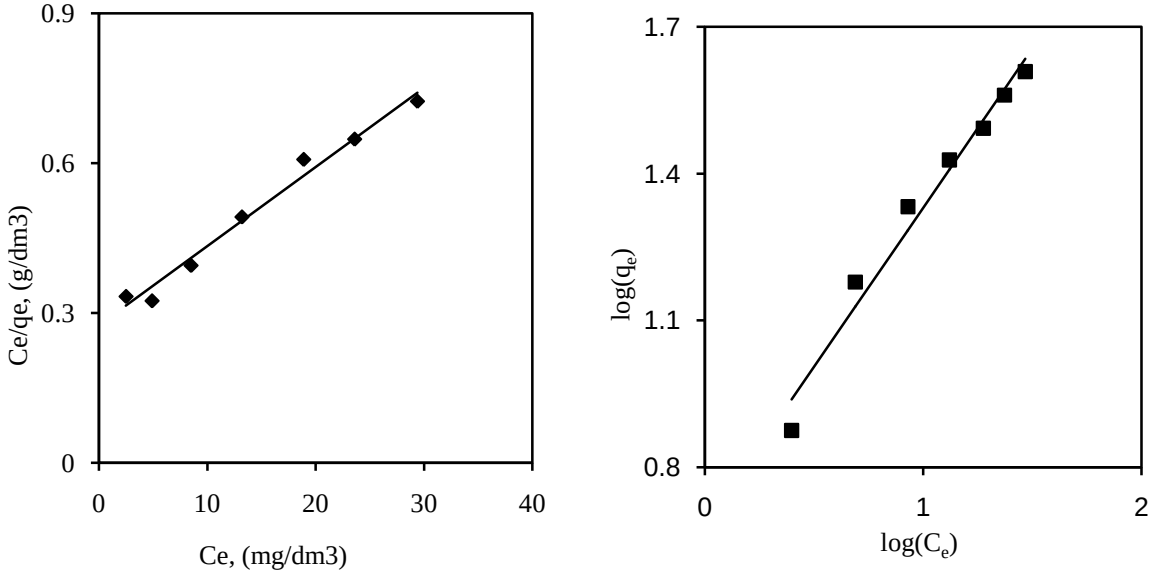


Fig 11&12: Langmuir & Freundlich isotherm model MPWAC

For both carbons the increase in initial concentration of dye solution increased the adsorption capacity. The experimental results are given in Table 2&3. From the Figs 9, 10, 11 & 12, it is clear that the adsorption of basic dye (MB) on MPWAC & MSWAC follows Langmuir model. So the removal of basic dyes from aqueous solution by activated carbon obtained from wood apple rind waste biomass gives monolayer coverage and the adsorption is mainly due to Chemisorptions. This is confirmed by the desorption studies also. There is no desorption of MB dye from loaded carbon from the solution. It is carried out by MB loaded carbon at various pH from 3 to 10. The Langmuir constant, K_a, reveals that MB is bound strongly on both MPWAC & MSWAC. Higher the K_a value, stronger is the affinity between the carbon surface and the dye molecules. The fitted isotherm parameters are given in Table 2.

Table 2: Fitted isotherm parameters for MPWAC system at pH 7

Isotherm model	Parameters	Units	MPWAC	MSWAC
Langmuir	q_m	mg/g	63	66
	K_a	L/mg	0.057	0.070
	r^2		0.981	0.980
Freundlich	K_F	g/L	0.650	0.647
	1/n		4.779	5.713
	r^2		0.973	0.965

IV. Conclusion

Systematic batch mode studies were carried out to find out the effect pH on removal of MB using MPWAC and MSWAC. The activated carbon prepared from wood apple rind waste by using sulphuric acid and phosphoric acid by microwave irradiation activation method can be used for wastewater treatment. The adsorption capacity is reasonably high for both the carbons. Through the IR spectrum of both the carbons, the functional groups presents on the surface of the carbon is identified. From the SEM images at various magnifications of MPWAC & MSWAC shows the size of the pores present on the surface. The adsorption equilibrium data fits into the Langmuir isotherm model which indicates monolayer adsorption. Experimental results of the present investigation showed that MPWAC and MSWAC is a potential adsorbent for the removal MB from aqueous solution over a wide range of concentrations.

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