Comparison of adsorption behaviour and kinetic modeling of bio-waste materials using basic dye as adsorbate

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Adsorption of basic dye Crystal Violet (CV) from aqueous solution by two low cost adsorbents Ground Nut shell powder (GNSP) and Ground nut shell ash (GNSA) has been investigated through batch mode adsorption process. The study involves the removal of CV using operational parameters; initial concentration (100-600 mg/L), different contact time, adsorbent dose (0.1-0.5 g/100mL) and pH (3-10). It has been found that percentage removal of dye improve with reduction in original concentration, intensification in time of contact and adsorbent dose. Basic pH of the dye solution show better adsorption capacity as compared to acidic dye solution. FTIR analysis reveals the presence of favourable alcoholic and carboxylic groups on the surface of the adsorbent. SEM analyses show irregular morphology with particles in micrometer range. Langmuir isotherm fit better than Freundlich adsorption isotherm and to Lagergren second order kinetic model fitted better than first order for both the adsorbents. Results show that GNSP performed better than GNSA. It can be concluded from the study that GNSP can be used as a low cost adsorbent.

Keywords: Crystal Violet, Ground nut shell powder, Ground nut shell ash, Adsorption isotherm, Kinetics.

Water is the primary necessity of life on earth. Water is required in almost all spheres of life. It has significant part in fulfilling a number of domestic, industrial and agricultural demands. Water resources are not only exploited but quality of the water has also been deteriorated due to startling increase in population, industrial activities and agricultural practices. Natural as well as waste water treatment systems are being polluted with a number of dyes which is being released from various sources like paper and pulp industries, textile industries, dye industries etc. Dyes are problematic as these are toxic and non-biodegradable because of their complex and stable structures, thus removal of dyes from waste water has become great challenge to all global scientists¹.

Numerous methods are reported in the literature for the elimination of dyes like chemical coagulation², ozonation^{3,4} and adsorption. Among the various practices for the removal of dyes, adsorption is preferred over other methods as it provides superior outcomes. For this purpose activated carbons are extensively used because of their efficiency and resourcefulness, however they arecostly. A number of studies have been conceded by different personnel on adsorption of coloured mixtures from solutions by the use of economical and eco-friendly adsorbents⁵⁻⁸. Several low cost and eco-friendly adsorbents for example activated carbon prepared from peanut shells⁹, *Phragmite australis* treated with 40% H₃PO₄ (Ref 10), Gmelina arborea bark¹¹, bottom ash¹², soil nano-clays¹³, sludge acquired from caustic industries¹⁴, tamarind (Tamarindus indica) fruit shell powder¹⁵, Tendu (Diospyros melanoxylon) leaves waste; a solid waste from bidi industry¹⁶, powdered mycelial biomass of Ceriporialacerate (CLB)¹⁷, Artocarpus heterophyllus (jackfruit) leaf powder (JLP)¹⁸, raw baggase¹⁹, grapefruit peel (GFP)²⁰, coniferous pinus bark powder (CPBP)²¹, activated carbons prepared from male flowers of coconut tree²², numerous fly ash samples having diverse unburnt carbon matters23, Syzygium cumini seed (SCSC)²⁴, Agricultural Waste Cocoa (*Theobroma cacao*) Shell²⁵, carbon prepared from waste prawn shells²⁶, chitin obtained from shrimp shell²⁷, mustard waste ash and buffalo dung ash²⁸ have been described in the literature.

This study describe the efficiency of two low cost bio adsorbent viz. ground nut shell powder (GNSP) and ground nut shell ash (GNSA) for the removal of crystal violet dye from aqueous solution.

Experimental Section

Preparation of Adsorbents

Ground Nut shells were gathered from native bazaar in Jalandhar, India. The precursor was

splashed with distilled water to get rid of the dirt and other contaminations, dried at 60°C for 12 h in hot air oven. Half of the dehydrated Ground nut shell was crushed and sieved (70 μ m) to obtain fine particles of Ground nut shell powder (GNSP). The rest half of dried Ground nut shells were turned to into ash (i.e. GNSA) by keeping it in a muffle furnace set at 500°C for 2 h.

