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Adsorption of Crystal Violet onto Adsorbents Derived from Agricultural Wastes: Kinetic and Equilibrium Studies

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ABSTRACT: Suitability of adsorbents derived from groundnut shell (GS) and bean pod (BP) in removing crystal violet from aqueous solution was evaluated in a series of batch experiments. Findings reveal that the points of zero charge (PZC) of GS and BP are 3.50 and 3.00 respectively. Findings also show that GS and BP exhibit maxima adsorption at pH values of 6.00 and 5.00 respectively. For both GS and BP, pseudo-second order model gives the best fit for the sorption kinetic data while Freundlich isotherm gives the best fit for the sorption equilibrium data. These findings suggest that the adsorption of crystal violent onto GS and BP probably involves chemisorption and the adsorption does not reach a plateau as the concentration of crystal violet in the aqueous solution increases. This study demonstrates the suitability of GS and BP as low-cost adsorbents for removing crystal violet from industrial wastewater. © JASEM

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KEYWORDS: Adsorption, Bean pod, Crystal violet, Equilibrium, Groundnut shell, Kinetics

INTRODUCTION

Many industries, such as dyestuff, textile, pharmaceutical, paper, plastic, and tannery, make use of dyes to color their products. Of these industries, textile industry is the largest consumer of dyes. It has been shown that about 10,000 different textile dyes with an estimated annual production of 7 x 10^5 metric tons are commercially available worldwide (Carmen and Daniela, 2012). As a result of the enormous utilization of these dyes, substantial amount of dyes are frequently released as wastewater into aquatic environment. In recent time, there is a growing interest and concern over contamination of the aquatic environment by dyes. This is because the release of dyes into aquatic environment causes reduction in the growth of algae due to obstruction of light required for photosynthesis, which subsequently leads to ecological imbalance in the aquatic ecosystem (de Sousa *et al*, 2012). Contamination of aquatic environment by dyes has also been shown to have toxic, carcinogenic, and aesthetic effects on humans (Ratna and Pahdi, 2012).

Crystal violet, also known as gentian violet, is a triarylmethane dye. Medically, the dye is used as an antiseptic with a selective action on gram-positive organisms. It is also used for the treatment of burns, boils, carbuncles, and mycotic skin infections (The Council of Pharmaceutical Society of Great Britain, 1973). Due to the deleterious effects of dyes on aquatic ecosystem, different approaches have been proposed for the removal of dyes in contaminated effluents. These methods can broadly be classified as biological, chemical and physical (Adinew, 2012). Specifically, decolourization, degradation or removal of crystal violent from aqueous solution has been investigated using biodegradation methods (Azmi *et al*, 1998; Oranusi and Ogugbue, 2005; Zablocka-Godlewska *et al*, 2009; Thorat and Sayyad, 2010; Gursahani and Gupta, 2011; Tom-Sinoy *et al*, 2011; Kunjadia *et al*, 2012), methods involving chemical reaction (Mohammed *et al*, 2011; Patil, 2011; Su and Wang, 2011; Fayoumi *et al*, 2012; Ovejero *et al*, 2012) and adsorption methods (Chen *et al*, 2011; Lin *et al*, 2011; Nidheesh *et al*, 2011; Patil *et al*, 2011; Rammel *et al*, 2011; Gandhimathi *et al*, 2012; Nidheesh *et al*, 2012a; Pandian *et al*, 2012; Patil and Shrivastava, 2012; Prasad and Santhi, 2012; Shouman *et al*, 2012). Among these three approaches, adsorption is the preferred method because it does not leave any toxic residue behind in the treated water.

Although adsorption by activated carbon is considered to be very effective in removing dyes from contaminated water, the cost of activated carbon and the difficulty associated with its regeneration impose a serious restriction on its routine use as adsorbent (Allen and Koumanova, 2005; Bhatnagar and

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Minocha, 2006; Foo and Hameed, 2010; Grassi *et al*, 2012). Recently, efforts are being made worldwide to invent more effective, low cost, environmental friendly and easily regenerated adsorbents from agricultural wastes (Mahmoud *et al*, 2012; Sharma *et al*, 2012; Rashed, 2013). It is in view of this current global endeavor that the present study aims at investigating the kinetics and equilibrium isotherms of groundnut shells and bean pods as low cost adsorbents for the removal of crystal violet from aqueous solution.

