



Research Paper

## Adsorption Behavior of Crystal Violet Dye on to Tin (IV) Molybdovanadate Cation Exchanger from Aqueous Solution

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**Abstract:** A newly synthesized tin(IV) molybdovanadate, a hetero poly acid based cation exchanger was characterized on the basis of FT-IR, XRD, TGA and SEM. The structural studies revealed semi-crystalline nature of the material. The ion exchange capacity, chemical stability, and effect of temperature on ion exchange capacity were carried out to understand the ion exchange capability. Distribution studies showed that the selectivity of the exchanger towards various metal ions was in the order  $Pb^{II} > Cu^{II} > Cd^{II} > Co^{II} > Zn^{II} > Bi^{III} > Ni^{II} > Mn^{II} > Al^{III} > Mg^{II} > Hg^{II}$ . The UV-Visible spectrophotometric studies revealed the enhanced adsorption ability of this material towards organic pollutants like dyes. Exchanger can be used for the removal of crystal violet dye from aqueous solution. The effect of some important parameters such as initial concentration of dye, adsorbent dosage, and contact time on the uptake of organic dye from aqueous solution was also investigated. Freundlich and Langmuir isotherm models were applied to the equilibrium data. It was observed that adsorbent showed higher adsorption capacity for crystal violet ( $1.7 \text{ mg g}^{-1}$ ). The adsorption of dye was best described by pseudo second order mechanism. Waste water from textile industries were selected for checking the validity of this material.

**Keyword:** adsorption, distribution coefficient, dye, ion exchanger, isotherm, kinetics

### Introduction

Textile industries have shown a significant increase in the use of synthetic complex dyes as colouring materials. Dyes used in textile industry may be toxic to aquatic organisms and can be resistant to natural biological degradation<sup>[1-3]</sup>. Dyes itself are toxic to some organisms and hence disturb the ecosystem. So, the removal of synthetic organic dyestuff from waste effluents becomes environmentally important. The studies have been performed in order to remove color and other contaminations using various methods include adsorption by ion exchange process<sup>4</sup>, coagulation, nanofiltration and ozonolysis, membrane filtration, oxidation process, etc.

Good number of published papers related to the removal of dyes by adsorption was found in literature<sup>[5-8]</sup>. However, in view of the high cost and associated problems of regeneration, there is a constant search for alternate low-cost adsorbents. Such alternatives include spent tea leaves<sup>9</sup>, waste orange peel<sup>[10]</sup>, banana pith<sup>[11]</sup>, palm ash<sup>[12]</sup>, wheat straw<sup>[13]</sup>, a rice husk<sup>[14]</sup>, etc. But those adsorbents were chemically activated and after the use of those adsorbents, they will be discarded to our environment and may cause

other environmental hazards. However there is limited number of report only in literature about the dye adsorption of cation exchanger. Due to the presence of structural hydroxyl group in ion exchanger, it is expected the dye could either be bound to the matrix material by hydrogen bonds or weak Van der Waals forces, making sorption and desorption easy and possible. Therefore present studies reveals the application of the newly synthesized ion exchanger tin(IV) molybdovanadate which is capable of removing organic dyes from aqueous solution. UV – Visible spectrophotometric technique is used to study the adsorption behaviour of crystal violet on to the material.

### Material and Methods

**Reagents:** Stannic chloride (E. Merck), Sodium molybdate (E. Merck) and Sodium vanadate (E. Merck) were used for the synthesis of the exchangers. All other reagents and chemicals used were of analytical grade.

**Apparatus and instruments:** A glass column was used for column operations. ELICO LI613 pH meter was used for pH measurements. FT-IR Spectrometer model Thermo-Nicolet Avtar 370 for IR studies, X-ray Diffractometer

Bruker AXS D8 Advance for X-ray diffraction studies, JEOL Model JSM - 6390LV for scanning electron microscopic analysis, TG Perkin Elmer Diamond TG/DTA Analysis System for thermogravimetric / derivative thermogravimetric analysis were used. UV-Visible Spectrophotometer model JASCO V660 was used for spectrophotometric measurements.

