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Comparative study of adsorption capacity of matki seed husk for the removal of congo red, crystal violet,and methylene blue from aqueous solution

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ABSTRACT:

Many industries such as paper, food, cosmetics, textiles etc. use dyes in order to colour their products. The presence of Congo red, Crystal violet and Methylene blue dyes in water even at very low concentration is highly visible and undesirable. The degradation by-products of organic dyes have dangerous impacts on the environment. The ability of the low cost materials viz. matki seed husk (MtSH) powder to remove Congo Red (CR), Crystal Violet (CV), and Methylene Blue (MB) dyes in aqueous solution was studied by using batch adsorption process. Effect of contact time, adsorbent dosage (g/L), pH, salt and effect of temperature were studied in batch technique and results showed that amount of the dye adsorbed increases with increase in contact time and adsorbent dosage studied. The extent of adsorption was strongly depends on pH of solution, free energy of adsorption (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) changes were calculated. Equilibrium adsorption isotherms and kinetic were investigated. The experimental data were analyzed by the Langmuir and Freundlich models and the isotherm data fitted well to the Freundlich isotherm. The kinetic data obtained were analyzed using a pseudo-first order and pseudo-second-order equation. The experimental data fitted very well the pseudo second-order kinetic model.

Key words: Adsorption, Matki seed husk, Dyes, Adsorption isotherm, Equilibrium kinetics, etc.

INTRODUCTION:

Pollution caused by the textile wastewater is a common problem faced by many countries. The effluents from textile, leather, food processing, dyeing, cosmetics, paper, and dye manufacturing industries are important sources of dye pollution. Wastewaters from dyeing and finishing operations in the textile industry are generally high in both colour and organic content. Colour removal from textile effluents has been the target of great attention in the last few years, not only because of its potential toxicity, but mainly due to its visibility problems. Recent estimates indicate that approximately 12% of synthetic textile dyes used each year is lost during manufacture and processing operations and 20% of these lost dyes enter theenvironment through effluents that result from the treatment of industrial wastewaters. Many dyes and their break down products may be toxic for living organisms [1]. Dyes may be carcinogenic, mutagenic or teratogenic. Additionally it may also cause severe damage to human beings such as dysfunction of kidney, reproductive system, liver brain and central nervous system. The dyes are generally resistant to environmental conditions like light, effect of pH and microbial attack. The removal of dyes from industrial effluents in an economical way is a growing concern these days. There are various methods for the removal of dyes including sedimentation and flotation, membrane separation, coagulation, ion exchange and adsorption. The cost of operation is the main drawback of these techniques[2]. Among these methods, adsorption is a widely used for dye removal from wastewaters [3, 4]. Generally biological aerobic wastewater systems are not successful for decolourization of majority of dyes. Thus, the use of several low-cost adsorbents has been tested and used for the removal of dyes from polluted water bymany researches. They have studied the feasibility of using low cost materials, such as wasteblack gram seed husk [5]; banana bith [6]; cotton waste, rice husk [7]; green gram [8]; neem leaves [9], treated guava seeds [10], palm kernal coat [11],Gram Seed Husk [12]; duck weed [13], almond tree bark powder [14],cucumber peel[15], have been employed for the removal of CR,CV and MB dyes via adsorption. In the present study, the potential of matki seed husk(MtSH) powder low cost bio-sorbent for the removal hazardousof congo red, crystal violet and methylene blue from the aqueous solution is studied.

MATERIALS AND METHODS:

The mature and fresh matkicrop seeds were purchased from local market and washed thoroughly by using distilled water to clean them from dirt and impurities. After that, the seeds are soaked into distilled water up to 24 hours. Then their skin was removing from their pulses and washed with distilled water. It is dried in shadow. After drying the husk was ground by grinder to constant size of 60 µm fine powders of seed husk. The dried fine powder adsorbent was kept in an air tight glass bottle ready for further experiments.

