



## Organobentonite as an Efficient and Reusable Adsorbent for Cationic Dyes Removal from Aqueous Solution

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In the present study, cetyltrimethylammonium bromide (CTAB) was used to modify raw bentonite (Ben) through the replacement of exchangeable cations to form CTAB modified bentonite (CTAB-Ben). Both Ben and CTAB-Ben were characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy and Brunauer-Emmet-Teller (BET) analysis. Adsorption potential of Ben and CTAB-Ben were explored for the removal of two cationic dyes i.e. Rhodamine B (RB) and Crystal violet (CV) from aqueous solutions. The maximum dye adsorption capacity of CTAB-Ben was found to be 93.15 and 14.76 mgg<sup>-1</sup> for CV and RB, respectively. The adsorption data of both the adsorbents was better explained by Freundlich isotherm whereas the pseudo second order (PSO) model better fitted the kinetics data. Regeneration studies revealed that CTAB-Ben could be reused upto five adsorption-desorption cycles.

**Keywords:** Adsorption, Clay, Crystal violet, Rhodamine B, Surfactant

### Introduction

In the present scenario, contamination of water resources with pollutants has evolved as a global concern. Among these pollutants, synthetic dyes contribute significantly in the contamination of water bodies.<sup>1</sup> These dyes are integral components of many industries like textile, paper, plastic, leather, pharmaceutical, cosmetic and many more.<sup>2</sup> During the coloring process total amount of dyes cannot be utilized and significant amount of these dyes remain unutilized and in most cases are released as such into the water bodies without any treatment.<sup>3</sup> After entering into the water bodies these dyes impart color to the water body which restricts sunlight to penetrate resulting in severe negative impacts to aquatic flora and fauna.<sup>4</sup> Moreover, many of these dyes are highly toxic, carcinogenic and resist degradation through biotic and abiotic components.<sup>5</sup> Therefore, it becomes essential to remove these dyes from the industrial effluents before releasing them into the water bodies. Both Crystal violet (CV) and Rhodamine B (RB) are known to cause skin allergy, digestive tract irritation and cancer in humans.<sup>6,7</sup> Major dye removal

techniques can be categorized into three groups: physical, chemical and biological. Among these techniques, physical methods have been found to be most feasible and economical.<sup>3</sup> Adsorption is a physical technique used for removal of contaminants from water and by far found to be the most promising in this aspect.<sup>1</sup> Most of the adsorbents cannot be utilized due to the problems like high cost (e.g. activated carbon), low adsorption and availability issues. Therefore, researchers have focused their studies to find out cheaper and efficient alternatives for this purpose. In this regard, naturally available clays being abundantly available, cheap and eco-friendly are being widely utilized for dye removal from aqueous solution.<sup>1</sup> The major lacuna for their large scale use is their low efficiency. Hence, efforts have been made to enhance their adsorption efficiency through structural modification of clay surface. At present, researchers are showing great interest for utilizing organoclays for organic pollutants removal from contaminated wastewater. Although before utilization of organoclays for such purpose, it is essential to determine its interaction with such contaminants such as adsorption-desorption pattern. In the present study, commercially available bentonite was modified with a cationic surfactant, CTAB

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modified bentonite (CTAB). The aim behind this modification in bentonite was to evaluate its effectiveness for removal of CV and RB from aqueous solutions.

## Experimental Section

### Materials

Commercially available bentonite and CTAB were procured from CDH Ltd. (India) and SD Fine Chemicals Ltd., Mumbai, respectively. CV and RB were purchased from Sigma-Aldrich, India. Double distilled water was used during the entire study. The solution pH was adjusted using dilute HCl and NaOH solution.

### Preparation of CTAB Modified Bentonite

Cation exchange capacity (CEC) was determined by a method performed by other researchers.<sup>8</sup> Raw bentonite and 0.1 M NaCl solution were mixed in the ratio (1:10) and the contents were stirred at 60°C for 6 h to replace all the exchangeable cations with the sodium cations. The resultant colloidal solution was then centrifuged, washed repeatedly with distilled water to remove excess of chloride ions and finally dried in an oven at 70°C to get Na-bentonite. CTAB modified bentonite (CTAB-Ben) was prepared through the cation exchange of sodium ions with alkylammonium ions of CTAB. Na-bentonite was mixed with CTAB (twice the CEC of bentonite) and the contents were stirred for 6 h at 60°C. This colloidal solution was then centrifuged, washed repeatedly with distilled water and dried at 70°C for 24 h to obtain CTAB-Ben.

