

Synthesis of Microporous Activated Carbon (MAC) Using Compressed Wood as Waste Biomass Via Pyrolysis for Adsorption of Crystal Violet

Sundus Saeed Qureshi, Sabzoi Nizamuddin*, Muhammad Tahir Hussain Siddiqui, Nabisab Mujawar Mubarak*, Humair Ahmed Baloch, Noor Rosyidah Sajuni

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Abstract: Microporous activated carbon (MAC) was produced using compressed wood as biomass residue by means of chemical activation by utilizing phosphoric acid as an activation agent through pyrolysis in N₂ atmosphere. The process was optimized by conducting the experiments at various ranges of operating conditions including impregnation ratio at 1:1 to 1:4 and pyrolysis temperatures of 500 to 900°C. Furthermore, MAC produced was employed for the adsorption of crystal violet. The adsorption efficiency was analyzed by varying process parameters such as pH, agitation rate and agitation time. From that, the highest adsorption efficiency of 73.2% was achieved. In addition, the adsorption isotherm exhibited an incredible adsorption capacity of 311.34 mg/g that shows great potential of MAC for environmental remediation. MAC was further characterized to explore structural up gradation and qualitative key functional groups by using field emission scanning electron microscopy (FESEM) and Fourier transform infrared (FTIR). The kinetic study revealed the fitness of pseudo second order kinetic model for the absorption of crystal violet dye. This study presents synthesis of cost effective synthesis of MAC that is highly effective for the adsorption of crystal violet.

Keywords—Activated Carbon, Compress Wood, Pyrolysis, Adsorption, Crystal Violet.

INTRODUCTION

Activated carbon is an amorphous material that is well known for its outstanding structural characteristics including high porosity, enormous surface area and having high stability. This allows the application of activated carbon in numerous areas including air purification, removal of contaminants from water, removal of industrial gases, and discoloration in chemical and pharmaceutical industries, even in the production of oral carbon tablets (Soleimani & Kaghazchi 2008). Activated carbon can be produced from a variety of carbonaceous materials such as coal, wood, sawdust, coconut shell, fruit stones like apricot, cherry stones, grape seeds, hard shells like almond, pistachio and pecan shell, sugarcane bagasse and many others (Hashemian et al. 2014). Activated carbon is commonly produced by physical activation or chemical activation. The properties of activated carbon, such as its porous structure, are always depending on the activating agent employed in the activation process, raw materials and the preparation method employed (Barroso-Bogeat et al. 2015). However, the cost of activated carbon is considerably high, so the agricultural and industrial waste with low cost and naturally available are utilized for the production of activated carbon to remove the pollutants or contaminants from

Sundus Saeed Qureshi, Institute of Environmental Engineering & Management, Mehran University of Engineering and Technology, Jamshoro, Sindh, Pakistan.

Sabzoi Nizamuddin*, School of Engineering, RMIT University, Melbourne, Australia. E-mail: nizamuddin248@gmail.com

Muhammad Tahir Hussain Siddiqui, School of Engineering, RMIT University, Melbourne, Australia.

Nabisab Mujawar Mubarak*, Department of Chemical Engineering, Faculty of Engineering and Science, Curtin University, Sarawak, Malaysia. E-mail: mubarak.yaseen@gmail.com

Humair Ahmed Baloch, School of Engineering, RMIT University, Melbourne, Australia.

Noor Rosyidah Sajuni, Department of Chemical and Petroleum Engineering, Faculty of Engineering, UCSI University Kuala Lumpur, Malaysia.

wastewater(Barroso-Bogeat et al. 2015).Nowadays, the production of activated carbon from agricultural waste or industrial waste is a concern topic of active research and investigated by different researchers(Gomez-Serrano et al. 1998).

In recent year, there has been a growing concern with environmental protection. Removal of contaminated media or decrease the influx of pollutants was required for the environment(Hesas et al. 2013). The hazardous dyes discharge from many industries and cause harmful effects on the human body such as vomit and inducing shock. Hence, the treatment of dyes is an important issue(Njoku et al. 2014). Activated carbon is one of the widely used and effective adsorbent due to its high adsorptive capacity that is milligrams of adsorbate retained per gram of carbon(Aworn et al. 2008). Activated carbon plays an important role in removing impurities, hazardous components, and diverse organic compounds such as heavy metal, dyes, pharmaceuticals and surfactants(Pragya et al. 2013).