Organic dyes (Congo Red, Crystal Violet and Methylene Blue) are purchased from different chemical laboratories such as methylene blue were purchased from Qualigens, Fine Chemicals, Mumbai (India), crystal violet and Congo red were purchased from Loba Chemicals Pvt. Ltd. Mumbai (India). All solutions were prepared in double distilled water. The concentration of dye solutions were determined by using UV-Visible single beam Spectrophotometer, (BioEra: Cal No.BI/CI/SP/SB-S-03). Stock solutions (500 ppm) were prepared by dissolving weighed quantities of these organic compounds (500 mg) in double distilled water (1000 ml). The experimental solutions were prepared by successive dilution using double distilled water. The solutions were carried out from the stock solution to prepare solutions in different concentrations. The concentration of solution was determined from calibration curve spectrophotometrically at their λmax that is Congo Red (λmax = 510 nm), Crystal Violet (λmax = 540 nm) and for Methylene Blue (λmax = 570 nm). The structures of dyes are shown in **Fig. 1.**

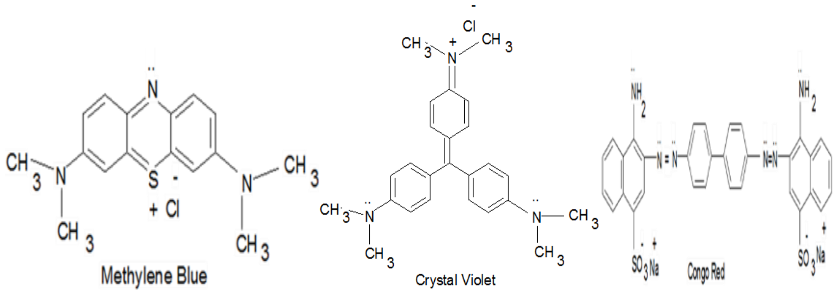

Fig:1. Chemical structures of Congo red,

Crystal Violet and Methylene blue dye.

Adsorption experiment was carried out by batch adsorption techniques at room temperature (306.2±3^0 k). The effect of pH on CR, CV and MB removal were studied by shaking 50 ml, 20 mg/L. of CR, CV and MB solution concentration with 0.5 g adsorbent dose in conical flasks. The effect of contact time and initial concentration were studied. After definite time intervals, a sample were withdrawn from the flask, the supernatant solution was analyzed for residual dye concentration. The optical density was analyzed using a UV-Visible single beam Spectrophotometer. The pH of the solution was adjusted by adding 0.1 M HCl or 0.1 M NaOH solution and measurement was done by digital pH-meter (Elico: LI 615). The amount of dye adsorbed per unite weight of husk adsorbent at time,'t **',** q_t(mg/L) and percentage dye adsorption capacity was calculated as

$$q_t = \frac{V(Co-Ct)}{M} \quad (1)$$

$$\%\ \text{adsorption efficiency} = \frac{(Co-Ct)}{C_0} \times 100 \quad (2)$$

Where,$C_0$is the initial concentration of dye (mg/L), C_t is the concentration of dye at any time t, 'V' is the volume of dye solution (mL) and M is the mass of matki seed husk (g).

RESULTS AND DISCUSSION:

Comparative study was conducted using matki seed husk powder surface as adsorbent for the adsorptiveremoval of Congo red, Crystal violet and Methylene blue dyes from its aqueous solution. Operationalconditions such as contact time, adsorbent dose, pH, and temperature wereoptimized for the dyes.

Effect of contact time**:** The time-dependent behavior of CR, CV and MB adsorption on matki seed husk (MtSH) powder were examined by varying the contact time between adsorbent and adsorbate in the range of 5 to 35 min. The results are shown in **Fig. 2**.

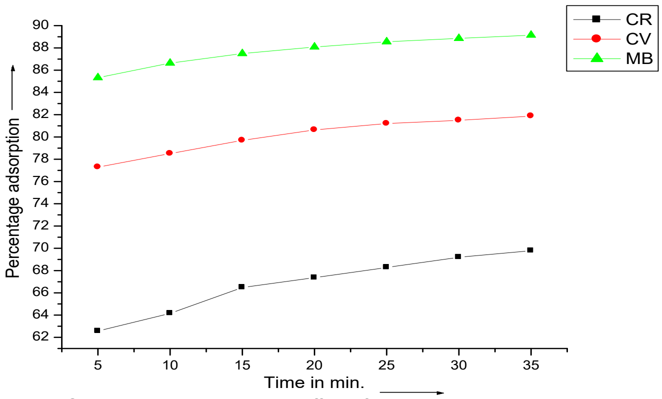

Fig.2: Comparative graph showing effect of contact time on percentage adsorption of CR,CV and MB on MSH.