Characterization of the adsorbent surface

Surface characteristics of the adsorbents were examined using scanning electron microscopic (SEM) analysis, energy dispersive X-ray spectroscopy (EDX) and Fourier transform infra-red (FT-IR) spectroscopy. In order to get the clear picture of particle size and external surface of material the images of adsorbent were taken by Scanning Electron Microscopy (SEM) (model: ZEISS EVO-40 series Scanning Electron Microscope (SEM): Carl Zeiss SMT Ltd., Cambridge, England). The images of adsorbents before as well as after the interaction with dye were recorded. For Energy Dispersive X-ray (EDX) detector system with X-FlashSilicon Drift Detector-4010, 10 mm², type 1106 Bruker AXS, Germany was used. FTIR spectra of GNSA and GNSP were taken using a Fourier transform infrared spectrophotometer (Shimadzu -8400s) to elucidate the functional group present on the surface of the adsorbents. For measuring IR spectra, the powdered samples were encapsulated in KBr pellet and a spectrum was obtained in the series of 400 to 4000 cm⁻¹.

Adsorption studies

Standard solution of Crystal Violet (CV) wasarranged by mixing 1 g of the dye in 1000 mL of the double distilled water (1000 mg/L). This solution was used to make desired concentrations using double distilled water. Hydrochloric acid and sodium hydroxide solution were used to adjust the pH of the dye solution.

Adsorption studies data was obtained using batch technique experiments. A pre-decided amount of the adsorbent was taken in 100 mL operational solution of various concentrations (100-600 mg/L) in 250 mL conical flask with stoppers. These flasks were retained in a thermostatic shaker sustained at a temperature of 25°C and 150±10 rotations per minute for sufficient time to accomplish equilibrium. Subsequently, the adsorbent material was alienated by centrifugation at 1800 ±100 rpm. Residual aqueous phase concentration of dye was examined with

UV-VIS spectrophotometer (Shimadzu-1800) by observing optical density at 591 nm.

The above mentioned process was repeated to discover the influence of different adsorption parameters like adsorbent load (0.05-0.35 g/1000mL), initial pH of the dye solution (3-10), time of contact and initial concentration of dye. Variation was done in one of the above mentioned parameters by keeping all other conditions at a constant value.

Adsorption Isotherms

Adsorption isotherm is a plot between amount of the dye adsorbed per gram of the adsorbent (Q_e) against concentration of the dye at equilibrium (C_e) . It is used to decide the practicability of using an adsorbent material for a specific purpose.

Quantity of dye adsorbed per gram of the adsorbent (Q_e) is computed according to following expression:

$$Q_{e} = \left(\frac{C_{o} - C_{e}}{m} \right) V$$

where C_o and C_e are original as well as concentrations of dyes at equilibrium respectively, m corresponds to the weight of adsorbent in grams and V specifies the volume in liters of the dye solution²⁹.

Most common isotherm for the adsorption is Langmuir isotherm. The assumptions of Langmuir model are:

- i. Adsorption is a reversible process.
- ii. The adsorbed molecules do not move on the surface of the adsorbent
- iii. The surface of the adsorbent has a precisequantity of positions where the adsorbatecan be adsorbed.
- iv. Only a monolayer is formed at the surface of the adsorbent.

Langmuir adsorption isotherm in linear form is represented as:

$$\frac{1}{Q_e} = \frac{1}{Q} + \frac{1}{bQC_e}$$

where Q and b are Langmuir constants indicates the Langmuir adsorption capacity (mg/g) and energy of adsorption (L/g) respectively. Q and b can be calculated from the slope and intercept of the graph between $1/C_e$ versus $1/Q_e^{30}$.