The carbonaceous materials produced by pyrolysis contain higher carbon content (Veksha et al. 2014). Pyrolysis is a well-known technique used for waste treatment and synthesis of activated carbon with high efficiency(AlOthman et al. 2014). The pyrolysis provides heating to the walls between the pores of carbonaceous material which causes the expansion of porous structure of activated carbon, ultimately producing more pores. In pyrolysis process, the carbonaceous materials are carbonized at high temperature under nitrogen flow (Nowicki et al. 2015). Furthermore, chemical activation of carbonaceous materials is a commonly used method for synthesis of activated carbon. The raw material is impregnated with chemical agent, and then pyrolyzed for carbonization in an inert atmosphere (Shim et al. 2001). The purpose of impregnation with chemical agents is to help in developing the porosity of activated carbon by means of dehydration and degradation. After impregnating the precursor by chemical agents and heat treatment of the mixture, the impregnating agent and its salts are removed by washing with acid/base and water, which makes the pore structure available in the activated carbon. Chemical activation presented several advantages such as it is conducted in a single step, low activation temperature, low energy costs, higher activated carbon yield, shorter activation time, high surface area and well-developed microporosity. Although, the chemical activation disadvantages are required for deep washing for the removal of impurities generated by the process as well as activating agents cost and cohesiveness of the general process(Yorgun &Yildiz 2015).

In this study, microporous activated carbon (MAC) was synthesized via conventional heating. The effect of process parameter for synthesis of activated carbon was discussed and was optimized. High-performance activated carbon was used for removal of crystal violet from waste water. The isotherm and kinetic study also were investigated.

MATERIALS AND METHODS

Raw Materials

Compress woods obtained from wood industries are used as raw material for production of activated carbon. Blender was used to crush the compress wood into very fine particles and then dried in an oven for 8 hours. Moreover, 200 mg/L of crystal violet dye was prepared by dissolving certain amount of crystal violet powder in distilled water.

Production of Activated Carbon

In this study, activated carbon was produced from compressed wood through chemical activation by using phosphoric acid (H_3PO_4) as a chemical activating agent. 30g of compressed wood was impregnated with H_3PO_4 at a different impregnation ratio of 1:1, 1:2, 1:3 and 1:4. The compress wood that impregnated with chemical agent was agitated by shaker at 150 rpm for 1.5 hour and dried in an oven at 100°C. The dried impregnated sample was then carbonized at 500-900°C by pyrolysis under the nitrogen flow for 30 min at heating rate 20°C/min. After activation, the carbonized sample was washed with distilled water to a neutral pH to remove the impregnating agent and its salts, which makes the porous structure available in the activated carbon. The sample was filtered after treatment, and resulting solid sample was dried in oven at 100°C.

Characterization of Activated Carbon

The activated carbon produced at optimum conditions was characterized for scanning electron microscopy and Fourier transform infra-red in order to study the structural properties and functional groups present on the surface of activated carbon. The surface morphology of activated carbon was determined by FESEM, (Brand: Zeiss, Model: Auriga), whereas FTIR analysis was carried out using a fourier transform infra-red spectroscope (Bruker, IFS66v/S).

Batch Adsorption Studies

The optimum production of activated carbon was at impregnation ratio of 1:1 with H_3PO_4 and carbonization temperature of $900^\circ C$. This optimized activated carbon was selected to study the batch adsorption experiments. Batch adsorption experiments were carried out to study effect of parameters such as initial operating pH, agitation rate, and agitation time for removal of crystal violet. The experimental runs of those variables were designed by Design Expert software. Initial pH of dye solution was set in the range of 2-10 and the pH was adjusted by addition of NaOH and HCl solution. 0.5 g of activated carbon was added into each flask with different pH of the dye solution and agitated by shaker at agitation speed of 100-150 rpm and agitation time of 20-80 min. After agitation with a certain period of time, sample was filtered to separate the adsorbent and adsorbate. The dye solution was placed in cuvettes and measured by UV-Vis spectrophotometer at 545 nm. Adsorption equilibrium was studied by isotherm models. Moreover, adsorption kinetic experiments were developed by agitating 200 ml of dye solution at different concentrations at 150 rpm for 100 min. Samples were pipetted and filtered at every 10 min intervals. After filtration, the concentration of each sample solution was measured by UV-Vis spectrophotometer at 545 nm. Different kinetic models were employed to analyze the kinetic experimental data.