The percentage adsorption of CR,CV and MB dyeswere increased with increasing contact time due to larger surface area available of the adsorbent. The maximum adsorption efficiency was 69.79 % for CR, 81.87 % for CV, and 89.14 % for MB at 35 min. The MB dye has higherpercentage adsorption capacity than CR and CV dyes due to the boundary layer resistance will be affected by the rate of adsorption and increase in contact time, which will reduce the resistance and increase the mobility of dye during adsorption [16].

Effect of adsorbent dose**:** The study of adsorbent dose for the removal of the CR, CV and MB dyes from aqueous solution was carried out using matki seed husk with their amount varying from 0.5 to 2.5 g.

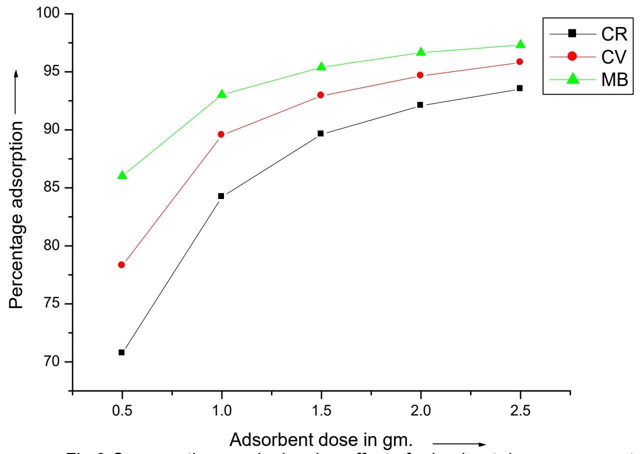

Fig.3:Comparative graph showing effect of adsorbent dose on percentage adsorption of CR,CV and MB on MSH.

The maximumadsorptions of dyes were 93.52 % of CR, 93.80 % of CV and 97.31 % of MB attained for adsorbent dose at 2.5 g. as shown in **Fig. 3.**The percentages adsorption of the dyes increases with increase in adsorbent dose due to increase in total number of exchange sites. The percentages adsorption capacity of MB dye has more than the percentages adsorption capacity CR and CV.

Effect of pH**:** Fig: 4 clearly showed the influence of the initial solution pH on the adsorption extent of CR, CV and MB on to matki seed husk powder.

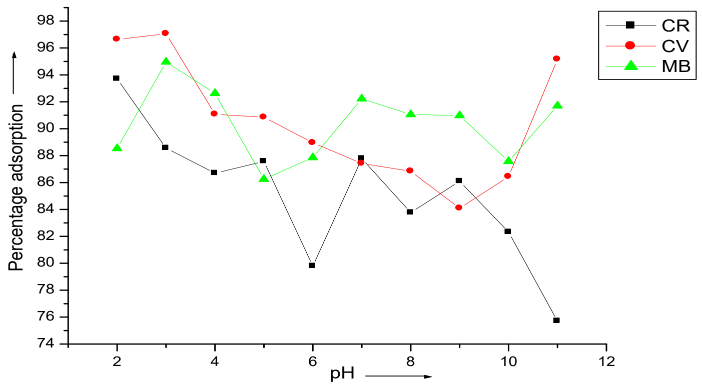

Fig.4:Comparativegraph showing effect of pH on percentage adsorption of CR,CV and MB on MSH.

The maximum adsorption of CR dye was93.70 % at pH 2, CV dye was 97.05 % and MB dye was 94.96 % at pH3 respectively. It can be seen from the figure that as the solution pH increases, the percentage adsorption capacity decreases. At higher pH the high negatively charged adsorbents surface sites did not favor the adsorption of deprotonateddyes due to electrostatic repulsion [17]. At higher pH, the percentage adsorption was found to decrease because the surface area of the adsorbent was more protonated and competitive adsorption occurred between H+ and free MB ions and their OH- towards the fixation sites. Therefore, H+ ions react with anionic functional groups on the surface of the adsorbent and results in restriction of the number of binding sites favorable for the adsorption of MB ions.

