



RESEARCH ARTICLE

ADSORPTION KINETICS AND MECHANISM OF METHYL ORANGE DYE ON  
MODIFIED SILICA GEL FACTORY WASTE

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ABSTRACT

The silica gel waste (SGW), collected from solid waste of a local factory at Kolkata, India was modified with cationic surfactant and was utilized as an adsorbing media for the removal of methyl orange dye from aquatic environment. The characterization of base adsorbent and modified adsorbent was carried out. In batch mode, the kinetic study, isotherm study and effect temperature and shaking speed were carried out. The removal efficiency of surfactant modified silica gel waste (MSGW) in case synthetically prepared wastewater was also examined. The sorption was found to follow the pseudo second order reaction kinetics model and also Langmuir isotherm model. The chemisorption was the rate limiting step for the adsorption process.

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INTRODUCTION

Dyes are widely used as one of the key ingredients in many industries like textile, paint and varnishes, ink, plastics, pulp and paper, cosmetics, tannery etc., and also to the industries that produces dyes. These industries discharged the dye in the environment with their wastewater. The main environmental concern with dyes is their absorption and reflection of sunlight entering the water and thus causing reduction in photosynthesis and DO level in river (Adak *et al.*, 2005; Donia *et al.*, 2009). In addition, some dyes degrade into compounds that have toxic, mutagenic and carcinogenic effect on living organism. Azo dyes can be particularly toxic upon degradation and this class of dyes is widely used in many industries (Devi *et al.*, 2009). Among various azo dyes, methyl orange (MO) serves as a model compound for common water-soluble azo dyes, which are widely used in chemical, textile and paper industries and it is harmful to the environment (Jalil, *et al.*, 2010). Among various treatment methods available for dye removal, the adsorption has been proved to be the most suitable and promising technologies or has become the most popular technique because of its effectiveness, operational simplicity, low cost and low energy requirement (Jalil, *et al.*, 2010). Various adsorbents like calcined lapindo volcanic mud, modified bentonite, activated carbon derived from *Phragmites australis*, amine modified silica, modified ultrafine coal powder, zinc-aluminium-chloride layered double hydroxide, calcined layered double hydroxides, mesoporous fertilizer

plant waste carbon, metal hydroxide sludge, de-oiled soya and bottom ash etc. have been used (Jalil, *et al.*, 2010; Tabak *et al.*, 2010; Chen *et al.*, 2010; Donia *et al.*, 2009; Zhaunian *et al.*, 2009; Gaini *et al.*, 2008; Ni *et al.*, 2007; Mittal *et al.*, 2007; Mall *et al.*, 2006; Netpradit *et al.*, 2004). Recently, adsorption of dyes has been carried out using various surfactant modified metal oxides (Adak *et al.*, 2005; Tabak *et al.*, 2010; Namasivayam and Sureshkumar, 2006; Mohamed, 2004). The ionic surfactants forms monolayer or bilayer structures on the charged metal oxide surface depending on surfactant concentration (Somasundaran and Fuerstenau, 1966). The surfactant layers pose the ability to solubilize different organic molecules within its structure. Use of various clay minerals and few industrial waste materials have been studied to serve as metal oxides since these are cheap and abundant (Tabak *et al.*, 2010; Mall *et al.*, 2006; Namasivayam and Sureshkumar, 2006). However, application of the clay minerals, especially in continuous mode reactor was found to be difficult due to their very fine particle size and cohesiveness. Silica gel, a material that is well known by high thermal and chemical stability, good selectivity, swelling resistance, possibility of repeated use and inexpensive cost (Donia *et al.*, 2009). Again, the waste generated from a silica gel factory poses disposal problem. Any attempt to make use of this waste would be useful. Moreover, in a recent study carried out by the present researchers, it was observed that the waste generated from a local silica gel factory was effective in adsorptive removal of the cationic surfactant from wastewater (Koner *et al.*, 2011). With this in view, the present study aims at further investigation on the reaction kinetics and mechanisms for the

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removal of MO dye from water environment by the silica gel factory waste after its use as a sorbent for cetyltrimethylammonium bromide (CTAB), a quaternary ammonium based cationic surfactant (CS).

## MATERIALS AND METHODS

### Reagents

Orange II, cetyltrimethylammonium bromide (CTAB) (Hi-Media, India), methyl orange (MO) and chloroform (Merck, India) were used as received. All other chemicals used in this study were of high purity and used without further purification. All the chemicals were of analytical reagent grade.