**Synthesis of the exchanger:** The precipitate of tin(IV) molybdovanadate (SnMoV) was prepared by adding gradually the solution of stannic chloride into the mixture of sodium molybdate and sodium vanadate with constant stirring at room temperature for 1 hr whereby a gel type slurry was obtained. The resulting precipitate was kept overnight in the mother liquor for digestion. After decanting off the supernatant liquid, the remaining precipitate was washed and filtered with demineralized water to remove any excess reagent. The material was dried in an oven at  $50 \pm 2^\circ\text{C}$ . In order to convert the material in  $\text{H}^+$  form, it was subsequently treated with 1.0 M  $\text{HNO}_3$  solution for 24 hrs with occasional shaking and intermittently replacing the supernatant liquid with fresh acid. The excess of acid was removed by washings with demineralized water and finally dried in an oven at  $50 \pm 2^\circ\text{C}$ . The material was sieved and kept in a desiccator.

**Ion exchange capacity (IEC) and Effect of temperature on IEC:** The ion exchange capacity of the material was determined by column method<sup>[15]</sup>. The effect of temperature on ion exchange capacity was studied<sup>[15]</sup>.

**Distribution coefficient ( $K_d$ ):** Distribution studies were carried out for various metal ions in demineralized water by batch process<sup>[16]</sup>.

**Adsorption Experiments:** Adsorption equilibrium and kinetics were determined by the batch method. Dye adsorption experiments were carried out by taking 10 mL stock solution of dye ( $10 \text{ mg L}^{-1}$ ) in a conical flask and treated with a dose of 200 mg of the adsorbent and kept for agitation for 2 hrs. The variable studied were agitation time, adsorbent dose, initial dye concentration. After desired time of treatment of samples were filtered to remove the adsorbent. The progress of adsorption was estimated using UV-Visible spectrophotometer at 580 nm. The percentage removal of dye from solution was calculated by the following:

$$\% \text{Removal} = \frac{(C_o - C_t)}{C_o} \times 100,$$

Where,  $C_o$  and  $C_t$  ( $\text{mg L}^{-1}$ ) are the initial dye concentration and concentration at time  $t$ , respectively.

The amount of adsorption at time  $t$ ,  $q_t$  ( $\text{mg g}^{-1}$ ) was calculated by

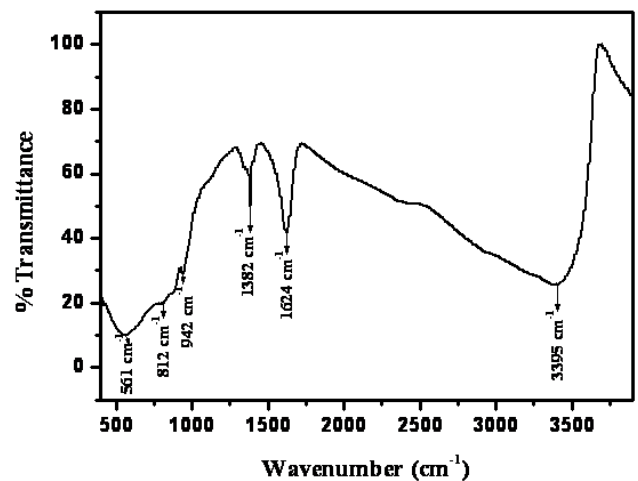
$$q_t = \frac{(C_o - C_t)V}{W}.$$

**Waste water treatment:** 100 mL of waste water from textile industries were collected. The solid waste was removed by centrifugation. The initial concentration of crystal violet in the waste water was estimated using UV-Visible Spectrophotometer. The 100 mL aliquant was equilibrated with 2.0 g of exchanger for 2hrs and the final concentration of crystal violet was measured by UV-Visible Spectrophotometer.

## Results and Discussion

The exchanger, SnMoV obtained as dark yellow transparent semicrystalline solid having maximum ion exchange capacity of  $1.01 \text{ meq g}^{-1}$  was selected for detailed study.

FT-IR spectra of SnMoV (Figure 1) showed a broad band in the region  $\sim 3395 \text{ cm}^{-1}$  which is attributed to symmetric and asymmetric  $-\text{OH}$  stretching, while the band at  $\sim 1624 \text{ cm}^{-1}$  is attributed to H-O-H bending. A band in the region  $\sim 1384 \text{ cm}^{-1}$  is attributed to the presence of  $\delta$  (MOH). This indicates the presence of structural hydroxyl protons in SnMoV, which is more evident from the obtained IEC values. Bands at  $\sim 942 \text{ cm}^{-1}$ ,  $812 \text{ cm}^{-1}$  and  $\sim 516 \text{ cm}^{-1}$  may be due to the presence of Sn-O-H, Mo-O-Sn and Sn-O bonds.