MATERIALS AND METHODS

Collection and Preparation of Adsorbents: Groundnut shells and bean pods were collected from agricultural wastes disposed of by subsistent farmers during harvest period. The shells and the pods were washed, dried, ground and sieved as described elsewhere (Ajmal *et al*, 2006; Gong *et al*, 2011; Choudhury *et al*, 2012; and Kiran *et al*, 2013). The adsorbents were sieved through a mesh with a pore size of 2 mm. Hence, all the adsorbents with particle size with diameter in the range of ≤ 2 mm were used in this study. The fine particles prepared from Groundnut Shells and Bean Pods were stored in clean, dried, air-tight containers and were labeled GS and BP respectively.

*Determination of Points of Zero Charge of the Adsorbents***:** Point of zero charge (PZC) has been defined as the pH value for which the surface charge of a material is equal to zero at some ambient temperature, applied pressure, and aqueous solution composition (Sposito, 2004). The points of zero charge of GS and BP were determined using solid addition method (Nidheesh *et al*, 2012b). In this method, 50 mL of solution containing 0.1mol/L of $NaNO_3$ was transferred into twelve 250 mL Erlenmeyer flasks. The pH of each solution was adjusted to values ranging from 1.00 to 12.00 by adding an appropriate amount of 1.0 mol/L HNO_3 or 1.0 mol/L NaOH solution before the final volume was made up to 50 mL with distilled water. Thereafter, 1.0 g of GS was added to each flask and the final pH of the supernatants measured using JENWAY 3505 pH meter after agitating the flasks at 200 rpm for 5 hours. The difference between the initial and the final pH was calculated using equation (1). A plot of ΔpH against pH_i was constructed and the point of interception on the pH_i axis gave the value of PZC for GC. The values of PZC for BP was similarly determined as described above for GS.$\Delta pH = pH_f - pH_i$ (1) Where pH_i is the initial pH, pH_f is the final pH and ΔpH is the difference between initial and final pH.

Preparation of Adsorbate Solutions: Crystal violet and other reagents were purchased from Zayo-Sigma Chemicals Ltd, Jos, Nigeria and were used without further purification. All the reagents used were of analytical grade. A stock solution containing 1000 mg/L of crystal violet was prepared by dissolving 1.0 g of the dye in 500 mL distilled water. The resulting solution was transferred into 1 L volumetric flask and then diluted to volume with distilled water. Other working solutions with concentrations of crystal violet ranging from 25 mg/L to 200 mg/L were prepared from the stock solution by serial dilution in 100 mL volumetric flasks. The pH and ionic strength of the working solutions were adjusted to the required pH and ionic strength by adding appropriate amounts of 1.0 mol/L HNO_3 solution and 1.0 mol/L $NaNO_3$ solution. All pH measurements were made using JENWAY 3505 pH meter.

Batch Experiment for Determining the Effects of pH on Removal Efficiency To investigate the effect of pH on the adsorption of crystal violet onto GS, 1.0 g of the adsorbent was added separately into seven 250 mL Erlenmeyer flasks. Thereafter, 50 mL of crystal violet (concentration = 100 mg/L) solution, maintained at constant ionic strength of 0.1mol/L, was then transferred into each flask. The pH of each flask was adjusted to values ranging from 1.00 to 7.00 by adding appropriate amount of 1.0 mol/L HNO_3 solution. This investigation was not carried out at pH values above 7.00 because decolorization of crystal violet occurs in alkaline medium. The flasks were then agitated in a batch experiment using Orbital Shaker WSZ Series at 200 rpm at ambient temperature (28 ± 1 ^{0}C) for 24 hours. All the 7 samples were later centrifuged and the equilibrium concentrations of crystal violent in the supernatants determined spectrophotometrically using UNICO UV-2100 Spectrophotometer. The removal efficiency (expressed as sorption percent) of crystal violet by GS was calculated using equation (2). The experiment described above was carried out twice and the average values of the data obtained were used to make plots of sorption percent against pH. The procedure described above was repeated using BP in place of GS.$Sorption\ (\%) = \left(\frac{C_i - C_e}{C_i}\right) X\ 100$ (2) Where C_i is the initial concentration (measured in mg/L) of crystal violet in solution and C_e is the residual concentration (measured in mg/L) of crystal violet in solution at equilibrium