### Instrumentation

A high precision electrical balance (CP 225D, Sartorius GMBH) was used for weighing. Digital pH meter (PB-11, Sartorius GMBH) was used for pH measurements. A spectrophotometer (Rayleigh, Beijing) was used for absorbance measurement. For SEM and EDX analysis, JEOLJSM5800 scanning electron microscope was used.

### Adsorbent

The silica gel waste was supplied by local factory located at Kolkata, India. After receipt of the material it was thoroughly washed with tap water followed by distilled water to remove the foreign substances and dried at 100° C. It was then sieved and about 70% of the total material lied in the particle size between 150 and 300 µm and that was used for the study. The final material after its washing and sieving was measured to be around 65% of the collected waste. The silica gel waste (SGW) was then treated with CTAB, a representative of CS to form micelle like structure on their surfaces and thus surfactant-modified silica gel waste (SMSGW) was prepared. This material was then for MO removal from water environment. The detail study on preparation of the SMSGW is described in later section.

### Analytical method

Spectrophotometric method was used for the determination of CS in the concentration range 0-12 mg/l (Few and Ottewill, 1956). Orange II ( $0.4 \times 10^{-3}$  M) chemically known as p-(β-naphthol-azo) benzene sulfonic acid as ion pairing agent with CS and chloroform as an extraction solvent were used. MO was measured in the concentration range of 0-12 mg/l by spectrophotometric method at a wavelength of 470 nm.

### Experimental studies

The batch experiments were carried out at the temperature 303±2 K using synthetic samples of MO prepared in distilled water (DW) and using simulated wastewater (WW) prepared in the laboratory as reported by Sapari (Sapari, 1996). The samples were shaken in a mechanical shaker at a shaking speed of 150 rpm. The pH of the solution was 7.1±0.1. The effect of adsorbent dose was studied at different temperature. The kinetic study was conducted taking three different initial

concentrations at 50, 100 and 200 mg/l and the shaking time was varied from 0 to 30 minutes. The effect of shaking speed was conducted for 100 mg/l MO concentration. Adsorption isotherm analysis was carried out at three different temperatures (288, 303 and 318 K) and the thermodynamic parameters were evaluated.

### Analysis of reaction kinetics data

In order to investigate the mechanism of solute adsorption onto the adsorbent, four kinetic models viz., first order reaction model (Benfield and Randall, 1980) based on the solution concentration, pseudo-first order equation of Lagergren (Lagergren, 1898) based on the solid capacity, second order reaction model based on the solution concentration (Benfield and Randall, 1980) and pseudo-second order reaction model of Ho and Mckay based on the solid phase sorption (Ho and Mckay, 1999) were analysed and a comparison of the best fit sorption mechanism was made. The linearized forms of different reaction models are shown below.

$$\text{First order: } \ln C_t = \ln C_o - k_1 t \quad (1)$$

$$\text{Pseudo first order: } \frac{dq_t}{dt} = k_{S1}(q_e - q_t) \quad (2)$$

$$\text{Second order: } \frac{1}{C_t} - \frac{1}{C_o} = k_2 t \quad (3)$$

$$\text{Pseudo second order: } \frac{t}{q_t} = \frac{1}{k_{S2} q_e^2} + \frac{1}{q_e} t \quad (4)$$

Where,

$C_t$  = solute concentration at any time  $t$

$C_o$  = solute concentration at time  $t=0$

$q_t$  = amount of solute adsorbed per unit weight of adsorbent at any time  $t$

$q_e$  = amount of solute adsorbed per unit weight of adsorbent at equilibrium

$k_1$  = first order reaction rate constant

$k_{S1}$  = pseudo-first order reaction rate constant

$k_2$  = second order reaction rate constant

$k_{S2}$  = pseudo-second order reaction rate constant

The experimental reaction kinetic data were analysed using the above four kinetic model.