Batch adsorption experiments were carried out by agitating dye solution of different initial concentration with activated carbon. In this experiment, 0.5 g of activated carbon was measured by electronic balance and poured into a conical flask. 200ml of 5 ppm initial concentration of dye solution was prepared and the pH of dye solution was adjusted to the optimum pH (pH 2) as get in the previous experiment. The sample was agitated in the optimum agitation speed (150 rpm) for 100 minutes. Samples were pipetted out at every 10 minutes intervals and filtered by filter paper. A few ml of dye solutions were pipetted into cuvettes after filtration for determined and analyzed by using UV-Visible Spectrophotometer at 545 nm. The procedure was repeated with different concentration of dye solutions, which are 10 ppm, 15 ppm, 20 ppm and 25 ppm.

Adsorption isotherms

Adsorption isotherms are interaction of adsorbate with an adsorbent and are used to optimize a critical use of adsorbent. The essential for adsorption data interpretation and calculation of relative experimental equilibrium data is to use either a theoretical or an empirical equation. The isotherm models can be used to describe adsorption isotherms from experimental data obtained. The most two famous isotherms, Langmuir and Freundlich isotherm models were applied to fit experimental equilibrium isotherm data on adsorption (Ahmed & Dhedan 2012). However, the amount of dye adsorbed can be calculated by using the equation as follows:

$$q_e = \frac{(C_o - C_e)V}{W} \quad (1)$$

Where C_o (mg/L) is an initial concentration of dye, C_e (mg/g) is a final concentration of dye at equilibrium, V (L) is the volume of solution and W (g) is weight of activated carbon used (Hameed & El-Khaiari 2008). The percentage of dye removal in solution was calculated by following equation:

$$\% \text{ Removal} = \frac{(C_o - C_e)}{C_o} \times 100 \% \quad (2)$$

Where C_o (mg/L) is the initial concentrations of dye solution, and C_e (mg/g) is the final concentrations of dye solution.

The Langmuir isotherm model assumes that adsorption takes place at specific homogeneous sites within the monolayer adsorption. The mathematical expression of Langmuir isotherm is shown by following equation:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (3)$$

Where C_e (mg/L) is the concentration of dye solution at equilibrium, q_m (mg/g) is the maximum amount of crystal violet adsorbed per unit mass of activated carbon and K_L (L/mg) is the Langmuir constant related to adsorption rate. The critical characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant called equilibrium parameter, R_L , which is written as following equation:

$$R_L = \frac{1}{1 + K_L C_o} \quad (4)$$

Where C_o (mg/L) is the maximum initial concentration of dye solution. The value of R_L indicates the type of isotherm to be reversible if R_L equal to 0, favorable if 0 smaller than R_L and R_L smaller than 1, linear if R_L equal to 1 or unfavorable if R_L larger than 1 (Ahmed & Dhedan 2012).

Freundlich isotherm model is a non-ideal, reversible and multilayer adsorption with non-uniform distribution of adsorption over a heterogeneous surface. This isotherm does not indicate the quantity or a restricted adsorption capacity of the adsorbent and it only be practically employed on the concentration ranges from low to intermediate adsorbate. The mathematical equation expression is as following equation:

$$qe = K_F C_e^{1/n} \quad (5)$$

Where K_F ((mg/g) (L/mg)^{1/n}) and n are Freundlich constants which give a measure of adsorption capacity and adsorption intensity, respectively (Ahmed & Dhedan 2012).

Adsorption kinetic

The adsorption kinetics studies were used to carry out the rate of adsorption process. It described the rate of adsorbate uptake at the solid-solution interface at the residence time. The adsorption kinetics of activated carbon was usually analyzed by using three kinetic models, which are pseudo-first order, pseudo-second order and intraparticle diffusion models (Arockiaraj et al.). The amount of dye adsorbed can be calculated by using the equation as follows:

$$q_t = \frac{(C_0 - C_t)V}{W} \quad (6)$$

Where C_t (mg/L) is the liquid phase concentrations of dye at time t (min), V (L) is the volume of the solution and W (g) is the weight of activated carbon used (Hameed & El-Khaiary 2008).

In pseudo-first order kinetic model, the rate constant of adsorption is identified. The equation of pseudo-first order was written as follows:

$$\ln(q_t - q_e) = \ln(q_e) - K_1 t \quad (7)$$

Where q_e (mg/g) is the amount of dye adsorbed at equilibrium, q_t (mg/g) is the amount adsorbed at time (t) and k_1 (1/min) is rate constant of adsorption (Ahmed & Dhedan 2012).

The pseudo-second order kinetic model equation was based on equilibrium adsorption. It can be written as following equation:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e} + \frac{t}{q_e} \quad (8)$$

where K_2 (g/mg, min) is the rate constant of second-order equation, q_e (mg/g) is the amount of dye adsorbed at equilibrium and q_t (mg/g) is the amount adsorbed at time (t) (Ahmed & Dhedan 2012).