Langmuir adsorption is also articulated in relation of a separation factor R_L which is a dimensionless entity³⁰ and is expressed as:

$$R_{\rm L} = \frac{1}{1 + bC_o}$$

 C_o represents maximum initial concentration of dye in mg/L and b signifies Langmuir isotherm constant. If R_L is greater than 1, then process is uncomplimentary, if R_L is equal to 1 then process is linear, if R_L lies between 0 to 1 then process is encouraging and if R_L is 0 then the process is irreversible.

Freundlich adsorption represents a plot relating the equilibrium concentration of an adsorbate present on the surface of the adsorbent, to the equilibrium concentration of the adsorbate in the liquid phase²⁸. Linear form of the Freundlich Adsorption Isotherm may be represented as:

$$LogQ_e = LogK_F + \frac{1}{n}LogCe$$

where;

 K_F represents Freundlich adsorption capacity and 1/n indicates adsorption intensity. Value of 1/n is greater and 1 and high value of 1/n means adsorption process is promising.

Adsorption kinetics

Data was fitted to Lagergren pseudo-first order and pseudo-second order kinetic models to evaluate the influence time of contact.

The linearized form of Lagergren pseudo-first order model is expressed as

$$Log(Q_e - Q_t) = LogQ_e - \frac{k_{1,ads}}{2.303}t$$

and Lagergren pseudo-second order model in linear form is given by

$$\frac{1}{Q_t} = \frac{1}{h} + \frac{1}{Q_e}t$$

where,

 Q_e is the amount of dye adsorbed on the surface of adsorbent at equilibrium (mg/g), Q_t is the amount of dye adsorbed at time t (mg/g), $k_{1,ads}$ is the reaction rate constant for pseudo first-order in minutes, h is $k_2 Q_e^2$ and k_2 = reaction rate constant of adsorption the for pseudo-second-order in mg/g/min³¹.

Results and Discussion

Fourier Transform Infrared (FT-IR) spectroscopy

Figure 1(a) represents the FTIR spectrum of GNSP. The broad and intense bands located at 3339-3000 cm⁻

¹ may be attributed to O-H group. The other absorption at 2926 cm⁻¹ is associated to alkene stretching. Peaks at 1649 and 1030 cm⁻¹ corresponds to C=O and C-H bending.

It can be deduced from the FTIR spectra of GNSA shown in Fig. 1(b) that absorption peak at 3421.83 cm^{-1} corresponds to O-H stretching and absorption at 2924.8 cm⁻¹ is attributed to alkene stretching. Peak at 1664.32 cm⁻¹ is C=O, the peak at 1452.32 cm⁻¹ resembles to C=C and peak at

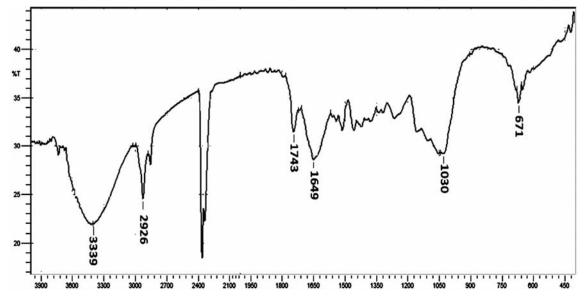


Fig. 1(a) -FTIR spectra of GNSP

1103.32 cm⁻¹ is C-O stretching vibrations. The peaks here, in this spectrum appeared to be less intense compare to the peaks of GNSP [Fig. 1(a)]. This could be due to the burning of GNSP into a GNSA.

It is clear from the above analysis that surface of the adsorbent contain groups like hydroxyl and carboxylic which are favourable for the adsorption of cationic dyes³².