Batch Experiment for Investigating Sorption Kinetics: Kinetics of crystal violet adsorption onto GS was investigated in a batch experiment by adding 1.0 g of GS into nine 250 mL Erlenmeyer flasks containing 50 mL of the dye solution each. The concentration of crystal violet in each flask was 50 mg/L. The content of each flask was maintained at constant ionic strength of 0.1mol/L and the optimum pH, as determined in the experiment described in the preceding section. Each flask was then agitated using Orbital Shaker WSZ Series at 200 rpm at ambient temperature (28 ± 1 ^{0}C) for 20, 40, 60, 80, 100, 120, 140, 160, or 180 minutes. The samples were then centrifuged and the concentrations of crystal violent remaining in the supernatants were determined spectrophotometrically at 590 nm using UNICO UV-2100 Spectrophotometer. The amount of crystal violet adsorbed on the adsorbent at any given time is calculated using equation (3). This experiment was carried out twice and the fitness of the average data obtained was tested using intraparticle diffusion model (equation 4), pseudo-first order model (equation 5) and pseudo-second order model (equation 6). The procedure described above was repeated using BP in place of GS. $q_t = \frac{V}{m}(C_i - C_t)$. (3) $q_t = k_{int} t^{1/2}$ (4) ; $log\ (q_e - q_t) = \log q_e - \frac{k_1}{2.303}\text{t}$ ……$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}\ t$ (6), Where q_t is the amount of crystal violet adsorbed on the adsorbent (measured in mg/g) at any given time, q_e is the amount of crystal violet adsorbed on the adsorbent (measured in mg/g) at equilibrium, C_i is the initial concentration of crystal violent in solution (measured in mg/L), C_t is the residual concentration of crystal violet in solution at time t, V is the volume of crystal violent solution (measured in L), m is the mass of adsorbent (measured in g), t is time (measured in minutes), k_1 is pseudo-first order rate constant, k_2 is pseudo-second order rate constant, and k_{int} is intraparticle diffusion rate constant

Batch Experiment for Investigating Equilibrium Isotherms: Sorption isotherm study was investigated by adding 1.0 g of GS to eight 250 mL Erlenmeyer flask, containing 50 mL of dye solution each. The concentrations of crystal violet in the flasks ranged from 25 mg/L to 200 mg/L (that is, 25, 50, 75, 100, 125, 150, 175 and 200 mg/L crystal violet). The content of each flask was maintained at constant ionic strength of 0.1mol/L and the optimum pH determined in the previous experiment. Each flask was agitated using Orbital Shaker WSZ Series at 200 rpm at ambient temperature (28 ± 1 ^{0}C) for 24 hours, centrifuged and the equilibrium concentrations of crystal violent in the supernatants determined spectrophotometrically at 590 nm using UNICO UV-2100 Spectrophotometer. The amount of crystal violet adsorbed onto the adsorbent at equilibrium was calculated using equation (7). This experiment was carried out twice and the fitness of the average data obtained was tested using Langmuir adsorption isotherm (equation 8), Freundlich adsorption isotherm (equation 9) and Temkin adsorption isotherm (equation 10). The procedure described above was repeated using BP in place of GS. $q_e = \frac{V}{m}(C_i - C_e)$ (7) $\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max}} bC_e$ (8) $ln\ q_e = ln\ K_F + \frac{1}{n} ln\ C_e$ …9) $q_e = B_1 lnK_T + B_1 lnC_e$ (10)
Where q_e is the amount of crystal violet adsorbed on the adsorbent (measured in mg/g) at equilibrium, C_i is the initial concentration of crystal violent in solution (measured in mg/L), C_e is the residual concentration of crystal violet in solution at equilibrium (measured in mg/L), V is the volume of crystal `violent solution (measured in L), m is the mass of adsorbent (measured in g), q_{max} is maximum adsorption capacity (mg/g), b is the affinity coefficient, B_1 is the maximum binding energy, K_T is the equilibrium binding constant, K_F is the Freundlich constant for sorption capacity, and n is Freundlich constant for intensity.

RESULTS AND DISCUSSION

The results for the determination of points of zero charge (PZC) of GS and BP are presented in Figure 1. As shown in the figure, the PZC values for GS and BP are 3.50 and 3.00 respectively. In other words, at normal atmospheric pressure, temperature of 28 ^{0}C and aqueous solution with ionic strength of 0.1mol/L, the charges on the surfaces of GS and BP are equal to zero at pH of 3.50 and 3.00 respectively. The implication of these findings is that, at pH values below PZC, the surface charges on the adsorbents are positive while at pH values above PZC, the surface charges of the adsorbents are negative (Oluwaseye *et al*, 2011 and Gusmao *et al*, 2013). Points of zero charge reported in literature for adsorbents derived from other agricultural wastes are 7.10 for oil palm fruit fibre (Abia and Asuquo, 2008), 5.00 for sugarcane bagasse (Zhang *et al*, 2011), 6.00 for watermelon shell (Banerjee *et al*, 2012) and 4.18 for orange peel (De Souza *et al*, 2012)