**Figure 1: FT-IR of SnMoV**

X-ray diffractogram (Figure 2) showed some prominent peaks together with a number of low intensity peaks which suggested semi-crystalline nature of the exchanger.

It was observed that on heating at different temperatures for 3 hrs, the dried sample materials retained its prominent IEC even at the temperature of  $500^\circ\text{C}$  (Figure 3).

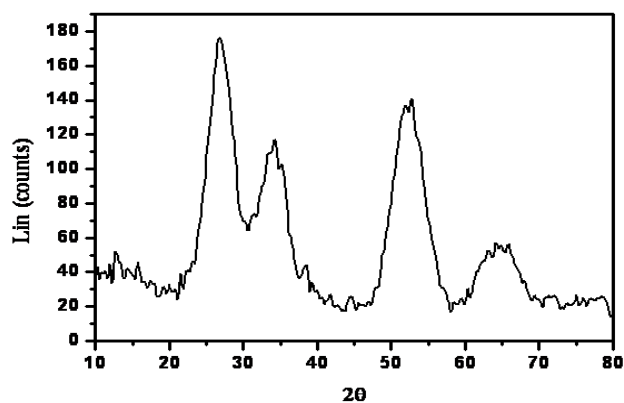


Figure 2: XRD of SnMoV

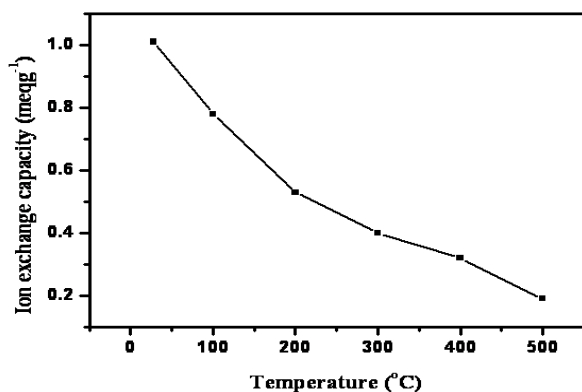


Figure 3: Temperature effect on Ion exchange capacity

The thermogram of SnMoV composite cation exchanger (Figure 4) exhibited the weight loss (about 14%) of the ion exchanger up to  $\sim 161$  °C which was due to the removal of free external water molecules. Further, a gradual loss of mass (about 6%) up to 500 °C may be account of the condensation of hydroxyl groups. Above 500 °C, the gradual loss in weight (about 1%) up to 800 °C was mainly because of the removal of structural water. There was no sudden decrease in the weight loss which indicated that there was no structural change of this material. The percentage weight loss with increasing temperature observed by TGA curve for SnMoV was also supported by the thermal stability experiment.

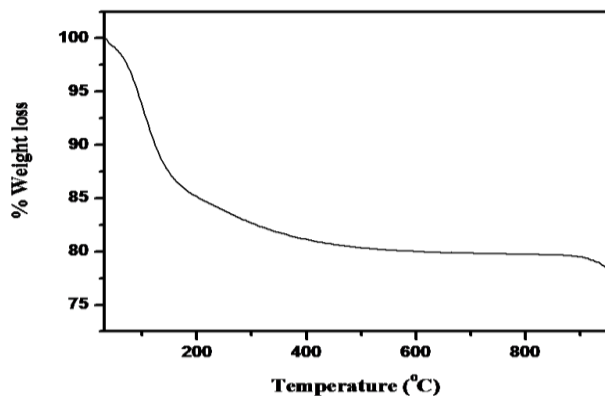


Figure 4: TGA curves of SnMoV

In order to explore the potentiality of the newly synthesized cation exchange material in the separation of metal ions, distribution studies for metal ions were performed in different solvent systems. The distribution studies of metal ions (Table 1) evidenced that the exchanger has affinity towards  $\text{Pb}^{\text{II}}$  in comparison to other metal ions studied. The selectivity was found to be in the order  $\text{Pb}^{\text{II}} > \text{Cu}^{\text{II}} > \text{Cd}^{\text{II}} > \text{Co}^{\text{II}} > \text{Zn}^{\text{II}} > \text{Bi}^{\text{III}} > \text{Ni}^{\text{II}} > \text{Mn}^{\text{II}} > \text{Al}^{\text{III}} > \text{Mg}^{\text{II}} > \text{Hg}^{\text{II}}$ . The effect of electrolyte concentrations on distribution coefficients designated that the value decreases with increase in electrolyte concentrations.