Surface characterization using SEM images

The surface morphology of the GNSP consists of irregular fibers structure with small grains in between the fibers as shown in the Fig. 2(a). The length of the fibers is 158 μ m and width is 3.87 μ m. The size of pores on the surface is 3.87 μ m. The SEM image of GNSP after the reaction where all the pores and void

between the spaces have covered by the dye adsorbed on its surface was shown in the Fig. 2(b).

The surface of GNSA consists of long uniform fibrous structure with some channels and pores as shown in Fig. 2(c). The length of fibers is 197.5 μ m. The width of fibers is 7.8 μ m with a channel size of 4.87 μ m. The Fig. 2(d) shows the image of GNSA after the reaction in which all the space of channel and pores are covered by dye molecules.

Energy dispersive X-ray spectroscopy

Elemental analysis of adsorbent was conducted through using Energy dispersive X-ray spectroscopy(EDX) for the two adsorbents and shown in Figs 3(a) and 3(b) for GNSP and GNSA

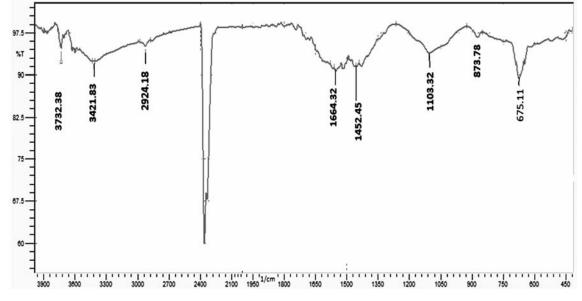


Fig. 1(b) - FTIR spectra of GNSA

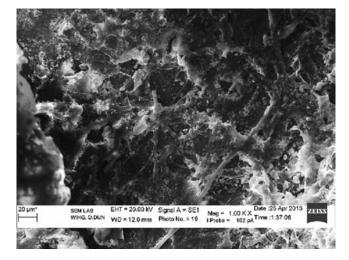


Fig. 2(a) -SEM image of GNSP before the reaction

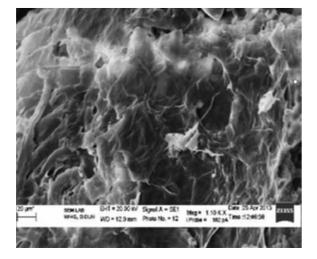


Fig. 2(b) -SEM image of GNSP after the reaction

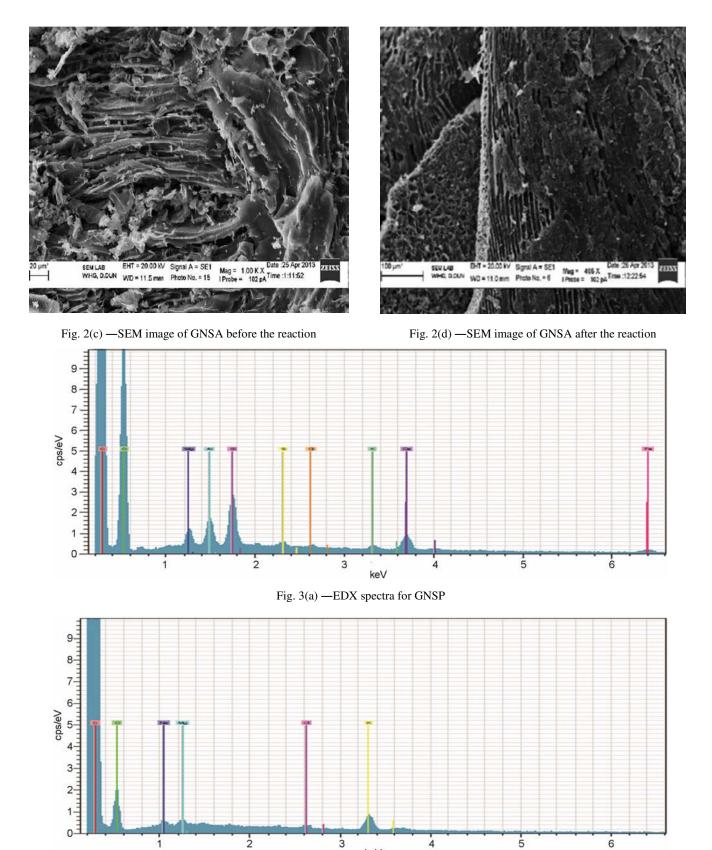


Fig. 3(b) -EDX spectra for GNSA

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respectively along with their spectrums. Both the adsorbents showed the high content of carbon and oxygen while the other elements such as magnesium, potassium, silicon, aluminum, calcium, iron, chlorine and sulphur are present in lower concentration. Presence of high content of oxygen indicates that adsorption is favorable for these adsorbents.

Effect of contact time

Effect of contact time on adsorption for the initial dye concentration 600 mg/L is depicted in Fig. 4. The figure indicated that the adsorption of the CV was rapid initially and there after it became slow while reaching equilibrium. Initially a number of adsorption sites were available for the adsorbate but at later stage these sites were reduced and repulsive forces may have started between the adsorbate molecules on the solid and bulk