Effect of addition of salt: Experiments had been carried out using KCl of different concentration ranging from 0.5 to 2.5 g. Increase in salt concentration decrease the percentage adsorption capacity of CR. CV and MB dyes with increasing ionic strength, adsorption capacity decreased due to screening of the surface charges. The maximum adsorption capacity of CR. CV and MB dyes were 95.14 %, 96.45 % and97.53 % respectively. Thus it shows that the percentage adsorption capacity of MB dye has more than CR and CV dyes. The graphical representation is shown in **Fig: 5.**

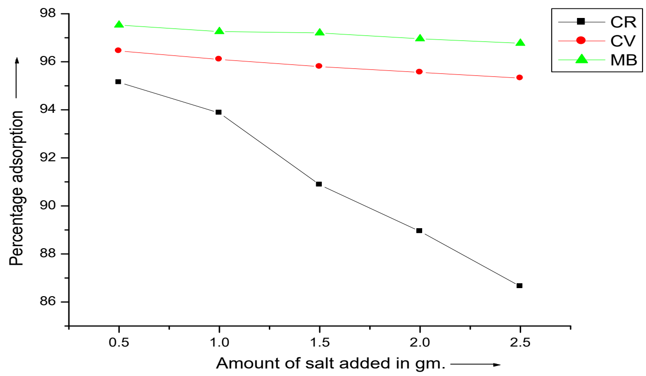

Fig.5: Comparative graph showing effect of salt added on percentage adsorption of CR,Cv and MB on MSH.

***Effect of Temperature*:** Temperature has a pronounced effect on the adsorption capacity of various adsorbents. The temperature effect was investigated for temperatures ranging 306.2 to 326.82 k. The results are shown in **Fig. 6**.

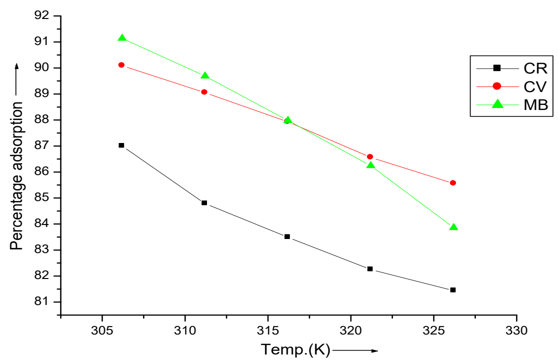

Fig.6: Comparative graph showing effect of temperature on percentage adsorption of CR,CV and MB on MSH.

The maximum adsorption efficiency of adsorbent was 87.00 %, 90.09 % and 91.14 % for CR, CV and MB respectively at 306.2 K. Since adsorption is an exothermic process, it would be expected that an increase in temperature of the adsorbate-adsorbent system would result in decreased adsorption capacity. Thus the adsorption of dyes were leading to a decrease in the residual forces on the surface of the adsorbent and hence causing a decrease in the surface energy of the adsorbent [18].

The Gibb's free energy (**ΔG°**), enthalpy (**ΔH°**), and entropy (**ΔS°**) changes for the adsorption were determined by using equation.

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ (3)$$

$$\log\left(\frac{q_e m}{Ce}\right) = \frac{\Delta S^\circ}{2.303R} + \frac{-\Delta H^\circ}{2.303RT} (4)$$

For the adsorbent concentration is unity (m = 1.0 g) equation (4) becomes

$$\log\left(\frac{q_e}{Ce}\right) = \frac{\Delta S^\circ}{2.303R} + \frac{-\Delta H^\circ}{2.303RT} (5)$$

$q_e$isthe amount of dye adsorbed per unite mass of husk (mg/g), Ceis the equilibrium concentration (mg/L) and T is the temperature in ^{o}k .$\frac{q_e}{Ce}$ is called adsorption affinity. The values of Gibb's free energy (**ΔG°**) has been calculated by knowing the enthalpy of adsorption (**ΔH°**) and entropy of adsorption (**ΔG°**) and (**ΔH°**) was obtained from a plot of $\log\left(\frac{q_e}{Ce}\right)$versus 1/T from equation (4) and (5). Once these two parameters were obtained, (**ΔG°**) is determined from equation (3). The calculated values are given in **Table:1**

Table: 1. Comparison of thermodynamic parameter values of CR, CV and MBdyes.