A plot of sorption percent against pH is presented in Figure 2. As shown in the figure, the removal efficiencies of crystal violent from aqueous solution by both GS and BP are less than 50 percent at pH below 4.00. However, as the pH values of the solutions were adjusted to 4.00 and above, the removal efficiencies continue to increase, reaching a maximum value of 80.8% at pH of 6.00 for GS and a maximum value of 84.4% at pH of 5.00 for BP. These findings can be explained using the values of PZC determined for GS and BP in the preceding paragraph.

The higher adsorption of crystal violet by GS and BP at pH values above their PZC is to be expected because crystal violet molecules exist as positively charged ions in aqueous solution, and therefore exhibit enhanced electrostatic attraction for the adsorbents since the surface charges on the adsorbents are negative at higher pH values.

Figures 3, 4 and 5 are plots of intraparticle diffusion, pseudo-first order and pseudo-second order models for testing the kinetics of adsorption of crystal violet on GS. Also displayed in Figures 6, 7 and 8 are plots of intraparticle diffusion, pseudo-first order and pseudo-second order models for testing the kinetics of adsorption of crystal violet on BP. The rate constants and coefficients of determination derived from these plots are summarized in Table 1. As shown in Table 1, pseudo-second order model gives the best fit for the sorption kinetic data for both GS and BP as indicated by the values of the coefficients of determination ($R^2 = 0.804$ for GS and $R^2 = 0.997$ for BP). The pseudo-second order rate constants for the adsorption of crystal violet onto GS and BP are 6.72 x 10^{-3} and 6.05 x 10^{-2} respectively (see Table 1). This result suggests that the rate of crystal violet adsorption onto BP is about nine times higher than the rate of crystal violet adsorption onto GS. Pseudo-second order model obtained in the present study agrees with previous findings reported in literature for the adsorption of crystal violent onto adsorbents derived from cocoa shell (Chinniagounder *et al*, 2011), mycelial biomass of *Ceriporia lacerata* P2 (Lin *et al*, 2011), *Chaetophora elegans* alga (Rammel *et al*, .
2011), *Leucaena leucocephala* Seed Pods (Patil and Shrivastava, 2012) and *Acacia nilotica* Leaves (Prasad and Santhi, 2012). The pseudo-second order model, according to Ho (2006), is base on the assumption that the adsorption process involves chemisorption, which requires valence forces through the sharing or exchange of electrons between the adsorbent and the adsorbate.

The Plots displayed in Figures 9, 10 and 11 are the Langmuir, Freundlich and Temkin adsorption isotherms for testing the equilibrium data of crystal violet adsorption onto GS. Also displayed in Figures 12, 13 and 14 are plots representing Langmuir, Freundlich and Temkin adsorption isotherms for testing the equilibrium data of crystal violet adsorption onto BP. The parameters derived from these plots are summarized in Table 2. As shown in Table 2, the adsorption data fitted well with Freundlich adsorption isotherm for both GS and BP as indicated by the values of the coefficients of determination ($R^2 = 0.805$ for GS and $R^2 = 0.886$ for BP). These findings lend credence to the assumption behind Freundlich equation that the isotherm does not reach a plateau as the concentration of adsorbate in solution increases (Limousin *et al*, 2007). Adsorption isotherms of crystal violet onto adsorbent prepared from sugarcane bagasse, chitosan, cocoa shell, mangrove plant, tamarind fruit shell, mango leaf, teak tree bark and almond tree bark have been described in the literature (Chinniagounder *et al*, 2011; Patil *et al*, 2011 and Shouman *et al*, 2012)

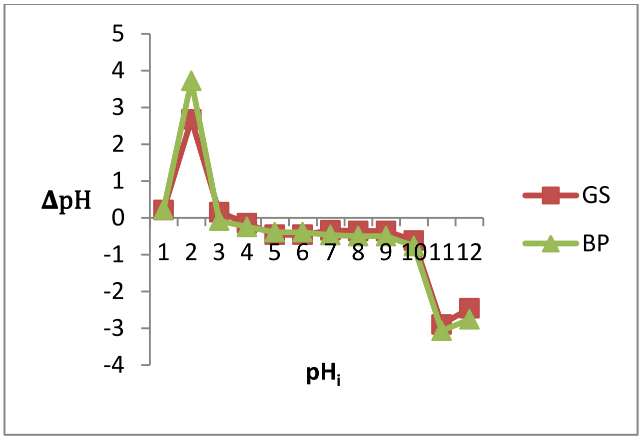

Fig 1: A plot of **Δ**pH against pH_i for the determination of points of zero charge for GS and BP

