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RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM WASTE TEA AND APPLICATION FOR DEGRADATION OF DYES

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ABSTRACT

The studies on characterization of different type of adsorbent such as activated carbon from waste tea has been performed. The characterization of the adsorbents includes estimation of various parameters such as proximate analysis moisture content, BET surface area, SEM, and FTIR. Surface area of adsorbents was found by BET surface area analyzer. The pore structure of activated carbon was observed through SEM analysis. The adsorbent that showed best surface properties was used for adsorption of methylene blue dye. Adsorption capacity of methylene blue from aqueous solution onto the activated carbon prepared from waste tea was investigated under various experimental conditions. Batch mode experiments were conducted to assess the potential of the above activated carbon for the removal of Methylene blue from aqueous solution. Kinetics studies have been done by varying the following three parameters: initial concentration of Methylene blue dye solution, adsorbent dose on the uptake of dye from the solution and effect of temperature .Adsorption kinetics studies indicated that the pseudo first - order model yielded the best fit for the kinetic data. These results indicate that waste tea could be utilized as a renewable resource to develop activated carbon which is a potential adsorbent for methylene blue.

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INTRODUCTION

Agricultural and food industries create various waste matters that need to be utilized and convert in value added product. In the present study the activated carbon was prepared from the carbonaceous kitchen waste, waste tea by using chemical activation agent potassium hydroxide and sodium carbonate at different temperature. These waste materials generated in oil mill industries or in various villages and can be collected on community basis for reuse. Activated carbon is a well known as porous material, with large specific surface area, which is useful in various application of both gases and solutes from aqueous solution (Hariprasad, 2016). Various raw materials, such as wood, peat, agricultural waste including plant materials like groundnut, cotton, wheat, sorghum, millet, maize, sesame, etc. There are two method for the preparation of activated carbon one is physical activation and second is chemical activation. Physical activation is a two stage process, it involves carbonization of raw material followed by activation at elevated temperatures in the presence of suitable oxidizing gases such as carbon dioxide, steam, air or their mixtures. Carbonization temperature ranges between 400^0C to 800^0C, and activation temperature ranges between 800^0C to 1100^0C.

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Preparation of activated carbon by chemical activation is a process in which carbonization and activation is carried out simultaneously. Initially the precursor is mixed with chemical activating agent, which acts as dehydrating agent and oxidant. Carbonization and activation temperature ranges between 300^0C to 600^0C. Activated Carbons as microporous materials are of the most important adsorbents, which have been extensively used as adsorbents, catalyst and catalyst supports in a variety of industrial and environmental applications. Activated carbon is an extremely versatile material as an effective adsorbent with its high adsorptive capacity and high surface area the activated carbon application attractive are availability, environmentally friendly material, safe and very low cost of the staring materials coupled with its high surface area (Hariprasad, 2016). Various materials are used to produce activated carbon and some of the most commonly used are agriculture wastes such as almond shell and walnut shell , palm shell , lapsi stone, palm oil shell, snail shell, cashew nut shell, sugarcane bagasse, wood, rice husk, mustard stalk, jute stalk, sesame stalk, wheat straw , hazelnut shell, bamboo, Sewage sludge, etc. Chemical activation has been given very effective carbon with high specific surface area. The most commonly used chemical activating agents are KOH, H_2SO_4, $ZnCl_2$, Na_2Co_3, K_2CO_3, NaOH, H_3PO_4 etc. $ZnCl_2$ create waste disposal problem and that is environmental unfriendly. This work had as a main objective to obtain activated carbon from

waste tea and to analyze their internal structure through Scanning Electronic Microscopy (SEM) and Fourier-Transform Infrared Spectroscopy (FTIR) (Sushmita Banerjee, 2014).

MATERIALS AND METHODS

All chemicals and reagents used were of analytical reagent grade. Potassium hydroxide (KOH), Methylene Blue dye were obtained from laboratory. The natural low cost material waste tea obtained from own house. The solutions of required concentrations were prepared by suitable dilution of the stock solutions. After desired adsorption, the supernatant solutions were taken as sample in sample bottle. Adsorption measurements were carried out using UV-vis spectrometer (UV-1800 UV-VIS specrophotometer).