The intraparticle diffusion model describes the possibility of intraparticle diffusion resistance affecting the adsorption and it was expressed as following equation:

$$q_t = k_p t^{1/2} + C \quad (9)$$

where k_p (mg/g min^{1/2}) is the rate constant of intraparticle diffusion and C (mg/g) is a constant that gives an idea about the thickness of the boundary layer (Ahmed & Dhedan 2012).

RESULTS AND DISCUSSION

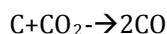
The Effect of Impregnation Ratio and Activation Temperature

Impregnation ratio is the most significant affected variable for the development of porous structure in activated carbon (Nowicki et al. 2015). Chemical agents used in chemical activation acted as a significant activation agent for the production of activated carbon, which helps to enhance the porosity of activated carbon. The carbonaceous raw materials are barely heated or burned without using a chemical agent. During the activation process, activated carbon obtains the energy from pyrolysis more easily after the enlargement of pore structure on carbonaceous materials by impregnation of the chemical activating agent. In this study, the effect of the different impregnation ratio on the pore volume and surface area was investigated. Compress wood as the precursor of activated carbon was impregnated with H_3PO_4 by an impregnation ratio of 1:1, 1:2, 1:3 and 1:4. H_3PO_4 is a strong dehydrating agent that accelerated the bond cleavage reactions and caused the carbonaceous materials to enlarge and opens the surface structure, and leading pore formation in activated carbon (Ioannidou & Zabaniotou 2007). The impregnated compress wood was then carbonized at temperature of 500, 600, 700, 800 and 900°C by pyrolysis under nitrogen flow. For production of activated carbon, activation temperature plays an important role in the pyrolysis of precursors, which directly affected its surface area and adsorption capacity. In pyrolysis, activated carbon is developed to a different structure of surface area which depends on raw materials used and operating temperature of pyrolysis (Nowicki et al. 2015). During pyrolysis, the tube of reactor was purged with nitrogen gas to provide an inert atmosphere. This is because the presence of oxygen in pyrolysis treatment will reduce the surface area and lower the porosity of activated carbon. Further, the optimum

production of effective activated carbon was carried out at an impregnation ratio of 1:1 and activation temperature of 900°C. Generally, the adsorption capacity was increased with intensification of activation process by increasing an impregnation ratio. But at the same time, the pores could be blocked as the impregnation ratio was increased beyond the optimum value due to excess of chemical activating agents that left on surface of carbon. It was leading to a reduction in accessible area of carbon materials. Other reasons for decreasing adsorption capacity of activated carbon by past expanded or heated the pores and causes damage of pores in carbon (Yorgun & Yildiz 2015). Based on the results, it is proved that an excellent adsorption capacity of activated carbon was yielded at higher temperature.

Characterization of activated carbon

Scanning electron microscope technique was employed to observe the surface morphology of prepared activated carbon sample. Fig 1 shows image of selected activated carbon sample produced at 900 °C and it was observed that the pores of different size and different shape were developed. During physical activation by CO₂ gas, the diffusion of the oxidizing agent through the carbonaceous matrix involves the removal of impurities and consumption of carbon to create porosity according to the following reaction



The micro porosity is opened and widened with even a shift to meso and macro porosity as the removal of exterior of particles is significant at high burn off (Rodriguez-Reinoso & Molina-Sabio 1992). This shows that CO₂ were effective in creating well-developed pores on surface of the precursor, hence produced activated carbon with large surface area and porous structure, which had a high adsorption capacity on crystal violet.