### Characterization

Shimadzu UV-1800 spectrophotometer was used to quantify the amount of dye present in the solutions. SEM analysis was done through Zeiss<sup>®</sup> EVO 18 to study the surface morphology. FT-IR spectra of the modified and unmodified bentonite were analyzed by Nicolet™ iS50 FT-IR Spectrometer. The XRD pattern was studied by Panalytical X'Pert Pro. The surface area was determined using Brunauer-Emmet-Teller (BET) method by Micromeritics 3 Flex 3500.

### Adsorption and Kinetic Studies

Adsorption experiments for CV were carried out in batch mode by taking 100 mg of adsorbent in conical flasks with 50 mL of aqueous solutions of different initial CV concentrations (25–250 mgL<sup>-1</sup>) at pH 7. The conical flasks were shaken for 240 min on a rotary orbital shaker (200 rpm) at room temperature.

Batch studies for RB were conducted in a similar manner with 300 mg adsorbent and different initial RB concentrations (20–120 mgL<sup>-1</sup>). The amount of dye remained in the solution after adsorption was calculated using the following equation:

$$q_e = \frac{(C_0 - C_e)V}{M} \quad \dots (1)$$

where,  $q_e$  is the amount of adsorbate at time  $t$  (mgg<sup>-1</sup>);  $V$  is the volume of dye solution (L),  $C_0$  and  $C_e$  are the initial and the equilibrium dye concentration respectively (mgL<sup>-1</sup>) and  $M$  is adsorbent amount (g).

Kinetic studies were also carried out in batch mode for each dye using the same method described above. The samples were taken out at pre-defined time intervals and the amount of dye remained in the solution at any time  $t$ ,  $q_t$  (mgg<sup>-1</sup>) was calculated using the following equation:

$$q_t = \frac{(C_0 - C_t)V}{M} \quad \dots (2)$$

where,  $C_t$  is the concentration of dye at time  $t$  (mgL<sup>-1</sup>)

### Regeneration Studies

Desorption of both adsorbents was achieved through 0.1 M NaOH. After conducting the adsorption experiment, the clays saturated with dye particles were added to 50 mL of 0.1 M NaOH. The contents were then shaken for 3 h to reach equilibrium. The clays were then recovered from the suspension by centrifugation and washed with distilled water for further use. The dye removal (%) was determined by the following equation:

$$\text{Dye removal (\%)} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad \dots (3)$$

where,  $c_0$  = initial dye concentration and  $c_e$  = equilibrium dye concentration

## Results and Discussion

### Characterization of Adsorbents

The N<sub>2</sub> adsorption-desorption isotherm of raw and modified bentonite revealed type IV adsorption-desorption isotherm. BET surface area of bentonite before and after modification is shown in Table 1. The obtained surface area of CTAB-Ben (41.865 m<sup>2</sup>g<sup>-1</sup>) was found to be significantly higher than the reported surface area (0.25 m<sup>2</sup>g<sup>-1</sup>) by other researchers.<sup>9</sup> BET surface area analysis indicated slight increase in surface area of CTAB-Ben (41.865 m<sup>2</sup>g<sup>-1</sup>) in comparison to Ben (35.068 m<sup>2</sup>g<sup>-1</sup>). This could be due

Table 1 — Physicochemical characteristics of prepared adsorbents

Adsorbent	BET Surface Area (m <sup>2</sup> g <sup>-1</sup> )	Total pore volume (cm <sup>3</sup> g <sup>-1</sup> )	BJH Adsorption cumulative volume of pores between 10.000 Å and 3,000.000 Å width (cm <sup>3</sup> g <sup>-1</sup> )	Adsorption average pore diameter (4V/A by BET) (Å)
Ben	35.068	0.135 <sup>a</sup>	0.133	154.813
CTAB–Ben	41.865	0.141 <sup>b</sup>	0.137	135.484