Scanning electron microscope (SEM) photograph of SnMoV depicted its irregular shape (Figure 5) and the rough surface with pleats, providing a good possibility for dyes to be trapped and adsorbed.

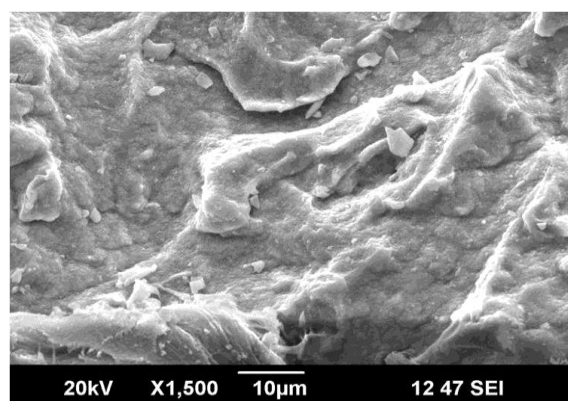


Figure 5: SEM image of SnMoV

### Removal of crystal violet

The effect of adsorbent dosage on the removal of CV has shown in the Figure 6a. To study the effect of adsorbent dose on dye adsorption, different amounts of adsorbents varying from 100 mg to 500 mg were respectively added to initial concentration of  $10 \text{ mgL}^{-1}$  of CV solution of 10 mL and kept for 2 hrs. Removal of dye with increasing adsorbent dosage was observed which is due to the availability of reactive sites<sup>9</sup>. The removal of CV ranged from 75.6 to 99.5 % for 2 hrs when treated with different doses of exchanger.

The studies on the removal of CV by exchanger were carried out at different concentration of dye from  $5 \text{ mgL}^{-1}$  to  $40 \text{ mgL}^{-1}$  by keeping the fixed adsorbent dosage of 200 mg shown in Figure 6 b. The uptake of CV per unit mass of adsorbent increased with the increase in initial concentration, although percentage of removal decreased with the increase in initial concentration. The increased uptake of CV from  $5 \text{ mgL}^{-1}$  to  $40 \text{ mgL}^{-1}$  concentration of CV may be attributed to an increase in the driving force of the concentration gradient with the increase in the initial dye concentration<sup>[17]</sup>. From the figure, it was revealed that there was significant removal of dyes even at higher concentration also possible with this exchanger. The percentage removal decreases with the increase in dye concentration indicated that there exists a reduction in

immediate solute adsorption, owing to the lack of available active sites required for the high initial concentration of dye (CV). The enhanced removal at low concentration could be due to the faster movement of dye into the activated sites of composite.

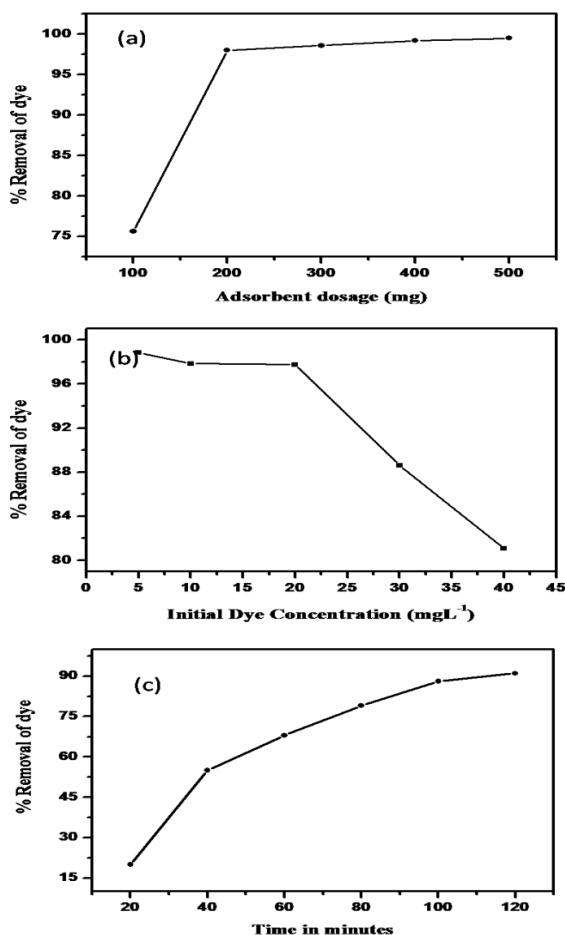
However, in higher concentration (40 mgL<sup>-1</sup>) the removal rate was decreased to 81.07% because the dye