## RESULTS AND DISCUSSION

### Adsorption of CTAB on SGW and preparation of SMSGW

Before preparation of SMSGW, it is important to find the maximum adsorption capacity and the initial CTAB concentration at which it would occur. The dye uptake capacity of SMSGW depends on the surfactant surface coverage on silica surface. More the surface coverage, more be the uptake capacity (Koner, *et al.*, 2010). Therefore, the adsorption isotherm was studied taking the initial CTAB concentration from 0 to 20000 mg/l. The adsorbent dose was kept fixed at 30 g/l. The contact time was taken as 30 minutes since this was the equilibrium contact time. The temperature and pH were 303±2 K and 6.9±0.1 respectively. From this isotherm study, the maximum adsorption capacity was found

to be 68.62 mg/g and it occurred when initial concentration of CTAB was 7500 mg/l. This condition was used for the preparation of SMSGW. Therefore, 180 g SGW (at a dose of 30 g/l) was shaken for one hour with 6 litres of CTAB solution having concentration of 7500 mg/l. After shaking, the supernatant was discarded and the SGW was washed thoroughly initially with tap water and finally with distilled water and then dried at 60°C for 24 hours. Thus the SGW was converted to SMSGW.

#### Characterisation of adsorbent

The characterization of the base adsorbent was carried out after proper washing and sieving of the collected waste. Table 1 summarizes the different properties of the base adsorbent (SGW) and modified adsorbent (SMSGW) such as size, bulk density, pH point of zero charge (pH<sub>pzc</sub>), BET surface area and pore volume. The surface morphology of the adsorbent was obtained from the Scanning Electron Microscopy (SEM) studies and the elemental composition from Energy Dispersive X-ray (EDX) analyses. The SEM photograph showed that the adsorbent particles are of ill defined shapes having irregular surfaces. It was also noted that after surfactant modification the surface of SGW became smooth (Fig. 1) and lowered the surface area. EDX analysis is a useful tool for identification of the kinds of elements contained in the solid specimen within short analysis period. The EDX analysis showed that the SGW contained 96% silica.

**Table 1. Characteristics of SGW and SMSGW**

Parameter	SGW	SMSGW
Particle size (µm)	150 - 300	150 - 300
Bulk density (g/cc)	0.615	0.624
pH <sub>pzc</sub>	2	10.2
BET surface area (m <sup>2</sup> /g)	264.6	194.4
Pore volume (ml/g)	0.66	0.38

**Table 2. Constants of Langmuir isotherm model for the MO removal by SMSGW**

Sample type	Temperature	Equation of linear fit line of Langmuir isotherm model	Maximum adsorption capacity, $q_m$ (mg/g)	Langmuir constant, $K_L$ (l/mg)	$R_L$	$R^2$
DW	288 K	$\frac{1}{q_e} = 0.0276 + 0.1439 \frac{1}{C}$	36.23	0.191	0.050	0.9678
DW	303 K	$\frac{1}{q_e} = 0.022 + 0.0947 \frac{1}{C}$	45.45	0.232	0.041	0.9846
DW	318 K	$\frac{1}{q_e} = 0.0212 + 0.0725 \frac{1}{C}$	47.17	0.292	0.033	0.9917
WW	303 K	$\frac{1}{q_e} = 0.0288 + 0.1882 \frac{1}{C}$	34.72	0.153	0.061	0.9815

#### Effect of adsorbent dose

The performance of SMSGW for removal of MO on varying dose was examined at three different temperature viz. 288 K, 303 K and 318 K using DW. The dose of SMSGW was varied from 0 to 10 g/l and the shaking time was 20 minutes. The higher removal efficiency obtained at higher temperature. It was noticed from Fig. 2, that optimum adsorbent dose was determined to be 5g/l at 288 K and 4 g/l at 303 K and 318 K. At the optimum condition, the removal efficiencies were 93% at 288 K and 303 K and 96 % at 318 K temperature. The batch experiments were also carried out to find out the effectiveness

of SMSGW in removing MO from synthetic wastewater samples at 303 K. The wastewater was prepared following the wastewater composition given by Sapari (Sapari 1996). Total dissolved solid (TDS) was 3030 mg/l and the turbidity was 120 NTU. The optimum dose was found to be 6 g/l which was come out to be greater than that of DW. The removal efficiency of in case of distilled water spiked sample was found to be greater than wastewater since the wastewater contained dissolved salts of sodium mainly chloride and sulphate that reduced the efficacy of SMSGW (Tabak *et al.*, 2010; Ni *et al.*, 2007; Netpradit *et al.*, 2004).