Thermodynamic parameters of CR, CV and MB Dyes								
(−ΔG°) KJ/mole			(−ΔH°) KJ/mole			(−ΔS°) J/mole.k		
CR	CV	MB	CR	CV	MB	CR	CV	MB
4.740	5.621	5.926	17.19	18.10	27.90	40.63	40.75	72.08
4.537	5.417	5.565						
4.334	5.214	5.205						
4.131	5.010	4.845						
3.928	4.806	4.484						

TheΔG^0 values obtained in present study for the CR, CV and MB dyes are < -10 KJ /mole, it indicates that physical adsorption was the predominant mechanism in the adsorption process. The Gibb's free energy indicates the degree of spontaneity of the adsorption process, where more negative value reflects a more energetically favorable adsorption process. The negative value of ΔG^0 indicates that the adsorption is favorable and spontaneous [19, 20]. The negative value of $\Delta S^0$and ΔH^0suggests that the decreased disorder and randomness at the solid solution interface with exothermic adsorption [21, 22].

ADSORPTION KINETIC MODELS:

Pseudo first order kinetic model assumed that the rate of solute up take with time was directly proportional to difference in saturation concentration and the adsorbed amount.

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (6)$$

Where, q_t and q_e are the amount of dye adsorbed (mg/g) at contact time t (min) and at equilibrium k_1 is the pseudo first order rate constant (min^{-1})

After integrating with the boundary conditions at $t = 0$, $q_t = 0$ and at $t = t$, $q_t = q_t$ and rearranging equation (6), the rate law for a pseudo first order reaction become.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303R} t \quad (7)$$

The plot of $\log(q_e - q_t)$ versus **t** gave a straight line with slope $-\frac{k_1}{2.303R}$ and intercept $\log q_e$ Adsorption rate were calculated from the slope and results are given in table (6)

Pseudo second order kinetic model was

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (8)$$

k_2 is equilibrium rate constant for pseudo second order adsorption (g/mg min).

After integrating with the boundary conditions at $t = 0$, $q_t = 0$ and at $t = t$, $q_t = q_t$ and rearranging equation (8), the rate law for a pseudo second order reaction become.

$$\frac{t}{q_t} = \frac{1}{k_{2}q_e^2} + \frac{t}{q_e} t \quad (9)$$

The plot of $\frac{t}{q_t}$ versus t gave a straight line with slope $\frac{1}{q_e}$ and intercepts $\frac{1}{k_{2}q_e^2}$ the calculated values of k_2, q_e values are given in **Table: 2.**

Table: 2. Comparison of the experiments and the kinetic model of CR, CV and MB dyes on MtSH adsorbent.

Dyes	**Pseudo-First order**			**Second order**		
	K_1 (min^{-1})	**q_e (mg/g)**	**R^2**	**K_2 (g/mg.min)**	**q_e (mg/g)**	**R^2**
CR	$16.523*10^{-3}$	173.476	0.980	$1.390*10^{-3}$	714.286	0.999
CV	$13.403*10^{-3}$	304.782	0.962	$0.965*10^{-3}$	2071.504	0.999
MB	$15.701*10^{-3}$	447.620	0.963	$0.624*10^{-3}$	4494.826	0.999

The R^2 values of CR, CV and MB with first order were 0.980, 0.962 and 0.963 respectively, while for second order R^2 values of CR, CV and MB are 0.999. It is clear that the adsorption of CR, CV and MB on MSH adsorbent was better represented by pseudo second order kinetics. This indicates that the adsorption system belongs to the second order kinetic model.