Preparation of Activated carbon: 3 waste tea samples were weighed and then subjected to physical activation in a muffle furnace, temperature ranging from 350ºC,450ºCand ,600ºC, for 1 hr ,and the samples were weighed after physical activation .Samples thus obtained were soaked in 1M KOH in 1:1 ratio for 24hrs followed by weighing the sample in order to know the impregnation of 1M KOH to the samples and is followed by activation in muffle furnace at temperature 300^0C for 2hr. The carbonized material was washed with distilled water to remove the free alkalis and dried at 100^0 $\pm5^0$ C 2hrs and weighed to calculate the yield (Demirbas, 2009).

Characterization Of Activated Carbon: The physico-chemical characterization of carbon samples prepared has been done; the yield of activated carbon is the % amount of activated carbon produced at the end of the activation process. This value indicates the activation process efficiency. The surface of activated carbon prepared has been analyzed using SEM (Scanning Electron Microscope), in order to identify the functional group responsible for adsorption Fourier transform infrared spectroscopy (FTIR) analysis was carried out.BET surface of both samples were identified using nitrogen adsorption, along with this pore size, created in samples were also identified (Yak out, 2011).

Batch adsorption studies: The adsorption of methylene blue dye on adsorbent of waste tea biosorbent was studied by batch adsorption technique. A known quantity of prepare adsorbent (e.g 0.1 g adsorbent)was added on 100 ml of dye namely (Methylene blue solution).the effect of initial concentration (20,40,60,80 ppm) and adsorbent dose (0.1 ,0.2 and 0.3 g) on percentage removal dye was investigated at fixed temperature (30°c,40ºc,50ºc) (Yak out, 2011 and Walker, 1997). The time of contact was kept constant 120 min in each batch experiment. The sample was collected at the time intervals (5, 10, 15, 30, 60, 90, 120) min contact and samples are taken at this time intervals. The concentration of dye remaining in solution was measured by uv-vis spectrometer. the percentage of dye removal and equilibrium adsorption capacity were calculated.

UV Analysis: Initially various concentrations of Dichlorvos solution were prepared. For instance 25 ppm, 20 ppm, 15ppm , 10 ppm and so on. These samples were checked for the Absorbance spectra on the UV/Vis Spectrophotometer (Lab India UV 3200). The spectra for Concentrations of various samples were recorded and a particular wavelength was selected (690 nm) from the literature survey. For this particular wavelength all the concentration samples were checked for absorbance. And a graph was plotted for selected samples. This graph should have a fitting of more than 0.98 to truly fit the data. An equation was obtained from the curve fitting and this equation is known as the calibration equation (Malik, 2004). Now once the calibrated curve was standardized any unknown sample could be checked for absorbance setting the wavelength at 690 nm. Using the obtained absorbance we can calculate the concentration of the unknown sample using the calibration equation.