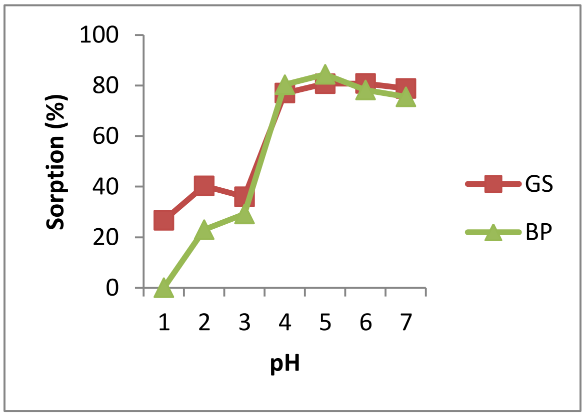

Fig 2: A plot of sorption percent against pH showing the effect of pH on removal efficiency

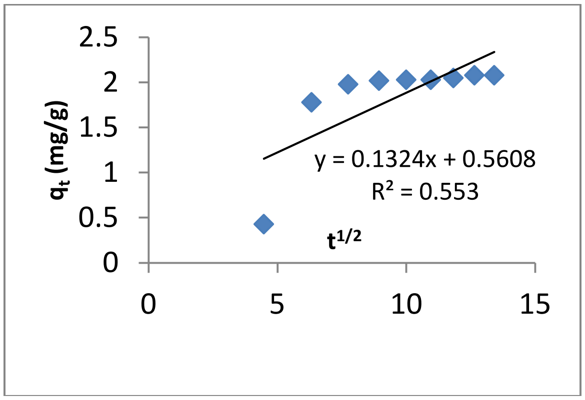

Fig 3: Intraparticle diffusion plot for the adsorption of crystal violet onto GS

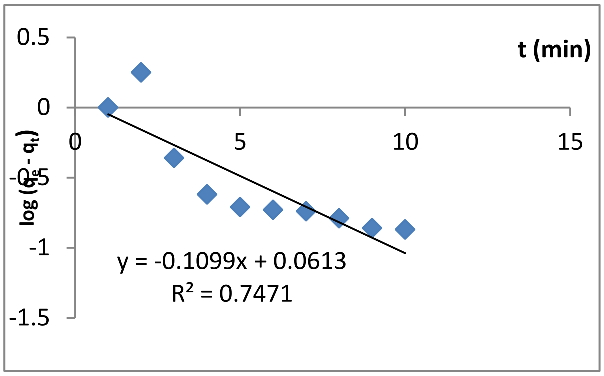

Fig 4: Pseudo-first order kinetic plot for the adsorption of crystal violet onto GS

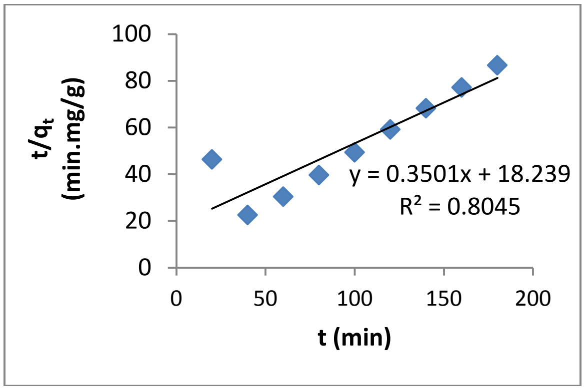

Fig 5: Pseudo-second order kinetic plot for the adsorption of crystal violet onto GS