<sup>a</sup>total pore volume of pores less than 2,094.397 Å width at P/Po = 0.990  
<sup>b</sup>total pore volume of pores less than 3,126.489 Å width at P/Po = 0.993

to the presence of more adsorption sites in comparison to bentonite. However, the obtained values are low as compared to the literature surface area value (600–800 m<sup>2</sup>g<sup>-1</sup>) of bentonite. The reason could be the less exfoliation of the layers of bentonite.<sup>10,11</sup> The obtained pore width and pore volume pointed towards the mesoporous pore structure of CTAB–Ben. FT-IR spectra of prepared adsorbents are shown in Fig. 1A. Spectroscopic analysis of Ben and CTAB–Ben gave an indication about the surface groups which in particular are responsible for the surface active sites for adsorption. The broad absorption bands in both adsorbents at 3620 cm<sup>-1</sup> were due to O–H stretching vibrations of the Si–OH (silanol) and Al–OH groups of Ben and CTAB–Ben.<sup>12</sup> Two bands at 2850 cm<sup>-1</sup> and 2920 cm<sup>-1</sup> in CTAB–Ben were due to the asymmetric and symmetric stretching vibrations of the –CH<sub>3</sub> and –CH<sub>2</sub> of the aliphatic chain of the CTAB.<sup>13</sup> The presence of these bands in CTAB–Ben thus confirmed the insertion of aliphatic chain in between the layers of raw bentonite. The absorption band at 700 cm<sup>-1</sup> was due to the –OH bending vibrations indicating octahedral sites occupied by Al<sup>3+</sup> ions. The Si–O–Al and Si–O–Si bending vibrations appeared at 532 and 466 cm<sup>-1</sup>, respectively.<sup>14</sup> XRD patterns (Fig. 1B) revealed that the basal spacing in Ben was 1.40 nm, corresponding to d<sub>001</sub> plane at 2θ = 6.27 whereas, in CTAB–Ben the d<sub>001</sub> peak shifted to 2θ = 6.03 and the corresponding interlayer spacing of CTAB–Ben increased to 1.46 nm. This indicated that alkylammonium ions intercalated between the layers of raw bentonite resulting in enhanced basal spacing.<sup>15</sup> The SEM images revealed significant changes on the surface of CTAB–Ben as compared to Ben due to the modification with CTAB. As shown in Fig. 1C, the raw bentonite exhibited nonporous surface whereas CTAB–Ben (Fig. 1D), showed much coarser porous surface with some fibers reaching out by introduction of CTAB, which further confirmed that the alkylammonium ions intercalated in between the layers of bentonite.

## Adsorption Studies

### Effect of pH and Adsorbent Dose

The dye solutions of pH varying from 3 to 11 were prepared using 0.1 N HCl and 0.1 N NaOH solutions. The initial dye concentration of CV and RB were kept at 200 and 100 mgL<sup>-1</sup> respectively. In both Ben and CTAB–Ben, the dye removal efficiency increased with increase in pH till 9 and thereafter became constant. Thus, pH 9 was selected to conduct adsorption studies. With increase in pH, positive charge on surface decreased and negatively charged sites increased resulting in increase in uptake of cationic dyes.<sup>12</sup> The adsorption of CV and RB increased sharply with increase in dose of bentonite. Comparable results were observed for CTAB–Ben but the saturation level reached at a lesser dose and the removal efficiency was also higher than Ben. Also, for both adsorbents, the percent adsorption was much higher for CV than RB at all the doses. The optimum dosage of adsorbents were selected as 100 and 300 mg for CV and RB respectively.

### Effect of Initial Dye Concentration and Contact Time

The initial dye concentrations of CV and RB were taken in the range of 25–250 mg L<sup>-1</sup> and 20–120 mg L<sup>-1</sup> respectively. For both Ben and CTAB–Ben, it was observed that on increasing the initial dye concentration, adsorption capacity also increased. At lower dye concentrations, equilibrium was achieved at a faster rate. For CV, the equilibrium time was 60 min for both Ben and CTAB–Ben whereas for RB, the equilibrium time was 180 minutes for both adsorbents. At higher dye concentrations, more competition occurred among dye molecules for active sites on adsorbent, leading to higher adsorption capacity at a specific dose.<sup>4</sup> Though Ben and CTAB–Ben reached equilibrium at different time intervals but for comparison purpose, entire experiment was conducted for 240 minutes.