phases³³. GNSP and GNSA presented a limited number of adsorption sites. CV dye adsorption exhibited a fullness tendency at 120 min for GNSP and 150 min for GNSA. It is also clear that out of the two adsorbents GNSP has better performance than GNSA as in this experiment adsorbent dose for GNSP was 0.1 g/100 mL as compared to 0.5 g/100 mL of GNSA, even then it has shown better percentage removal. Moreover equilibrium time observed with GNSP is less than the GNSA which indicate that rate of adsorption for GNSP is greater than that of GNSA. This may be attributed to the higher amounts -OH and -CO groups existing at the surface of GNSP as indicated by the intensity of absorption peaks in the FTIR spectra.

Effect of adsorbent dose

Adsorbent to adsorbate ratio greatly influence the overall adsorption process and optimization of adsorption is usually done with regard to adsorbent

90 80 70 60 % removal 50 removal/GNSP 40 % removal(GNSA) 30 20 10 0 0 20 40 100 120 140 160 60 80 Time (min)

Fig. 4 — Effect of contact time on the adsorption process for CV onto GNSP and GNSA (Temperature= 25° C, RPM= 150 ± 1 , dose= 0.1 g/100mL of GNSP and 0.5 g/100mL of GNSA, pH=7.8, concentration of dye =600mg/L)

dose in a fixed volume of adsorbate solution. Influence of adsorbent dose for the removal of CV at initial CV concentration of 600 mg/L, by varying adsorbent dose from 0.05 to 0.35 g was investigated. Fig. 5 revealed that the percentage of dye removal improved with rise in adsorbent dose. Percentage dye removal increased from 36.21 to 84.99% for GNSP and from 24.91 to 66.91% for GNSA. This is because of the extra number of adsorption sites accessible with an increase in the adsorbent dose. On the other hand amount of dye adsorbed per gram of the adsorbent reduced from 509.94 to 145.69 mg/g for GNSP and from 298.91 to 92.09 mg/g for GNSA. This may be attributed to the fact that the adsorption sites are efficiently used at lower adsorbent dose. With an increase in the adsorbent dose, it could be possible that a substantial portion of accessible adsorption sites remains exposed, which lead to less per gram adsorption. Similar results have been stated for the removal of cationic dye on biowaste material³⁴.