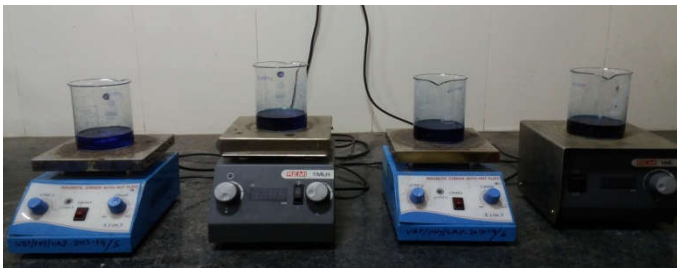

Fig. 1. Dye degradation on conventional method

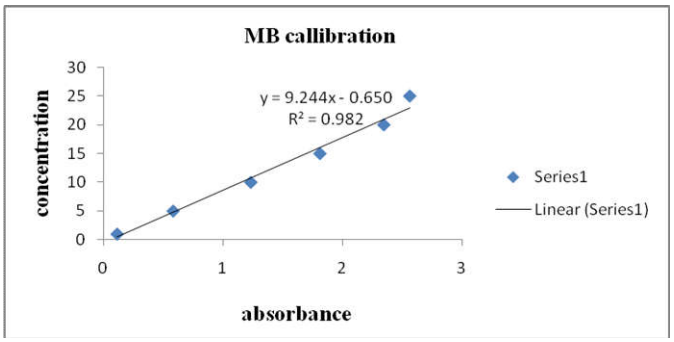

Fig. 2. MB calibration

Adsorption kinetics: Kinetic study is important to the adsorption process because it depicts the uptake rate of adsorbate and controls the residual time of the whole adsorption process. To explore the controlling mechanism of the adsorption process, the experimental data were fitted to the pseudo-first-order. The linear forms of the pseudo-first-order are represented by

$$\ln(C_A - C_0) = \ln C_A - K_1 t$$

where $K1$ (min−1) are the rate constants of the pseudo-first-order respectively (Adekola and Adegoke, ? and Pragya, 2013). The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations. As canbe seen, the R^2 values obtained from the pseudo first-order were consistently higher. In addition, the C_A values coincided with the expected $C_A$values (C_A,exp). That indicates that the adsorption perfectly obeys the pseudo-first - order model meaning the controlling rate step is chemisorption. Meanwhile, the result showed that the rate of adsorption depended on the availability of adsorption sites on the surface of adsorbent rather than MB concentration in bulk solution.

RESULT AND DISCUSSION

The preparation of activated carbon has been carried out from waste tea at 350ºC, 450ºC, 600ºC and chemical. Impregnation has been done with 1 M KOH in 1:1 ratio, the yield of KOH

activated carbon has been decreased gradually when activation temperature has increased from 350ºC to 450^{0}C after physical activation, but after chemical activation the yield has been increased up to 600ºC temperature range due to KOH impregnation (Pragya, 2013). The porosity has been not much changed when temperature increased, but pore size has been found increased when temperature increased, moisture content, decolourising power, was also increased slightly when activation temperature increased. The surface area of waste tea KOH activated carbon has found to be increased up to the temperature 600^{0}C (230 M^2/g Surface area). This is due to increase in pore diameter and microspore volume.

Table 1. Yield of carbon under different activation methods

Sample name	Waste tea	Waste tea	Waste tea
Activation temperature	350^0 C	450^0 C	600^0 C
Initial weight in gm	20gm	20gm	20gm
Yield in gm	6.4gm	6.5gm	5.8gm
Yield after chemical activation in g	6.8gm	7.4gm	6.4gm
Moisture content %	6.25 %	13.84 %	10.34 %
% yield	94.11%	87.83%	90.62%

SEM analysis: Scanning electron microscope images were taken to observe the surface topography of the sample. Basically, the pore structures of the activated carbons were observed. Themicrographs of the activated carbon are shown in below (Mui, 2004).