### Adsorption Isotherms

Adsorption data of both adsorbents for each dye were fitted with non-linear Langmuir, Freundlich

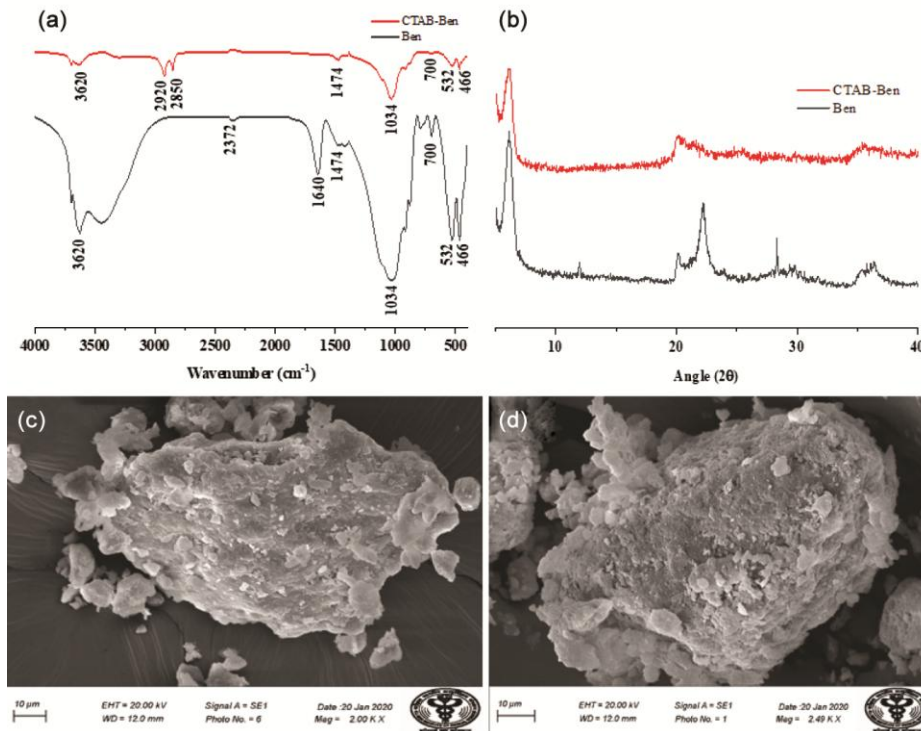


Fig. 1 — (A) IR spectra (B) XRD pattern (C) SEM image of Ben (D) SEM image of CTAB-Ben

and Temkin adsorption isotherm models<sup>16</sup> (Table 2). The adsorption data obtained experimentally were fitted with the non-linear adsorption isotherms and corresponding constants determined for both adsorbents for each dye are shown in Table 3 and corresponding curves are illustrated in Fig. 2. The partition coefficient ( $K_d$ ) values for CV and RB adsorption on Ben and CTAB-Ben were Ben @ CV (3.92), Ben @ RB (0.55), CTAB-Ben @ CV (6.80), CTAB-Ben @ RB (1.29). The results of adsorption studies clearly depicted that CTAB-Ben is more efficient in removing cationic dyes as compared to Ben. For both the adsorbents, Freundlich isotherm model fitted best to the obtained adsorption data and the predicted adsorption constants for both dyes (CV and RB) were in close approximation with observed  $K_d$ . The fitting of Freundlich model in the adsorption data indicated the occurrence of adsorption of dyes onto the heterogeneous surface of both adsorbents.<sup>17</sup> In case of both the adsorbents, the Freundlich adsorption constant ( $1/n_F$ ) values for CV and RB were found to be less than 1 displaying L-type adsorption. This suggested the occurrence of a physical adsorption involving weak van der Waals forces.<sup>18</sup>

#### Adsorption Kinetics

In order to understand the kinetics behind the CV and RB removal process, non-linear Pseudo first order