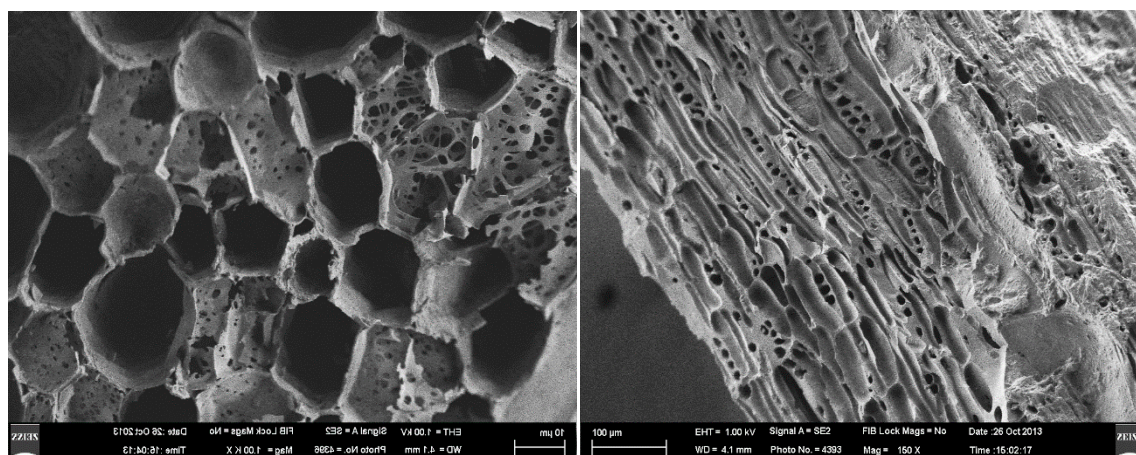


Figure 1: SEM analysis of activated carbon produced at impregnation ratio of 1:1 with H₃PO₄ and 900 °C.

The spectra for selected compressed wood based activated carbon displayed the following bands with functional group. The intensity of the band in the 3596-2833 cm⁻¹ region shows -OH groups resulted from the presence of phenol, the width and asymmetry of this band indicate the presence of strong hydrogen bonds. These hydrogen bonds could be a result of strongly adsorbed water on hydrophilic functional groups on the AC surfaces. The band at 1865 cm⁻¹ shows a carbonyl compound (carbonyl group); while at 1626 cm⁻¹ shows the presence of -NH₂; the bands at 1396, 1002, 975 and 829 cm⁻¹ contain saturated carbon (methyl group, -CH₃); and finally the bands at 700, 835 cm⁻¹ include vibration of the bond C-Cl and CH₂Cl. These results agreed with surface chemistry of other agricultural by products.

Adsorption Isotherm

The adsorption of crystal violet with different pH of dye solutions was analyzed by Langmuir and Freundlich models.

The Langmuir plot for adsorption study was shown in the Figure 2. From the graph of Langmuir, the correlation coefficients of R² were carried out that higher than 0.950 at all the pH values. In this case, the Langmuir isotherm was favorable for the adsorption of crystal violet by activated carbon that was synthesised from compressed wood.

This is because of the values of R_L which were determined between 0 and 1. The values of q_m were calculated from the general equation of Langmuir, the maximum amount of crystal violet adsorbed per unit mass of activated carbon in pH 2, 6 and 10 are 182.56 mg/g, 206.06 mg/g and 140.13 mg/g, respectively.

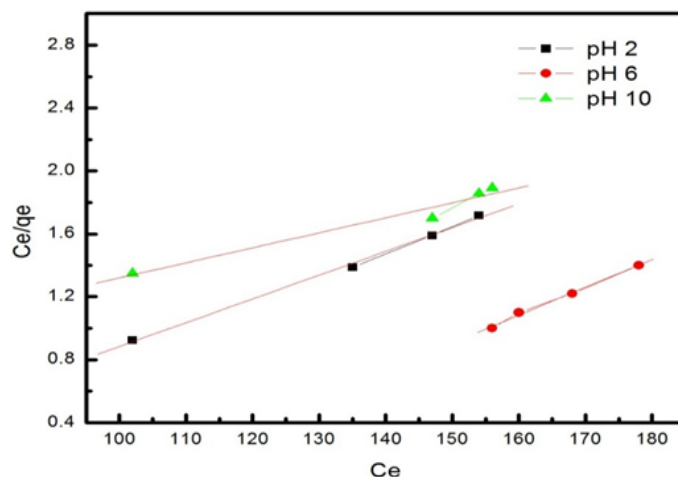


Figure 2: Langmuir plots for adsorption of crystal violet at pH 2 of dye solution, agitation rate of 150 rpm and agitation time of 80 min.

In Freundlich model, k_f and $1/n$ can be determined from the linear plot of $\log q_e$ versus $\log C_e$ as shown in Figure 3. From the data in Table. 1, the slope of $1/n$ value was larger than 1 which measured adsorption intensity or surface heterogeneity. The $1/n$ values indicated unfavorable adsorption of crystal violet on activated carbon. The results revealed that the adsorption data fitted by the Langmuir model was preferred than Freundlich model.