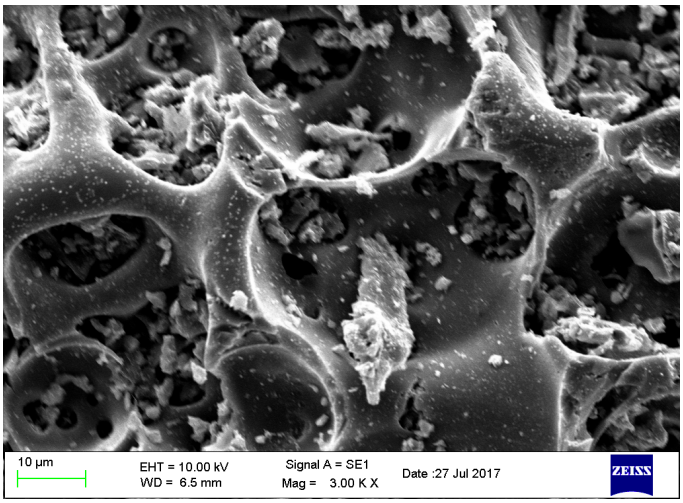

Fig. 3. SEM analysis of KOH waste tea Activated carbon sample

FTIR analysis of sample waste tea

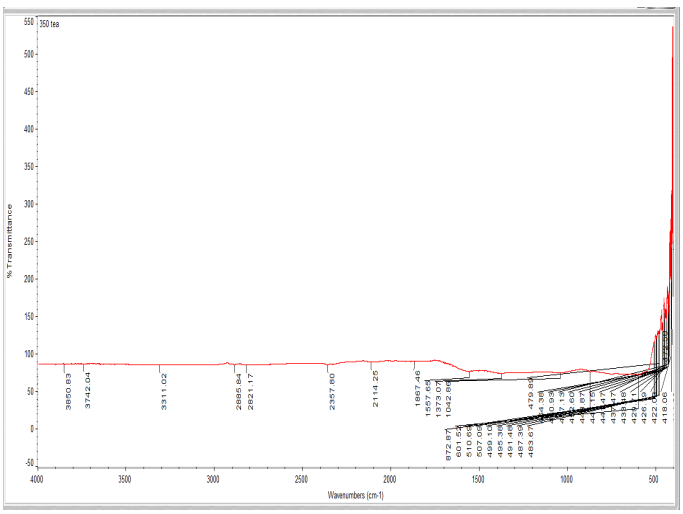

Fig. 4. FTIR analysis – sample waste tea

Table 2. FTIR analysis

Sample name and temp.	Waste tea 350 ºC	Waste tea 450 ºC	Waste tea 600 ºC
Wave no (cm^{-1})	3,495 2,950 and 2,880 403-672	1,745 1,640–1,430 700-1,260	1,730 1,625 400–4700 666 and 990
Functional Groups Identified In Ftir Analysis	O–H stretching in hydroxyl groups in phenolic and aliphatic structures C–H and symmetric C–H bands	C=O stretching from ketones, aldehydes or carboxylic groups , C=C stretching vibrations in the aromatic. C–O–C stretching in ethers	C=O, aromatic rings or C=C bond, C–O single bond in carboxylic acids, alcohols phenols and esters. P=O groups and to P=O stretching in linear and cyclic polyphosphate and inorganic species

The FTIR analysis shows the presence of functional groups present in the surface of activated carbon. It shows that P=O, functional group has been removed when temperature has raced in to 600^{0}C and conj=O bond has been appeared in the surface of activated carbon.

Graphical Representation of Removal of MB dye by Mechanical Stirring

Effect of initial dye concentration

Initial of solution concentration of solution plays an important role in adsorption of MB dye. The effect of initial concentration of MB on its percentage removal of dye. In this study, the initial concentration of solution was varied between 20 ppm, 40ppm, 60 ppm, 80 ppm. In all the cases, an increase in the percentage removal of MB was observed with an increase in the concentration. The of adsorption was found to be decreased with an increase in initial concentration (Mattson, 1971).

Sample of waste tea at 350ºC

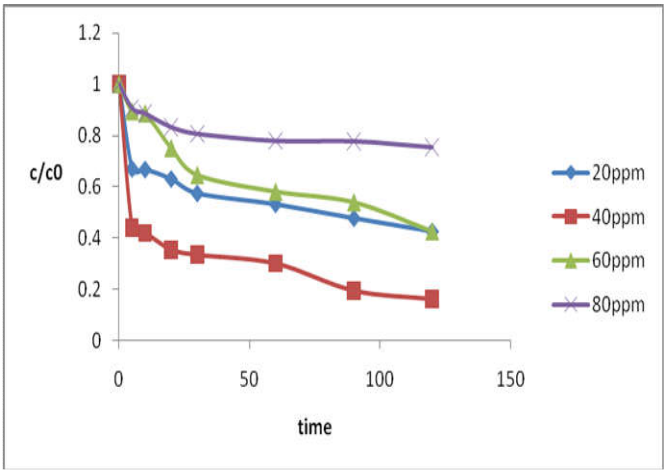

Fig. 5. Time Vs C/C0

Figure Shows Effect of Intial Concentration for the adsorption of MB dye onto conventional stirring process (The initial dye concentration 20,40,60,80 ppm, adsorbent dose 0.1 g/L and time 2hr at 30◦C). Figure show The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations.