#### Kinetics study and rate limiting step

Kinetics study was conducted to find out the equilibrium contact time for MO uptake by SMSGW at different initial concentrations (50, 100 and 200 mg/l). The shaking time was varied from 0 to 30 minutes with a fixed SMSGW dose of 4 g/l. It was observed from Fig. 3, that the reaction attained equilibrium in 15 minutes for all the cases. However, a contact time of 20 minutes was considered to be sure that full equilibrium was attained. The equilibrium time obtained in this case was found to be the lowest so far observed than any other adsorbent used for removal of anionic dye (Gupta and Suhas, 2009). Such lower value of equilibrium contact period would be advantageous, since it would facilitate to reduce the reactor volume in case of continuous mode operation in the field. Reaction kinetics data for the removal of MO by SMSGW were analyzed using four reaction models as mentioned earlier. After analysing the above four models, it was observed that the pseudo-second order kinetics fitted best for all the cases. This indicated that the rate-controlling step for the MO removal by SMSGW was chemical adsorption or chemisorption (Ho and Mckay, 1999). In order to gain insight into the mechanism and rate controlling steps affecting the

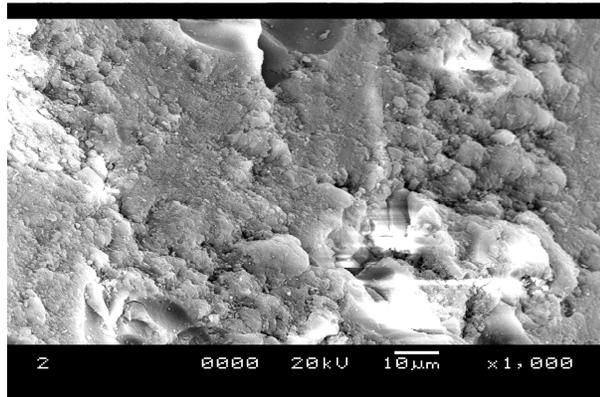
kinetics of adsorption, the kinetic experimental results were fitted to the Weber's intra-particle diffusion (Weber Jr. and Morris, 1963). The kinetic results were analyzed by the intra-particle diffusion model to elucidate the diffusion mechanism, which is expressed as

$$q_t = k_{id}t^{1/2} + C \quad (5)$$

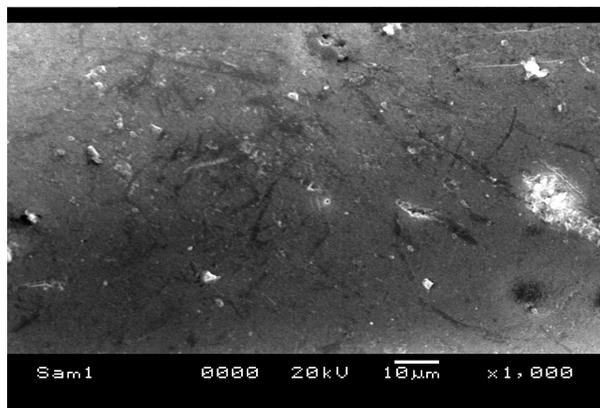
Where  $C$  is the intercept and  $k_{id}$  (mg/g min<sup>0.5</sup>) is the intra-particle diffusion rate constant. Weber and Morris (1963) reported that if film diffusion is involved in the process then a

**Table 3. Thermodynamic parameters of the MO adsorption process on SMSGW**

Temperature (K)	ln (K <sub>L</sub> )	ΔG <sup>0</sup> (kJ/mol)	ΔH <sup>0</sup> (kJ/mol)	ΔS <sup>0</sup> (kJ/mol/K)
288	11.05	-26.47		
303	11.24	-28.33	10.68	0.129
318	11.47	-30.34		

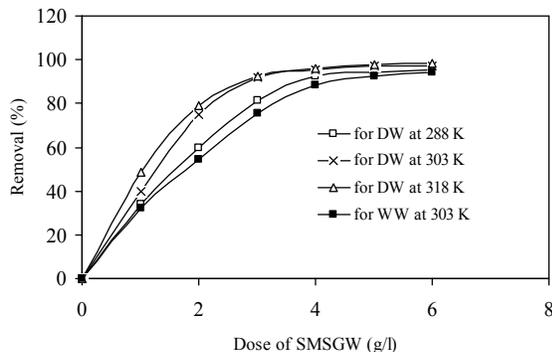


(a)



(b)