molecules needed to diffuse to the adsorbent sites by intra particle diffusion. In addition, steric repulsion between the solute molecules could slow down the adsorption process and thereby decreases the removal rate. A similar trend was observed for the adsorption of methyl violet by agricultural waste<sup>18</sup>.

**Table 1: K<sub>d</sub> values of various metal ions in different electrolyte**

Metal ion	DMW	0.001 M HNO <sub>3</sub>	0.01 M HNO <sub>3</sub>	0.1 M HNO <sub>3</sub>	0.001 M NH <sub>4</sub> NO <sub>3</sub>	0.01 M NH <sub>4</sub> NO <sub>3</sub>	0.1 M NH <sub>4</sub> NO <sub>3</sub>
Pb <sup>II</sup>	200.98	175.98	100.09	78.50	189.20	116.70	52.33
Zn <sup>II</sup>	34.40	20.78	10.00	4.50	34.01	17.88	2.10
Mn <sup>II</sup>	21.60	134.10	98.60	42.10	123.10	67.30	21.20
Ni <sup>II</sup>	26.50	18.90	12.10	1.10	25.90	15.00	1.90
Hg <sup>II</sup>	9.40	NA	NA	NA	4.50	NA	NA
Al <sup>III</sup>	18.00	14.50	3.80	NA	15.23	3.20	NA
Co <sup>II</sup>	43.30	38.80	24.11	11.10	40.00	21.98	5.66
Cu <sup>II</sup>	59.60	39.12	24.10	15.30	59.10	23.43	10.01
Bi <sup>III</sup>	33.30	32.11	11.90	1.00	30.10	12.00	NA
Mg <sup>II</sup>	13.60	12.90	7.30	1.01	13.10	7.20	NA
Cd <sup>II</sup>	46.20	34.21	20.90	14.60	45.21	15.90	5.78

NA: No observable adsorption



**Figure 6: (a) effect of adsorbent dosage, (b) effect of dye concentration, (c) effect of time on the removal of CV by SnMoV**

The contact time between the pollutant and the adsorbent is of significant importance in the wastewater treatment by adsorption. In physical adsorption, most of the adsorbate species are adsorbed within a short interval of contact time. However, strong chemical binding of the adsorbate with adsorbent requires a longer contact time for the attainment of equilibrium. This is obvious from the fact that a large number of vacant surface sites are available for adsorption during the initial stage, and after a lapse of time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases. The effect of contact time on the adsorption of CV at 30 °C was studied for a period of 2 hrs (Figure 6c).

**Adsorption Isotherms:** The study of adsorption isotherm has been of important and significant in the waste water treatment by adsorption technique as they provide an approximate estimation of the adsorption capacity of the adsorbent. The equilibrium data for the removal of dye on exchanger were used in the Langmuir<sup>[19]</sup> and Freundlich<sup>[20]</sup> isotherms.

Freundlich isotherm  $\log q_e = \log K_f + (1/n) \log C_e$   
 Langmuir isotherm  $C_e/q_e = (1/Q_{ob}) + (C_e / Q_o)$

The data obtained from the adsorption experiments were fitted into Langmuir and Freundlich isotherms respectively by plotting  $C_e/q_e$  and  $\log q_e$  against  $C_e$  and  $\log C_e$ . They are found to be linear, indicating the formation of monolayer of adsorbate on the outer surface of adsorbent and after that no further adsorption takes

place (Figure 7). Further, the essential characteristics of the Langmuir isotherms can be described by a separation factor ( $R_L$ ).