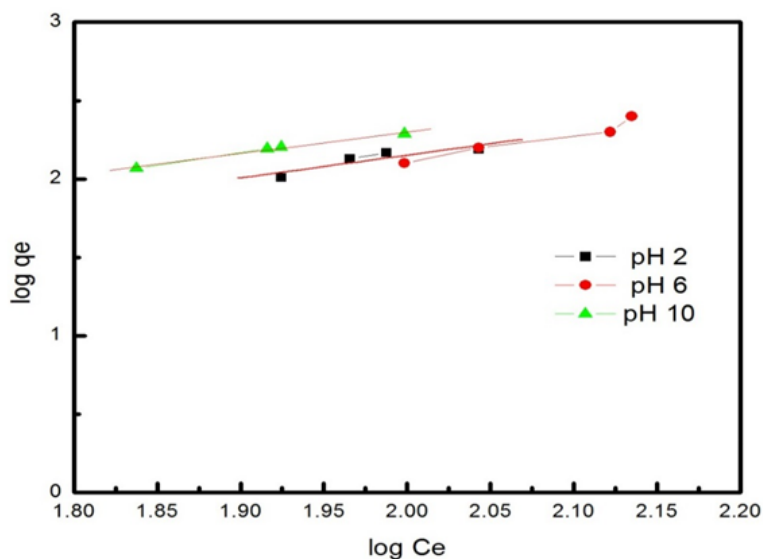


Figure 3: Freundlich plots for adsorption of crystal violet at pH 2 of dye solution, agitation rate of 150 rpm and agitation time of 80 min.

Table 1: Langmuir and Freundlich parameters for adsorption of crystal violet at pH 2 of dye solution, agitation rate of 150 rpm and agitation time of 80 min.

Langmuir Isotherm					Freundlich Isotherm		
pH	q_m (mg g^{-1})	K_L (L mg^{-1})	R^2	R_L	K_F (Lmg^{-1})	n	R^2
2	206.06	0.015	0.997	0.9944	0.757	0.687	0.806
6	182.56	0.011	1.000	0.9936	1.896	0.514	1.000
10	140.13	0.021	0.983	0.9924	0.440	0.73	0.987

Adsorption Kinetic Studies

For adsorption kinetic studies, different initial concentrations (5-25 ppm) of crystal violet solution were agitated with 0.5 g of activated carbon at room temperature. The adsorbed amount of crystal violet was calculated from the equation (6) and plotted in Figure 4.

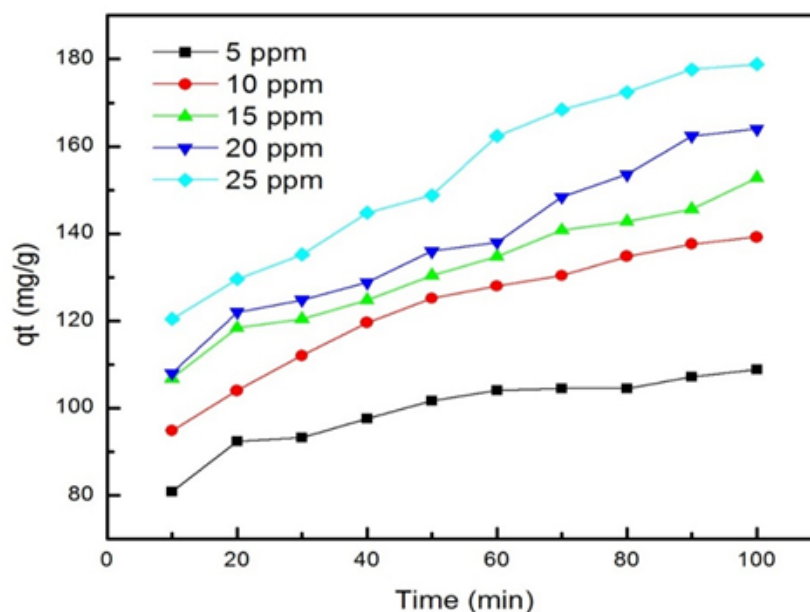


Figure 4: Effect of agitation time on crystal violet adsorbed amount at different initial concentrations (pH = 2, dose = 0.2g/L, and agitation rate of 150 rpm).

The adsorption kinetics data were analyzed by pseudo-first order (Figure 5), pseudo-second order (Figure 6) and intraparticle diffusion (Figure 7) models. The values of constant and adsorbed crystal violet of each model were listed in Table 2.