Table 3. Percentage removal calculation

Percentage Removal, %R = (C0−Cf) × 100 / C0

Sr no		20ppm		40ppm		60ppm		80ppm	
1	350ºC–waste tea	57.45%		83.99%		57.45%		24.5%	
2	450ºC -waste tea	27.9%		45.65%		40.08%		25.33%	
3	600ºC -waste tea	45.95%		52.81%		27.71%		34.05%	
	Kinetic models parameters	K1	R^2	K2	R^2	K3	R^2	K4	R^2
1	350ºC–waste tea	0.008	0.05	0.018	0.12	0.007	0.85	0.003	0.09
2	450ºC -waste tea	0.003	0.59	0.004	0.93	0.003	0.77	0.002	0.93
3	600ºC -waste tea	0.006	0.62	0.005	0.91	0.002	0.78	0.004	0.47

Result : the result was found in the waste tea of 350ºC was 83.99 % removal of dye at 40ppm on 0.1 g/L

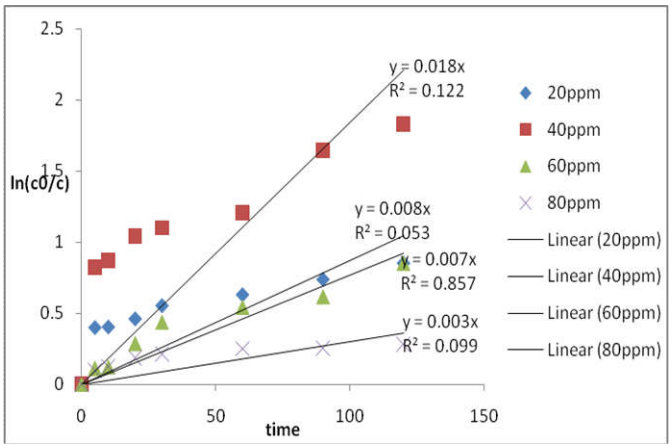

Fig. 6. Time Vs Ln(C0/C)

Sample of waste tea at 450ºC

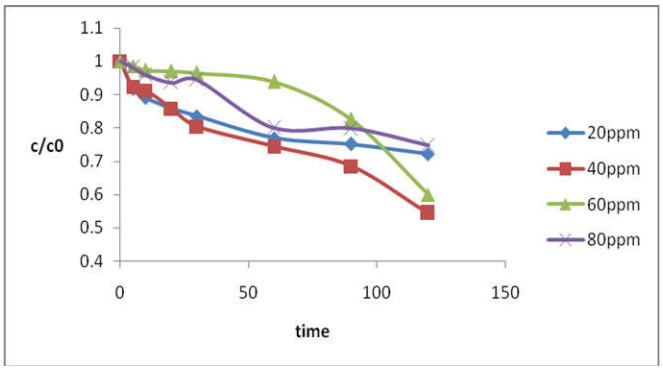

Fig. 7. Time Vs C/C0

Figure Shows Effect of Intial Concentration for the adsorption of MB dye onto conventional stirring process (The initial dye concentration 20,40,60,80 ppm, adsorbent dose 0.1g/L and time 2hr at 30◦C).