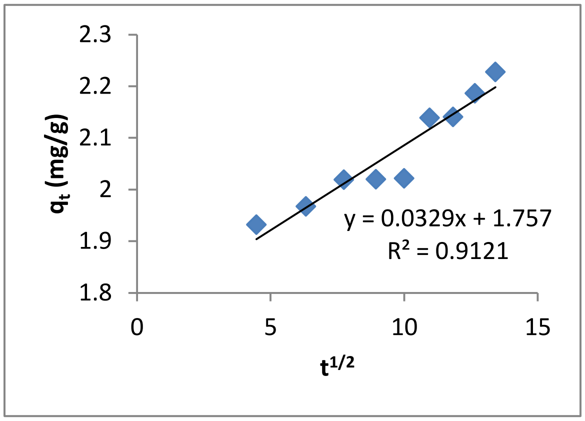

Fig 6: Intraparticle diffusion plot for the adsorption of crystal violet onto BP

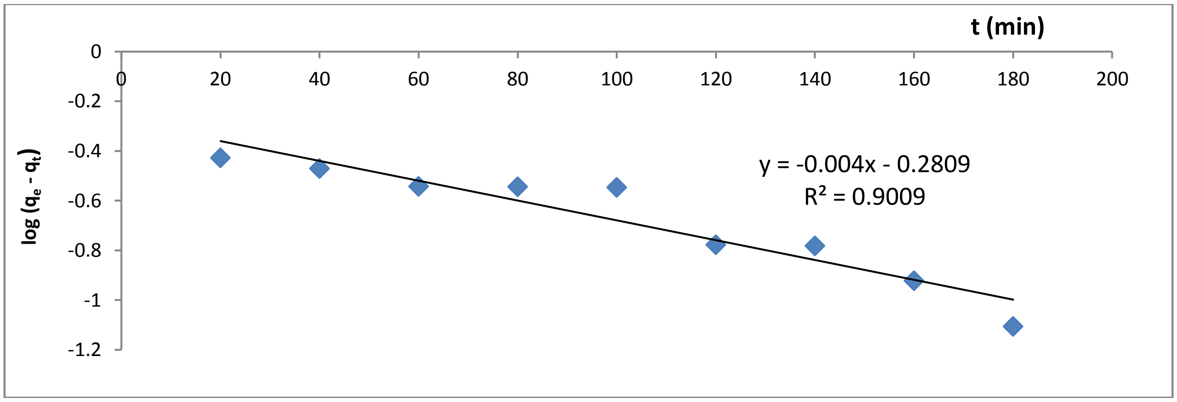

Fig 7: Pseudo-first order kinetic plot for the adsorption of crystal violet onto BP

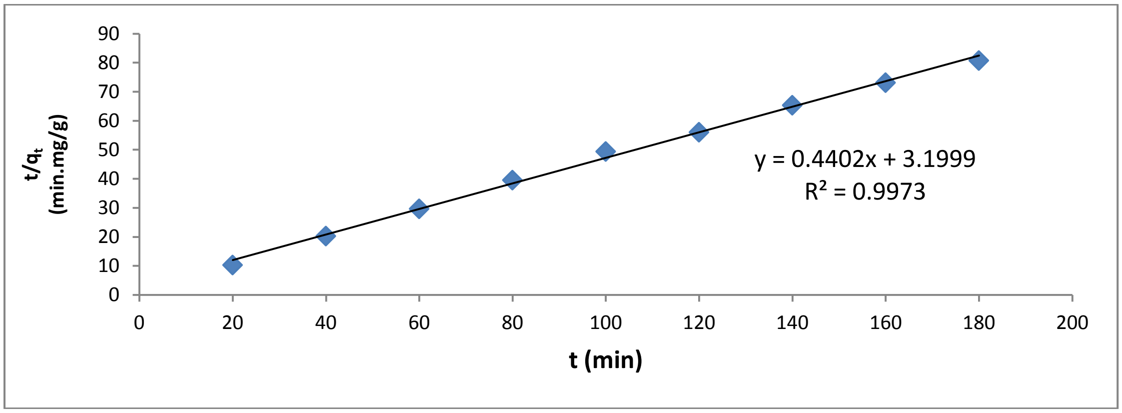

Fig 8: Pseudo-second order kinetic plot for the adsorption of crystal violet onto BP