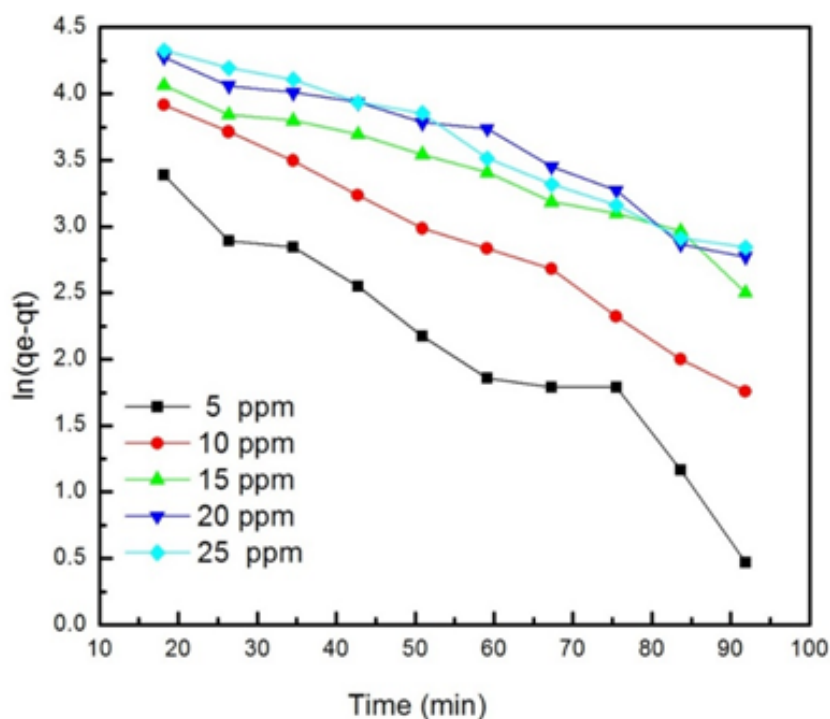


Figure 5: Pseudo-first order model for adsorption of crystal violet at different initial concentrations (at pH 2 of dye solution, dose = 0.2g/L and agitation rate of 150 rpm)

The values of k_1 were obtained from the slope and intercept of the pseudo-first order plot. The data of k_1 values and the corresponding linear regression, correlation coefficient R^2 were tabulated in Table 2. The values of q_e were calculated from the general equation of pseudo-first order. It represented the values of crystal violet adsorbed at equilibrium for 5ppm, 10ppm, 15ppm, 20ppm and 25ppm are 1.017 mg/g, 1.065 mg/g, 1.093 mg/g, 1.104 mg/g and 1.107 mg/g, respectively.

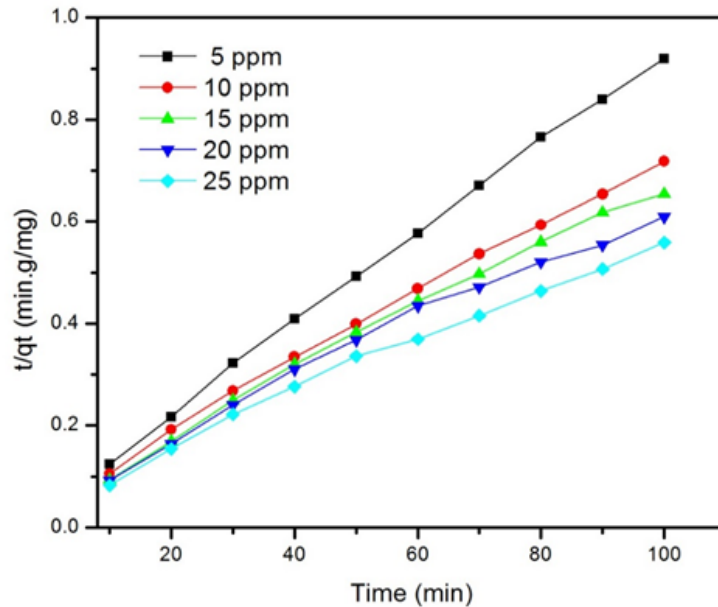


Figure 6: Pseudo-second order model for adsorption of crystal violet at different initial concentrations (pH = 2, dose = 0.2 g/L, and agitation rate of 150 rpm)

The equilibrium adsorption capacity (q_e) and the rate constant of second order (k_2) can be carried out by the intercept and slope of graph of pseudo-second order model. The data of k_2 values, q_e value and the corresponding linear regression, correlation coefficient R^2 were tabulated in Table 2. The adsorption capacity of 5, 10, 15, 20 and 25 ppm are 124.86, 209.84, 254.83, 285.79 and 311.34 mg/g, respectively. The correlation coefficient values of pseudo-second order kinetic model obtained was preferred as compared to the pseudo-first order kinetic model. Therefore, the pseudo-second order was better fit than pseudo-first order kinetic model.