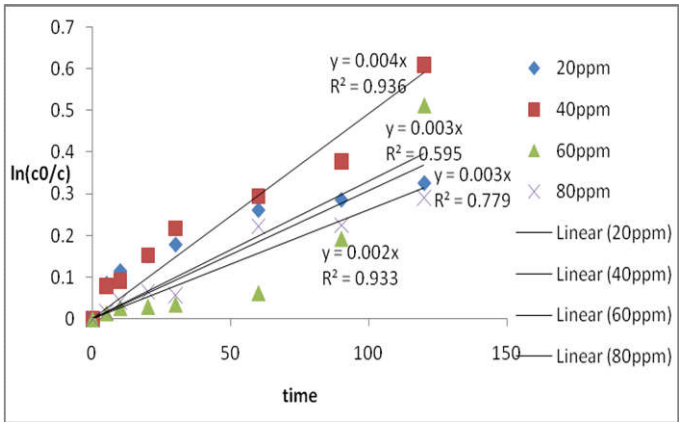

Fig. 8. Time Vs Ln(C0/C)

Figure show The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations.

Sample of waste tea at 600^{0}C

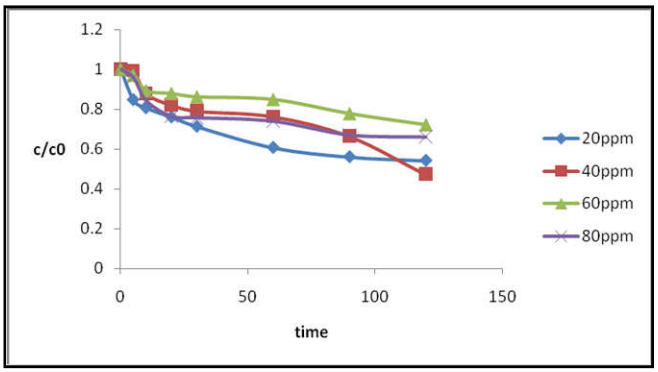

Fig. 9. Time Vs C/C0

Figure Shows Effect of Intial Concentration for the adsorption of MB dye onto conventional stirring process (The initial dye concentration 20,40,60,80 ppm, adsorbent dose 0.1g/L and time 2hr at 30◦C).

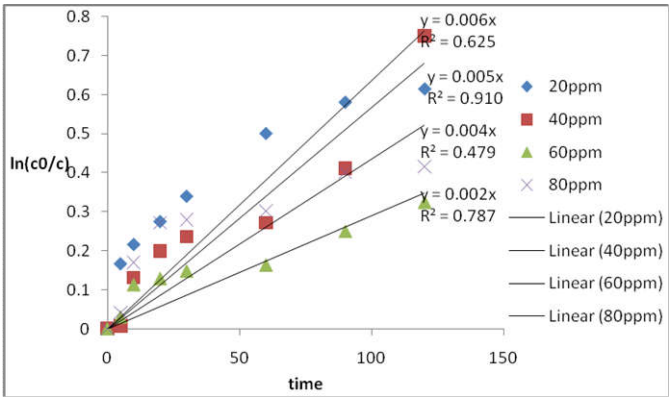

Fig. 10. Time VsLn(C0/C)

Figure show The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations. That indicates that the adsorption perfectly obeys the pseudo-first-order model .Meanwhile, the result showed that the rate of adsorption depended on the availability of adsorption sites on the surface of adsorbent rather than MB concentration in bulk solution.

Effect of adsorbent loading on Removal of MB

Adsorbent load have a great influence on methylene blue dye removal. It is shown by graphically the effect of 0.1g/lit, 0.2g/lit and 0.3g/lit. It was found that in all cases the percent

removal of dye increases with an increase in adsorbent dose. It is attributed to an increase it the removal efficiency of dye with an increase in the number of available active adsorption sites. Amount of adsorbent added to the solution gives the extra number of binding sites which are available for adsorption (Smisek, 1970).

Effect of contact time on Removal of MB: The influence of contact time for the removal of dye from the aqueous solution using biosorbent was analyzed at different initial solution concentration at different time intervals. Samples are taken at different time interval (5, 10, 15, 30, 60, 90 and 120 min). If contact time increases then rate of dye removal also increases it is observed from experimental work and it shown by graph

Sample of waste tea at 350 ºC: Sample taken of waste tea at 350ºC because we got 83.99% of removal dye at 40 ppm so we finalized this 350 ºC temperature parameter and 40 ppm for waste tea sample.