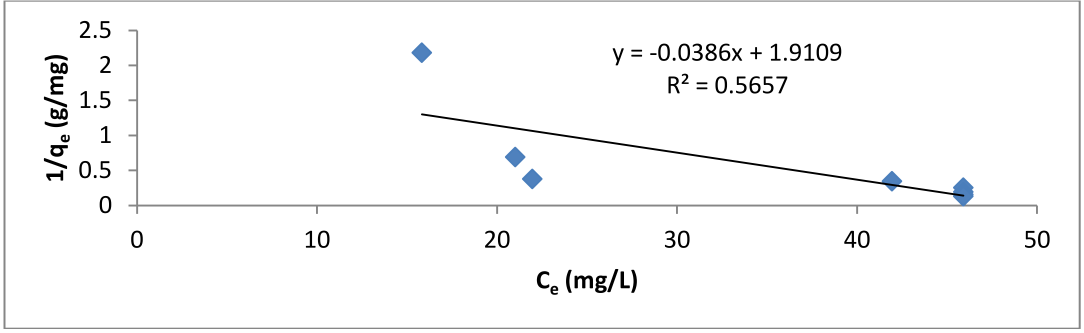

Fig 9: Langmuir isotherm for crystal violet adsorption on GS

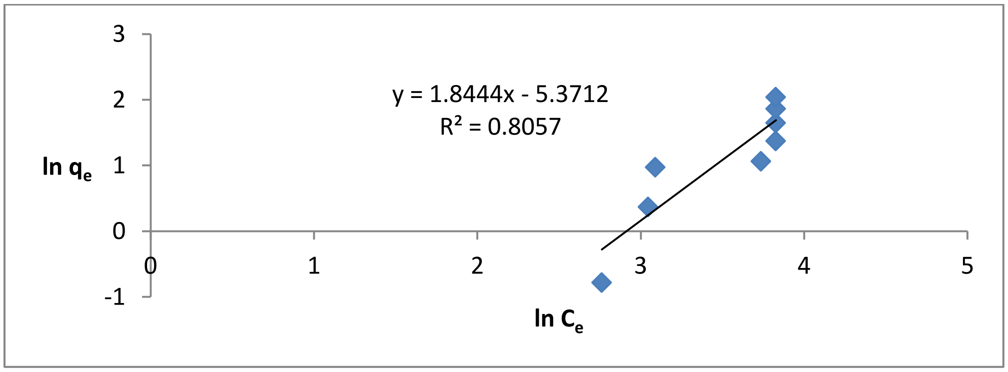

Fig 10: Freundlich isotherm for crystal violet adsorption on GS

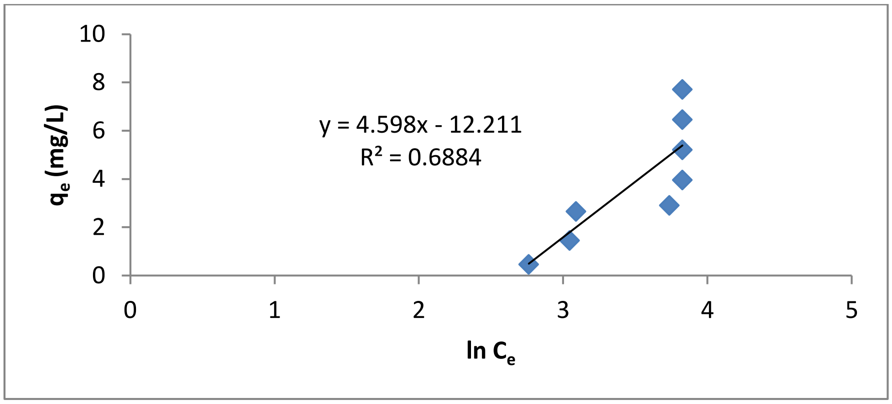

Fig 11: Temkin isotherm for crystal violet adsorption on GS

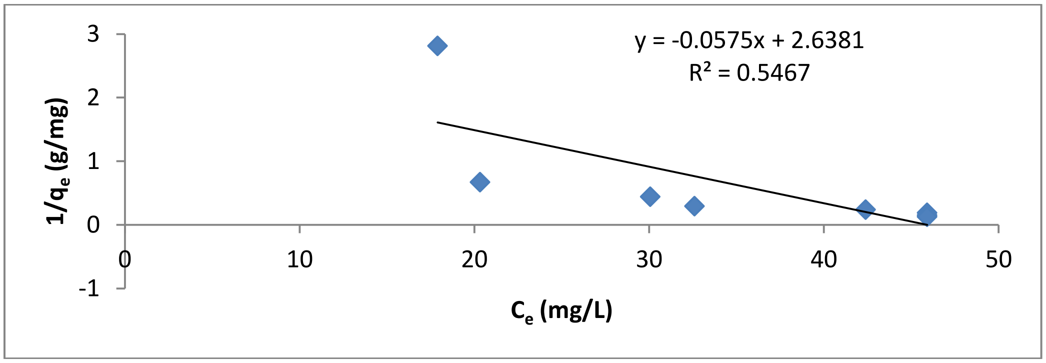

Fig 12: Langmuir isotherm for crystal violet adsorption on BP

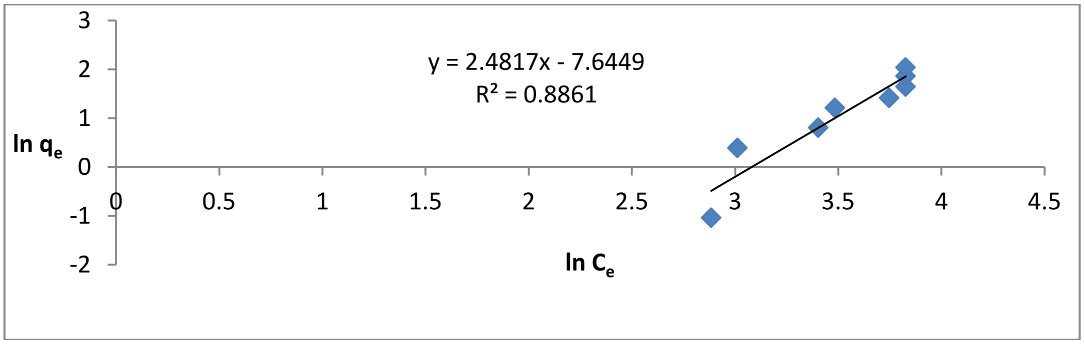

Fig 13: Freundlich isotherm for crystal violet adsorption on BP

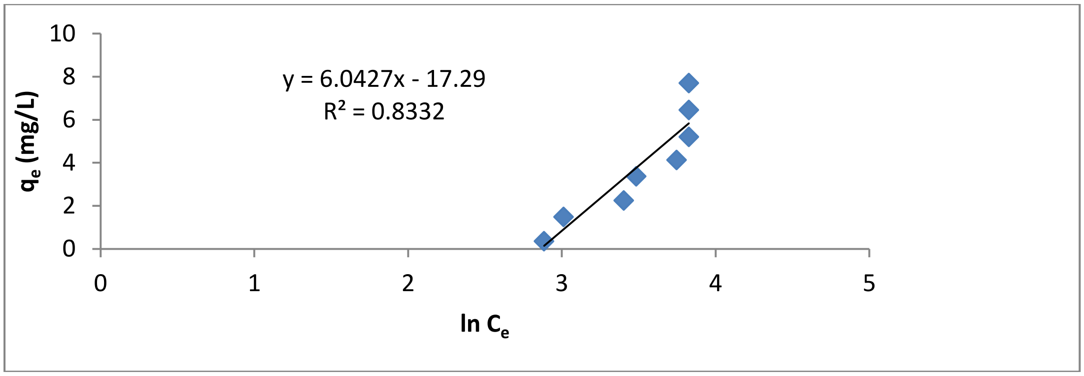

Fig 14: Temkin isotherm for crystal violet adsorption on BP

Table 1: Rate constants and coefficients of determination for the adsorption of crystal violet onto GS and BP

	Intraparticle diffusion model		**Pseudo-first order model**		**Pseudo-second order model**	
Adsorbent	k_{int}	R^2	k_1	R^2	k_2	R^2
GS	1.32×10^{-1}	0.553	2.51×10^{-1}	0.747	6.72×10^{-3}	0.804
BP	3.20×10^{-2}	0.912	9.21×10^{-3}	0.900	6.05×10^{-2}	0.997

Table 2: Equilibrium isotherm constants and coefficients of determination for the adsorption of crystal violet onto GS and BP

	Langmuir isotherm			**Freundlich isotherm**			**Temkin isotherm**		
Adsorbent	q_{max}	b	R^2	K_F	n	R^2	K_T	B_1	R^2
GS	0.524	-0.02	0.565	4.65×10^{-3}	0.542	0.805	7.03×10^{-2}	4.598	0.688
BP	0.380	-0.02	0.546	4.79×10^{-4}	0.403	0.886	5.72×10^{-2}	6.042	0.833

Conclusion: Understanding the performance characteristics of an adsorbent requires, among other things, the study of its sorption kinetics and equilibrium isotherms. In this study, the suitability of adsorbents derived from groundnut shell and bean pod for the removal of crystal violet in aqueous solution was evaluated in a series of batch experiments. The results of these experiments revealed that the adsorption of crystal violet by both groundnut shell and bean pod involves some forms of chemical interactions between the surfaces of the adsorbents and the adsorbate. The adsorption of crystal violet on the adsorbents was also found not to reach a plateau as the concentration of adsorbate in solution increases. This study demonstrates that adsorbents derived from groundnut shell and bean pod are suitable for removing crystal violet from contaminated wastewater.

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