Effect of initial pH of the solution

Solution pH of dye solution greatly influences charge at the surface of the adsorbents along with the extent of ionization of the solute particles. The H⁺ and OH ions are adsorbed powerfully and for this reason adsorption of additional ions are influenced by pH of adsorbate solution³⁵. The influence of pH on the Crystal violet adsorption onto GNSP and GNSA is represented in Fig. 6. The adsorption of CV has increased for both the adsorbents with rise in the pHof the dye solution from 3 to 10. As for the cationic dye, a net negative charge on the surface is expected to favour the adsorption which in turn can be maintained by basic pH. It can be observed from Fig. 6, that at pH value of 3 a clear positive charge because of presence of hydronium ion developed due to which

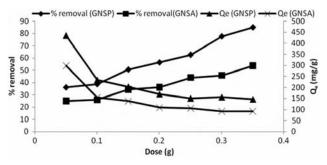


Fig. 5 — Effect of adsorbent dose on the adsorption process of CV onto GNSP and GNSA. (Temperature= 25° C, RPM= 150 ± 1 , concentration of dye =600 g/L, *p*H=7.8, equilibrium time= 120 min for GNSP and 150 min for GNSA)

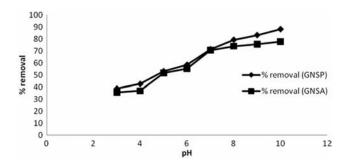


Fig. 6 — Effect of *p*H on the adsorption process of CV onto GNSP and GNSA (Temperature= 25°C, RPM= 150±1, dose= 0.1 g of GNSP and 0.5 g of GNSA, *p*H=7.8, concentration of dye = 600 mg/L, equilibrium time = 120 min GNSP and 150 min for GNSA)

minimum adsorption was observed at pH 3. Therefore as the pH was raised, a negative charge on the surface of the adsorbent was developed which increased with the further increase in pH. This enabled adsorbents for greater dye removal. Percent adsorption of dye was improved from 39-88% for GNSP and 36-78% for GNSA when pH was raised from 3-10.

Effect of initial dye concentration

The extent of CV adsorbed per unit mass of adsorbent improved with the rise in initial concentration and percent adsorption of CV from bulk phase decreased with the rise in original concentration (Fig. 7). The quantity of CV adsorbed per gram of the adsorbent (Qe) increased from 10.824 mg/g to 44.298 mg/g for GNSA and it increased from 70.178 gm/g to 276.546 mg/g for GNSP when the original concentration of dye was raised from 100 to 600 mg/L. Rise in the original concentration of the dye adds to the impelling cause to overcome the mass transfer opposition of dye molecules between the adsorbent and adsorbate bulk phase which increases the acceptance of dye molecules. Moreover, number of collisions between dye molecules and the adsorbent also increases which results in increase of the adsorption³⁶. However, the CV percentage removal decreased from 70.2 to 46.1% for GNSP and it decreased from 54.1 to 36.9 % for GNSA as the CV concentration was raised from 100 to 600 mg/L. This could be because of the reduction in number of active sites required for high initial concentration of dye. At first, molecules of dye quickly touch the periphery of adsorbent by mass transfer and thereon they gradually diffuse from periphery to the adsorbent due to the occupation of the active sites. This lead to the diffusion of dye into the porous arrangement of the adsorbent³⁷.

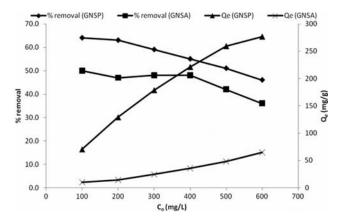


Fig. 7 — Effect of initial concentration of CV onto GNSP and GNSA (Temperature= 25°C, RPM= 150±1, dose= 0.1 g of GNSP and 0.5 g of GNSA, *p*H=7.8, equilibrium time= 120 min for GNSP and 150 min for GNSA)

Table 1 — Langmuir and Freundlich Constants for MB using
GNSP and GNSA

Langn	nuir Consta	ants	Freundlich Constants			
Parameter	GNSP	GNSA	Parameter	GNSP	GNSA	
Q (mg/g)	617.3	308.6	K _F	6.51	0.465	
b (L/mg)	0.003	0.0006	n	1.49	1.250	
R	0.994	0.989	R	0.979	0.968	
SD	0.580	0.005	SD	0.054	0.073	
R _L	0.341	0.728	-	-		

Adsorption Isotherms

Isotherms show an association among amounts of dye adsorbed per gram of the adsorbent sample (Q_e) and the concentration of dye at equilibrium (C_e) of aqueous phase. Adsorption isotherm for data obtained was drawn which is similar to that of type I adsorption isotherm which indicate that adsorbent pore sizes was approximately equal to the molecule size of adsorbate.