Table 2 — Mathematical expressions for non-linear isotherm and kinetic models

Isotherm <sup>16</sup>	Equation
Langmuir	$q_e = \frac{q_m K_L c_e}{1 + K_L c_e}$
Freundlich	$q_e = K_F c_e^{1/n_F}$
Temkin	$q_e = \frac{RT}{b_{Tem}} \ln A_{Tem} C_e$
Kinetic model <sup>19</sup>	Equation
PFO	$q_t = q_e(1 - \exp(-k_1 t))$
PSO	$q_t = \frac{q_e^2 k_2 t}{1 + q_e k_2 t}$

(PFO) and Pseudo second order (PSO) models were employed to correlate the kinetics data<sup>19</sup> (Table 2). The plots of adsorption kinetics using PFO and PSO are shown in Fig. 3 and the corresponding parameters are given in Table 4. The results of adsorption kinetics of CV and RB adsorption on Ben and CTAB-Ben exhibited the enhancement in dye adsorption upon increasing the contact time although adsorption rates varied. On the basis of kinetic studies, the equilibrium time intervals for adsorption of CV and RB were 60 min and 180 min respectively.

Adsorbent	Dye	Langmuir		Freundlich			Temkin				
		$q_{m,cal}$	$q_{m,exp}$	$K_L$	$r^2_{adj}$	$K_F$	$n_F$	$r^2_{adj}$	$b_{Tem}$	$A_{Tem}$	$r^2_{adj}$
Ben	CV	$190.351 \pm 167.61$	47.003	$0.029 \pm 0.033$	0.917	$8.865 \pm 2.837$	$1.469 \pm 0.286$	0.942	$228.512 \pm 53.651$	$5.084 \pm 3.832$	0.776
	RB	$10.777 \pm 1.283$	9.268	$0.405 \pm 0.203$	0.859	$3.390 \pm 0.427$	$2.675 \pm 0.351$	0.9542	$1196.448 \pm 159.03$	$5.733 \pm 2.704$	0.918
CTAB-Ben	CV	$245.573 \pm 53.205$	93.15	$0.046 \pm 0.016$	0.982	$16.074 \pm 1.155$	$1.480 \pm 0.061$	0.996	$91.82 \pm 16.806$	$2.055 \pm 0.968$	0.854
	RB	$32.444 \pm 4.411$	14.762	$0.066 \pm 0.016$	0.984	$2.843 \pm 0.352$	$1.552 \pm 0.123$	0.9817	$446.37 \pm 52.463$	$1.058 \pm 0.253$	0.935

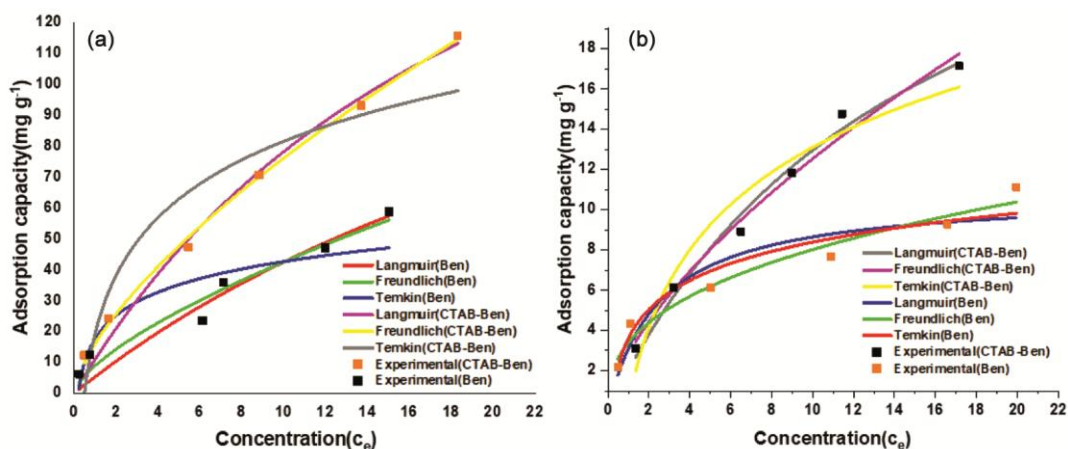


Fig. 2 — Plots of Langmuir, Freundlich and Temkin isotherm models for the adsorption of CV (left) and RB (right) onto Ben and CTAB-Ben