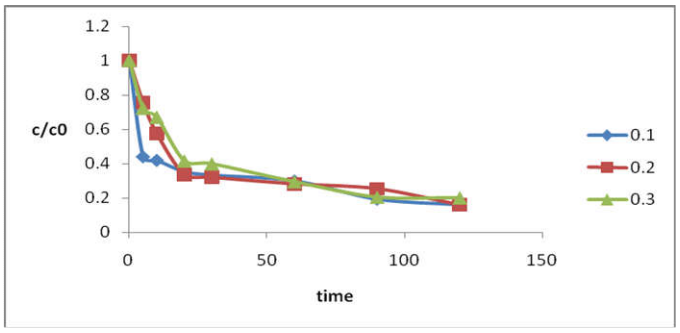

Fig.11. Time Vs C/C0

Figure Shows Effect of Intial dye Concentration for the adsorption of MB dye onto conventional stirring process (The Adsorbent doses of 0.1 ,0.2 ,0.3 g/L, Dye concentration of 40 ppm and time 2hr at 30◦C).

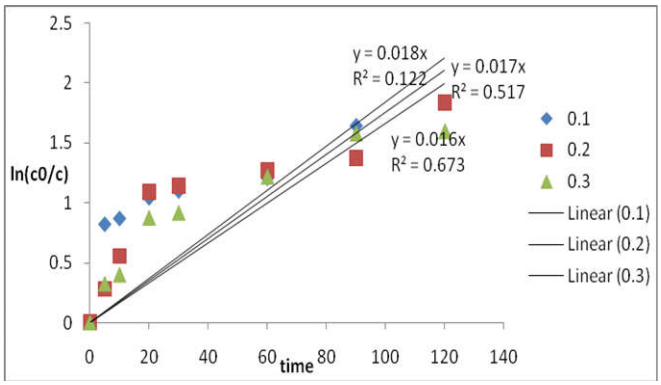

Fig. 12. Time vs ln C/C0

Figure show The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations.

Percentage Removal, %R = (C0−Cf) × 100 / C0

Table 4. Percentage removal calculation

Sr no	40ppm	0.1 g/L		0.2 g/L		0.3 g/L	
1	350ºC–waste tea	83.99%		84.04%		79.83%	
	Kinetic models						
Sr.no	parameters	K1	R^2	K2	R^2	K3	R^2
1	350ºC–waste tea	0.018	0.122	0.017	0.51	0.016	0.67

Result : the result was found in the waste tea 84.04 % removal of dye at 40ppm on 0.2g/L

That indicates that the adsorption perfectly obeys the pseudo-first-order model. Meanwhile, the result showed that the rate of adsorption depended on the availability of adsorption sites on the surface of adsorbent rather than MB concentration in bulk solution.

Effect of Temperature: The adsorption of MB on-conventional stirring process was investigated as a function of temperature and maximum removal of MB was obtained. Experiments were performed at different temperatures of 30, 40 and 50 °C for the initial MB concentration of 80 mg/L at constant adsorbent dose of 0.1g/L.

Sample of waste tea at 350 ºC: Sample of waste tea at 350ºC because we got 83.99% of removal dye at 40 ppm so we finalized this 350 ºC temperature parameter and 40 ppm for waste tea sample.

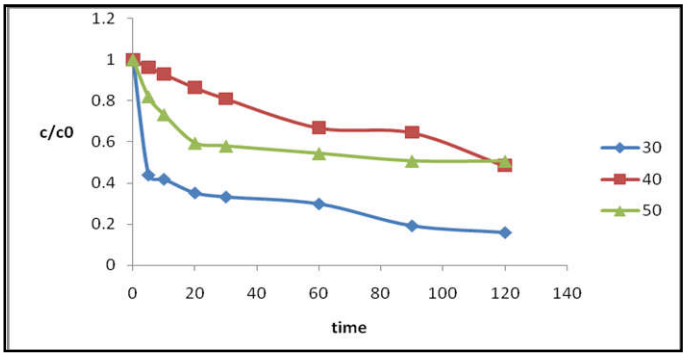

Fig. 13. Time Vs C/C0

Table 5. Percentage removal calculation

Sr no	40ppm	30 ºC		40 ºC		50 ºC	
1	350 ºC –waste tea	83.99%		51.67%		49.37%	
	Kinetic models						
Sr.no	parameters	K1	R^2	K2	R^2	K3	R^2
1	350 ºC –waste tea	0.018	0.12	0.005	0.96	0.007	0.88

Result: the result was found in the waste tea 83.99% removal of dye on 40ppm at 30 ºC.