Experimental data for batch adsorption studies was applied to the Langmuir and Freundlich models. The results obtained from Langmuir and Freundlich isotherms indicated strong adsorption capacity of GNSP and GNSA for crystal violet. Adsorption constant for Langmuir (b and Q) and Freundlich isotherm (K_F and n) were computed from slope and intercept and which are presented in Table 1. Data was fitted well to Langmuir isotherm than the Freundlich isotherm as indicated by the 'R' values in Table 1. It is evident that GNSP performed better than GNSA. A special parameter ' R_L ' was also calculated and its value for GNSP was found to be 0.34 and 0.73 GNSA which is between 0 and 1, and directs that adsorption process was favourable.

Table 2 — Constants for Lagergren pseudo-first order and pseudo-second order adsorption kinetic model										
Adsorbent	Pseudo-first order kinetic model			Pseudo-second order kinetic model						
	$Q_e (mg/g)$	$k_{1,ads}$ (min ⁻¹)	\mathbf{R}^2	SD	Q _e (mg/g)	h (mg/g .min)	k ₂ (g/mg .min)	\mathbb{R}^2	SD	
GNSP	673.4	0.032	0.95	0.105	833	9.41	.0001	0.96	0.012	
GNSA	123.4	0.025	0.96	0.083	153.8	1.37	0.0006	0.93	0.082	

Values of Langmuir adsorption capacities (Q) were compared with the values reported in literature for Crystal violet using low cost materials²⁸ and it was found that GNSP and GNSA has comparable or better adsorption capacities than much of the low cost material already reported.

Adsorption kinetics

Adsorption data achieved after the variation in interaction time was fitted in the Lagergren pseudo-first order and pseudo-second order adsorption kinetic models. Correlation coefficients (\mathbb{R}^2) for the Lagergren pseudo-first order and pseudo-second order kinetic plots were found to be 0.952 and 0.994 respectively. From these linear plots values of Q_e and rate constants were calculated and are presented in Table 2. It can also be verified from Table 2 that correlation coefficients (\mathbb{R}^2) for pseudo-second order kinetic model are higher that the pseudo-first order kinetic which indicate that pseudo-second order kinetic model fitted better on the adsorption data for both the adsorbents.

Conclusions

This study highlights the potential of ground nut shell powder (GNSP) and ground nut shell ash (GNSP) for the removal of Crystal Violet dye from aqueous solutions by batch adsorption studies. It can be concluded that:

- 1. FTIR spectrum show that alcoholic, carboxylic groups are present on the surface of both the adsorbent which favour the adsorption process for cationic adsorbates.
- 2. GNSP and GNSA have different morphology. GNSP has irregular fibrous structure with small grain in between the fibers whereas GNSA consists of fibers having channels and pores in between. Particle sizes for both the adsorbents are falling in the micrometer range.
- 3. The percentage of dye removal increases with the rise in adsorbent dosage, and contact time. Adsoprtion also increases with rise in the *p*H of the CV dye solution.
- 4. The adsorption data is fitting well to Langmuir adsorption isotherm than Freundlich adsorption

isotherm and to Lagergren second order kinetic model than first order, for both the adsorbents.

- 5. GNSP outperforms GNSA in terms of adsorption capacity which may be due to the greater amount of negatively charged functional groups available on the surface.
- 6. The results indicate that both GNSP and GNSA have similar adsorption capacity pertaining to other low cost adsorbents reported in literature. Therefore these could be engaged as low cost substitute to commercially available adsorbents for the removal of basic dyes.

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