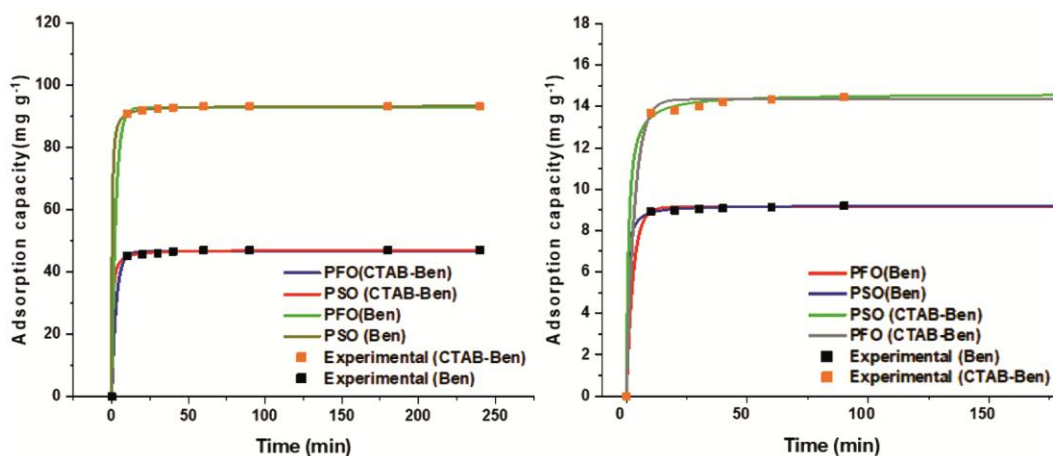


Fig. 3 — Plots of pseudo-first-order (PFO), pseudo-second order (PSO) models for the adsorption of CV (left) and RB (right) onto Ben and CTAB-Ben

Results of kinetic studies of CV and RB suggested that the adsorption onto Ben was slightly better explained by PSO model in comparison to PFO. However, results obtained from the kinetic experiments for CV onto CTAB-Ben displayed clear fitting of PSO model and the  $r^2_{Adj}$  values varied

significantly for PSO ( $r^2_{Adj}=0.973$ ) and PFO ( $r^2_{Adj}=0.701$ ). Similarly, the adsorption of RB onto CTAB-Ben, PSO model fitted slightly better in the kinetics data than PFO, but the  $r^2_{Adj}$  values were comparable. Thus, the results of kinetic studies of both dyes suggested the involvement of physical

Table 4 — Non-linear kinetic model parameters for CV and RB adsorption onto Ben and CTAB–Ben

Adsorbent	Dye	PFO				PSO				IPD		
		$q_{e,exp}$	$q_{e,calc}$	$k_1$	$r^2_{Adj}$	$q_{e,exp}$	$q_{e,calc}$	$k_2$	$r^2_{Adj}$	$k_i$	C	$r^2_{Adj}$
Ben	CV	47.003	46.628±0.197	0.344±0.037	0.998	47.003	47.109±0.131	0.044±0.006	0.999	10.946±2.892	14.263±7.807	0.624
	RB	9.268	9.143±0.038	0.366±0.046	0.998	9.268	9.229±0.027	0.260±0.046	0.999	2.150±0.566	2.794±1.528	0.626
CTAB–Ben	CV	93.150	92.869±0.177	0.378±0.023	0.701	93.150	93.420±0.071	0.036±0.002	0.973	0.906±0.281	90.083±0.807	0.571
	RB	14.761	14.344±0.126	0.3012±0.050	0.995	14.761	14.614±0.098	0.080±0.171	0.998	3.460±0.837	4.129±2.259	0.667

Table 5 — Standard error of prediction of studied non-linear isotherm and kinetic models for Ben and CTAB–Ben

Adsorbent	Dye	Isotherm models			Kinetic models		
		Langmuir	Freundlich	Temkin	PFO	PSO	IPD
Ben	CV	34.095	23.976	92.638	0.268	0.065	0.368
	RB	0.612	0.489	0.875	0.010	0.002	3.452
CTAB–Ben	CV	28.744	5.665	235.742	0.216	0.019	0.311