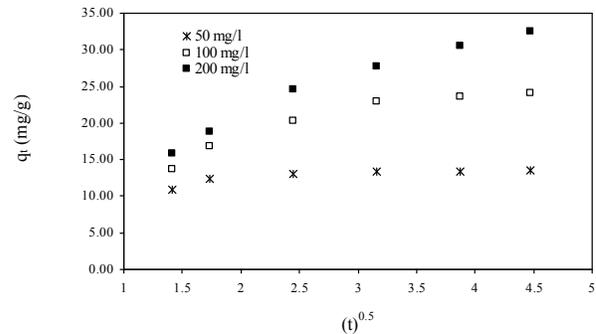
**Fig. 1. SEM images of (a) SGW and (b) SMSGW**



**Fig. 2 Effect of adsorbent dose for MO removal by SMSGW (Contact time 20 minutes, temperature 303 K)**

plot of adsorbate uptake vs. the square root of time would result in a linear relationship and the film diffusion would be

the rate limiting step if this line passes through the origin. As indicated in Fig. 4, the results could be represented by such a linear relationship but they did not pass through the origin. This indicated that film diffusion was involved in the process, but it was not the rate limiting mechanism. Moreover, the agitation speed in the range of 70-210 rpm had no effect on MO removal by SMSGW. This also indicated that the film diffusion was not rate limiting. Otherwise, increase/decrease in agitation speed would also increase/decrease the removal efficiency. Again the material was not porous, which indicated that pore diffusion was also not rate limiting step. Actually the removal of MO by SMSGW occurred due to the solubilization of MO molecules within the surfactant micelle formed on the silica surface.



**Fig. 4. Intraparticle diffusion plots for adsorption of MO on SMSGW for different initial MO concentrations**

*Adsorption isotherm and evaluation of thermodynamic parameters*

The adsorption isotherm defines the equilibrium state of the process. The isotherm study was conducted at three different temperatures (288, 303 and 318 K). The results were analysed by Langmuir isotherm model and Freundlich isotherm model and it was found that Langmuir isotherm fitted more accurately compared to Freundlich isotherm. Langmuir isotherm in linearized form is expressed as

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L} \frac{1}{C_e} \tag{6}$$

Where  $C_e$  is the concentration of dye solution at equilibrium (mg/l),  $q_e$  is the amount of MO adsorbed per unit of adsorbent at equilibrium (mg/g),  $q_m$  is the maximum amount of adsorption with complete monolayer coverage on the adsorbent surface (mg/g) and  $K_L$  is the Langmuir constant related to the energy of adsorption (l/mg). The linear plot of Langmuir isotherm for the present study is shown in Fig. 5. The values of the constants for Langmuir model were calculated and presented in Table 2. The adsorption capacity of SMSGW for the removal of MO at 303 and 318 K temperature were found to be 45.45 mg/g and 47.17 mg/g respectively. Temperature plays an effective role in the adsorption of MO on SMSGW. As the temperature of the solution increases, the extent of adsorption increases. In Indian subcontinent, the average temperature lies above the 303 K. So, it is expected to obtain higher capacity in the field application. The adsorption capacity of SMSGW for removal of MO from simulated wastewater is 34.72 mg/g at 303 K, which was found to be lower than the distilled water spiked

sample. The capacity so obtained for uptaking MO was found to be greater than other sorbents like modified and unmodified ultra fine coal powder (Zhaunian *et al.*, 2009), de-oiled soya and bottom ash (Mittal *et al.*, 2007), activated alumina (Iida *et al.*, 2004) and orange and banana peel (Annadurai *et al.*, 2002) etc. except few other sorbents like lapindo volcanic mud (Jalil, *et al.*, 2010), activated carbon derived from *Phragmites australis* (Chen *et al.* 2010;) and layered double hydroxides (LDH) (Gaini *et al.*, 2008; Ni *et al.*, 2007) etc. which have higher adsorption capacity. However, the application of volcanic mud in continuous flow type reactor in the field might be problematic due to its very fine particle size. Again, the preparation of LDH was found to be difficult.