ADSORPTION EQUILIBRIUM:

To study the validity of Freundlich adsorption isothermthe following equation has been used

$$\text{Log } x/m = \log K_f + (1/n) \log C_e \quad (10)$$

$K_f$is the Freundlich constant [mg/g $(L/g)^{1/n}$] related to bonding energy, and n is the heterogeneity factor. The plot of Log x/m against log C_e gives straight line which exhibits monolayer coverage of the adsorbate on the other surface of the adsorbent. The value of n ranging in between 2-10 indicates good adsorption.

The equilibrium data was also analyzed in the light of Langmuiradsorption model.

$$\frac{1}{q_e} = \left(\frac{1}{Q_0}\right) + \frac{1}{bQ_0C_e} \quad (11)$$

Where, C_e (*mg/L*) is the equilibrium concentration of the adsorbate, q_e(*mg/g*) is the amount of adsorbate adsorbed per unit mass of adsorbent, at equilibrium, Q_0 (*mg/g*) and *b* (*L/mg*) are Langmuir constants related to maximum monolayer adsorption capacity and energy of adsorption respectively. The values of Q_0 and *b* are calculated from the slope and intercept of plot of $\frac{1}{q_e}$ against $\frac{1}{C_e}$ respectively. The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameterR_L. Which is a dimensionless constant referred to as separation factor or equilibrium parameter [23].

$$R_L = \frac{1}{1+bC_0} \quad (12)$$

Where, C_0 is initial concentration in ppm and *b* is Langmuir constant related to the energy of adsorption. R_LValue indicates the adsorption nature to be either unfavorable if $R_L > 1$, linear if $R_L = 1$, favorable if $0 < R_L < 1$ and irreversible if, $R_L = 0$ [24]. The calculated values are given in **Table: 3**

Table: 3. Comparisons of Langmuir and Freundlich isotherm constants for the adsorption of CR, CV and MB dyes on MtSH.

Dyes	Langmuir constants				Freundlich constants		
	Q_0 (mg/g)	b $*10^{-3}$ (L/g)	R_L	R^2	n	K_f $(mg/g(L/g))^{1/n}$	R^2
CR	214022.77	1.214	0.997	0.999	1.145	6.1290	0.998
CV	40153.55	11.118	0.643	0.999	1.159	10.2740	0.997
MB	99908.08	7.149	0.583	0.999	1.113	16.1395	0.999

The R_L value was found to be between 0 and 1 for CR, CV and MB studies, it is confirm that the ongoing adsorption of CR, CV and MB on MSH adsorbent is favorable. The data of MB reveal that the Langmuir model and Freundlich model yields better fit than CR and CV while the data of CR and CV indicates that the Langmuir model better fit than Freundlich isotherm model. The value of n is greater than unity, ($1< n <10$), that means favorable adsorption. [25]. From Table: 3 value of n was found to be between 1 and 10; this indicates that adsorption is favorable.

CONCLUSION:

Adsorption of dyes like Congo red, Crystal violet and Methylene blue were investigated using low cost bio-waste adsorbents, viz. matki seed husk. Conclusion from this study can be represented as follows

1. The percentage adsorption of CR, CV and MB dyes on MtSH increases with increasing dose of adsorbent, contact time and decreased with increasing temperature.
2. It was found that pH of dyes has a significant effect on the adsorption of dyes.
3. The negative value of ΔG^0 confirms that the feasibility of the reaction and spontaneous nature of the adsorption. Negative value of ΔS^0 andΔH^0 suggests that the decreased disorder and randomness at the solid solution interface with exothermic adsorption.
4. In case of MB both Langmuir and Freundlich isotherm model fitted well, while in case of CR and CV the Langmuir model was better fitted than Freundlich isotherm model for MtSH adsorbent.
5. The suitability of pseudo-first-order kinetic and pseudo-second-order kinetic models for the adsorption of CR, CV and MB dyes on MtSH were also discussed.

The kinetic modeling study has shown that the experimental data were found to follow the pseudo-second-order model suggesting a chemisorption process.

6. The values of the separation factor, R_L, indicated that the dyes were a favorable adsorption.

Since the raw material matki seed husk abundantly in large quantities, cheap, locally available, renewable adsorbent for the removal of colour of aqueous solutionof CR, CV and MB dye as well as textile dyeing effluent.

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