Figure Shows Effect of Temperature for the adsorption of MB dye onto conventional stirring process -(The At various temperature 30,40,50ºC, Dye concentration of 40 ppm and time 2hr and 0.1 g/L.

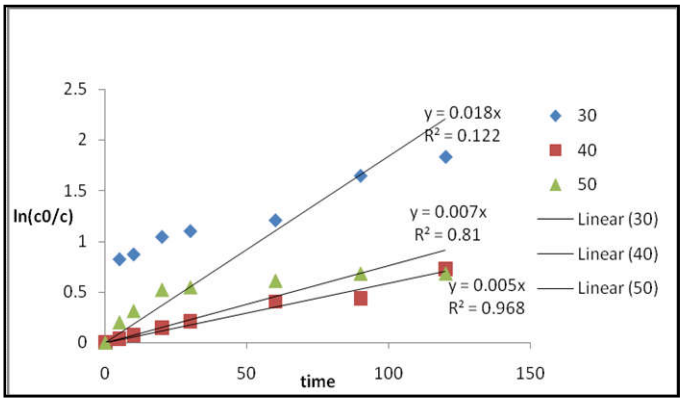

Fig. 14. Time Vsln(C0/C)

Figure show The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations.

That indicates that the adsorption perfectly obeys the pseudo-first-order model. Meanwhile, the result showed that the rate of adsorption depended on the availability of adsorption sites on the surface of adsorbent rather than MB concentration in bulk solution.

Conclusion

The adsorption of Methylene blue dye on low cost bio-waste adsorbent i.e. biosorbent from waste tea have been studied by mechanical stirring .The prepared biosorbent from the waste tea have been successfully used in the present study for removal of methylene blue dye from the waste water. The removal efficiency of prepared biosorbent was well controlled by biosorbent concentration and initial solution concentration. Based on the above results, it can be concluded that the prepared activated carbon could be used as low-cost adsorbent for the colour removal of dyes. Characterization tests where we found preferable and desired results for the waste tea activated carbon, the SEM test was carried out to observe pore structure of better adsorbent activated carbon. The experimental data were found to the pseudo-first-order. Kinetic study is important to the adsorption process because it depicts the uptake rate of adsorbate and controls the residual time of the whole adsorption process. To explore the controlling mechanism of the adsorption process. The parameters of kinetic models parameters and the correlation coefficient (R^2) values for different initial concentrations.

As can be seen, the R^2 values obtained from the pseudo first-order were consistently higher. In addition, the C_A values coincided with the expected C_A values (C_A,exp). That indicates that the adsorption perfectly obeys the pseudo-first -order model. Meanwhile, the result showed that the rate of adsorption depended on the availability of adsorption sites on the surface of adsorbent rather than MB concentration in bulk solution. Finally at 350ºc temperature parameter of waste tea activated carbon is good absorbent. because its gives at initial concentration on removal of MB is 83.99% .and adsorbent loading on Removal of MB is 84.4 % adsorbent loading was 0.2 g/L .and the temperature on Removal of MB is 83.99 % temperature was at 30 ºC .all the percentage value is highest than other two. The parameters of kinetic models parameters and the correlation coefficient (R^2) values for initial concentrations on removal of MB the K1 is 0.008 and R^2 is 0.05 .and adsorbent loading on removal of MBthe K1 is 0.018 and R^2 is 0.122 and temperature on removal of MB the K1 is 0.018 and R^2 is 0.12.

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