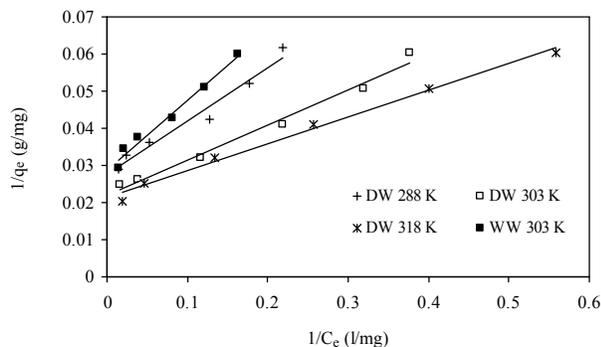


Fig. 5. Linear plot of Langmuir isotherm model for MO adsorption onto SMSGW

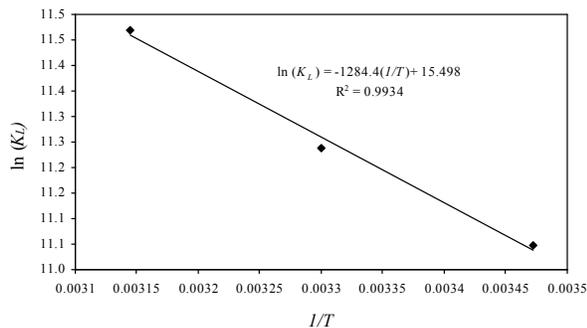


Fig. 6. Plot of  $\ln(K_L)$  vs.  $1/T$  for estimation of thermodynamics parameter for MO adsorption onto SMSGW

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless constant separation factor  $R_L$  (Hall *et al.*, 1966), which is given by

$$R_L = \frac{1}{1 + C_o K_L} \quad (7)$$

Where,  $C_o$  is the initial concentration of dye (mg/l) and  $K_L$  (l/mg) is Langmuir constant. The value of  $R_L$  indicated nature of adsorption; irreversible ( $R_L = 0$ ), favourable ( $0 < R_L < 1$ ) and unfavourable ( $R_L = 1$ ). The low value of  $R_L$  (Table 2) indicated the high and favourable adsorption (Al-Degs *et al.*, 2000). The thermodynamic parameters like change in standard free energy ( $\Delta G^0$ ), enthalpy ( $\Delta H^0$ ), and entropy ( $\Delta S^0$ ) of adsorption were calculated using the following equations:

$$\Delta G^0 = \Delta H^0 - \Delta S^0 T \quad (8)$$

$$\ln K_L = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (9)$$

Where,  $R$  is the universal gas constant (8.314 J/mol K),  $T$  is the absolute temperature (K) and  $K_L$  is the Langmuir constant. The plotting of  $\ln(K_L)$  against  $1/T$  (Fig. 6) gave the straight line with slope and intercept equal to  $-\Delta H^0/R$  and  $\Delta S^0/R$  respectively. The calculated values of  $\Delta H^0$  and  $\Delta S^0$  are reported in Table 3. The positive value of  $\Delta H^0$  indicated that adsorption process was endothermic. The decrease in  $\Delta G^0$  values with increasing temperature indicated in the feasibility of the adsorption at higher temperatures. The positive value of  $\Delta S^0$  suggested that there was increased randomness at the solid-solution interface during the adsorption. The negative value of  $\Delta G^0$  showed that the adsorption was spontaneous (Jalil, *et al.*, 2010).

## Conclusions

Surfactant-modified silica gel waste (SMSGW) could effectively be used for the removal of methyl orange, an anionic dye from dye bearing wastewater. The optimum adsorbent dose was determined to be 5g/l at 288 K and 4 g/l at 303 K and 318 K for 100 mg/l MO dye solution. At the optimum condition, the removal efficiencies were 93% at 288 K and 303 K and 96 % at 318 K temperature. The kinetics study showed that it followed pseudo second order kinetic model best. The Langmuir isotherm model was fitted better than Freundlich isotherm model. The maximum capacity obtained from Langmuir isotherm model could be compared with many other adsorbents especially which are generated from 'waste' product. The removal efficiency and adsorption capacity was found to be lower for wastewater in comparison to distilled water spiked samples. Temperature had significant effect on sorption process and the efficiency was found to be proportional with temperature. However, the shaking speed had no effect on sorption process. The chemisorption was only the rate limiting step for the adsorption process. The film diffusion was involved in the process, but it was not found to be the rate limiting step. Therefore, an industrial waste after its use for removal of surfactant could be reused for dye removal from water environment. Such effective use of a 'waste' made the process not only cost-effective but also environment-friendly.

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