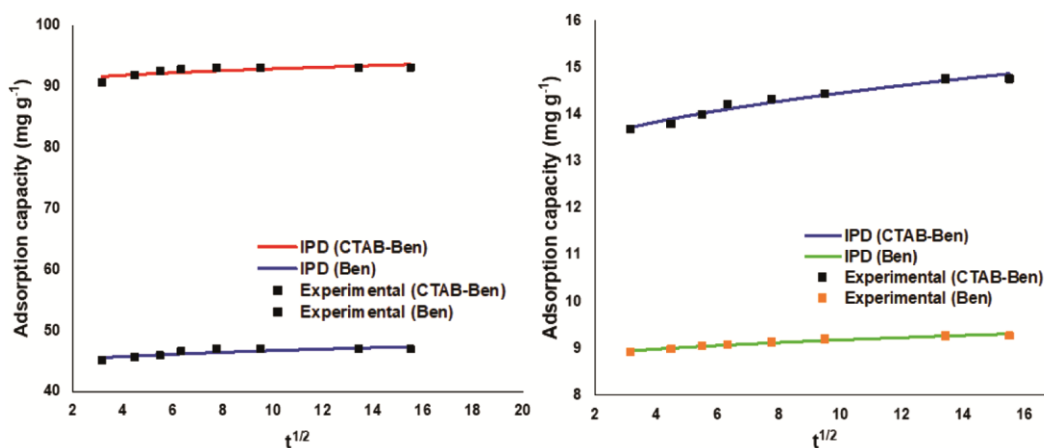


Fig. 4 — Plots of IPD model for the adsorption of CV (left) and RB (right) onto Ben and CTAB–Ben

adsorption assisted with chemical adsorption, a fast process and a probable reason for high dye adsorption at lower concentrations.<sup>20</sup>

#### Adsorption Mechanism

The intra-particle diffusion (IPD)<sup>19</sup> model was utilized to understand the adsorption mechanism behind the adsorption of CV and RB onto Ben and CTAB–Ben. The IPD plots,  $q_t$  versus  $t^{1/2}$  for Ben and CTAB–Ben are represented in Fig. 4 and their respective parameters are given in Table 4. The standard error of prediction of studied non-linear kinetic and isotherm models have been shown in Table 5. The results indicated that the boundary layer effect was significant in adsorption of CV onto Ben and CTAB–Ben whereas for RB it is not that much significant. Moreover, the value of the intercept for adsorption of both dyes was greater for CTAB–Ben than Ben. As observed from the IPD plots (Fig. 4), the plotted line did not pass through the origin. This suggested that IPD alone was not the rate determining

factor and boundary layer diffusion also determined the adsorption process.<sup>21</sup>

#### Regeneration Studies

To assess the feasibility and economic viability of the adsorbents, adsorption-desorption studies were conducted. For this purpose, 0.1 M NaOH was used as desorbing agent to remove adsorbed dyes from clays. Both adsorbents were exposed to five cycles of adsorption-desorption. Both the adsorbents exhibited highest desorption during first cycle for both CV and RB and after that it gradually decreased with each cycle. The dye removal (%) for Ben ranged between 80 to 65 and 76 to 60 for CV and RB respectively whereas for CTAB–Ben ranged between 82 to 70 and 78 to 65 for CV and RB respectively. Hence, the CTAB modified bentonite could be repeatedly used for dye removal.

#### Conclusions

Raw bentonite was modified with cationic surfactant (CTAB) and was used to remove CV and

RB from aqueous solution. Characterization studies revealed that CTAB modification induced changes in structure and surface properties of raw bentonite due to the intercalation of alkylammonium ions in between the layers. The maximum adsorption capacity of CTAB-Ben was 2 and 1.5 times higher than Ben for CV and RB respectively. The adsorption data of CV and RB onto CTAB-Ben followed PSO kinetic model and the Freundlich adsorption isotherm. Adsorption-desorption studies revealed high dye removal potential of CTAB-Ben upto five cycles. Outcomes of this study suggested that CTAB-Ben hold promise for dye removal and can be utilized on large scale.

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### Conflict of interest

The authors declare that there is no conflict of interest.

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