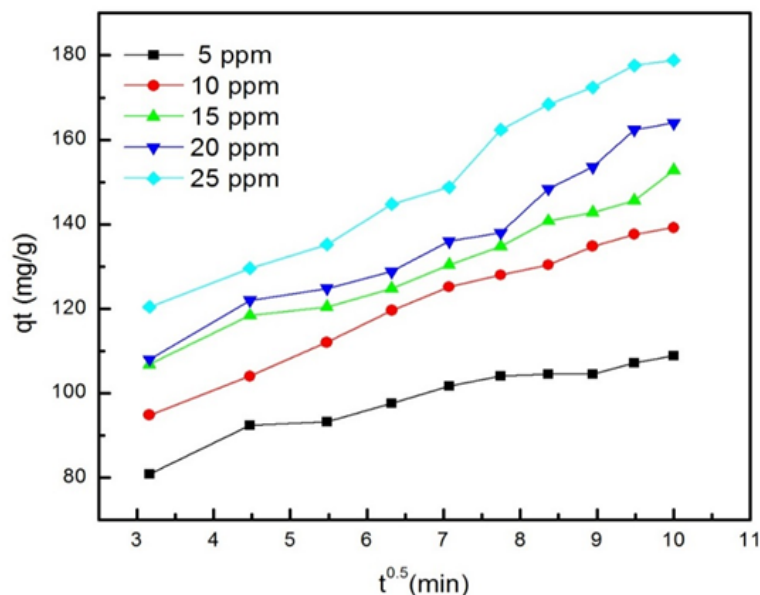


Figure 7: Intraparticle diffusion model for adsorption of crystal violet at different initial concentrations (pH = 2, dose = 0.2 g/L, and agitation rate of 150 rpm)

According to intraparticle diffusion model, the graph that plotted should be linear if intraparticle diffusion is involved in the adsorption process. A value of C close to zero represents that diffusion is the only controlling step of adsorption process. Thus, if the linear line passes through the origin, then intraparticle diffusion is the rate controlling step (Demiral et al. 2008, Kalderis et al. 2008). The data of C values, k_3 values and corresponding linear regression, correlation coefficient R^2 were tabulated in Table 2. Based on the graph, the line was not passed through the origin and this represented of some degree of boundary layer control. It shows that the intraparticle diffusion is not the only rate limiting step, but also the kinetic model which can control the rate of adsorption.

Table 2: Kinetic parameters of pseudo-first order, pseudo-second order and intraparticle diffusion for crystal violet adsorption on different concentration of dye solution.

C ₀ mgL ⁻¹	Pseudo-First order			Pseudo-Second order			Intraparticle diffusion model		
	q _e , cal. (mg g ⁻¹)	K ₁ (min ⁻¹)	R ²	q _e , cal. mg g ⁻¹	K ₂ (min/g mg ⁻¹)	R ²	q _e , cal. (mg g ⁻¹)	K ₃	R ²
5	1.017	0.280	0.947	124.86	0.088	0.988	84.96	2.632	0.864
10	1.065	0.236	0.992	209.84	0.067	0.997	96.42	4.751	0.937
15	1.093	0.153	0.957	254.83	0.062	0.993	106.10	4.659	0.979
20	1.104	0.164	0.944	285.79	0.056	0.991	105.50	6.009	0.980
25	1.107	0.177	0.982	311.34	0.051	0.986	116.10	6.860	0.976

CONCLUSION

We have successfully synthesized micro porous activated carbon using agricultural waste i.e. compress wood and produced activated carbon was used for crystal violet removal from wastewater. The optimum production of activated carbon was at impregnation ratio of 1:1 with H₃PO₄ and carbonization temperature of 900°C. The effect of parameter such as pH, agitation rate and time was studied. The highest percentage for removal of crystal violet by activated carbon was 73.02%, which at pH 2 of dye solution, agitation rate of 150 rpm and agitation time of 80 min. This study proved that by increasing carbonization temperature, agitation rate and agitation time, the adsorption capacity of activated carbon was increased. Moreover, adsorption isotherms were developed by Langmuir and Freundlich isotherm model. The result shows that, the adsorption data fitted by the Langmuir model was preferred than Freundlich model. The adsorption kinetic was studied by pseudo-first order kinetic model, pseudo-second kinetic model and intraparticle diffusion model. However, pseudo-second order fits better than pseudo-first order kinetic model with 311.34 mg/g of crystal violet adsorbed amount. Therefore, the optimum operating condition of activated carbon was carried out in this study and it proved that the production of activated carbon from compressed wood has a good adsorption capacity for the removal of crystal violet.

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