The values of separation factor ( $R_L$ ) indicates the shape of the isotherm and nature of the adsorption process as given here,  $R_L$  values Nature of the adsorption process,  $R_L > 1$  is Unfavorable,  $R_L = 1$  is Linear,  $0 < R_L < 1$  is Favorable and  $R_L = 0$  Irreversible<sup>[21]</sup>. Values of  $R_L$  were found to be  $< 1$  (0.01 - 0.10) which confirmed that the exchanger is favourable for adsorption of CV under experimental conditions used in this study.

The fitting data results suggest that Langmuir model could be applied to the adsorption of CV ( $R^2 = 0.9968$ ) while Freundlich model ( $R^2 = 0.8975$ ) is not very suitable for this adsorption. The Freundlich adsorption isotherm is an indication of surface heterogeneity of the adsorbent while the Langmuir isotherm assumes that the adsorption occurs in a monolayer, or the adsorption may only occur at a fixed number of localized sites on a surface on which all adsorption sites are identical and energetically equivalent. This leads support to the conclusion that the surface of the treated sample investigated was likely made up of small homogenous adsorption patches, which were quite similar to each other in respect of adsorption phenomenon. This implies that the adsorption of CV takes place as monolayer adsorption on a surface that is homogeneous in surface affinity. On the basis of the Langmuir analysis, the maximum adsorption capacity was determined to be  $1.7 \text{ mg g}^{-1}$ .

**Adsorption kinetics:** Adsorption kinetics provides valuable information about the reaction pathways and mechanism of the reactions. Pseudo first-order<sup>[22]</sup> and pseudo second-order<sup>[23]</sup> models are employed to elucidate the mechanism embedded in this process.

The pseudo-first-order equation is expressed as follows<sup>[24]</sup>:  

$$dq_t / dt = k_1 (q_e - q_t)$$

The integrated form of the equation is as follows:  

$$\text{Log} (q_e - q_t) = \text{log} q_e - k_1 t / 2.303$$

where  $q_e$  and  $q_t$  are the amounts (mg/g) of solute bound at the interface at the equilibrium and after time  $t$  (min), respectively,  $k_1$  is the rate constant of the pseudo-first-order adsorption ( $\text{min}^{-1}$ ).

The pseudo-second-order kinetic model expressed in the most commonly used form, formulated by Ho and McKay is as follows:

$$d_e / dt = k_2 (q_e - q_t)^2$$

The integrated form of equation is:

$$t / q_t = 1 / k_2 q_e^2 + t / q_e$$

where  $k_2$  is the rate constant of the pseudo-second-order adsorption (g/mg min),  $q_t$  is the adsorbed amount at time  $t$  (mg/g).

The plots validating the pseudo-first-order model ( $\text{log} (q_e - q_t)$  versus  $t$ ) and the pseudo-second-order model ( $t/q_t$  versus  $t$ ) at various initial concentrations of the CV, at 302 K, are given in Figure 8a and b, respectively. Parameters of the fitted kinetic models with the correlation coefficients,  $R^2$ , are 0.885 and 0.986 for pseudo first order and pseudo second order kinetic models. On examining the models in terms of the  $R^2$  value, it shows that the pseudo second order kinetic model describes the system much better than the pseudo first order kinetic model. Similar results have been reported in literature and according to this assumption, chemical sorption involving valence forces through sharing or exchange of electrons between adsorbent and adsorbate might significantly affect the rate-limiting step<sup>[25-27]</sup>.

**Waste water treatment:** The utility of the exchanger prepared was evaluated for the treatment of textile waste water which contains crystal violet collected from different area of Kannur, Kerala. The concentrations of total crystal violet in the two samples were  $0.99 \text{ mg L}^{-1}$  and  $0.95 \text{ mg L}^{-1}$ . pH of the samples was 7.45 and 7.32. The treatment results are presented in Table 2.

## Conclusion

In this study adsorption of crystal violet dye from aqueous solution was investigated using tin(IV) molybdovanadate cation exchanger as an adsorbent. UV spectra reveal that the exchanger has high adsorption capability for the removal of crystal violet from aqueous solution as well as waste water obtained from various textile industries. In addition the adsorbent can be recycled thrice without any loss in its capacity and efficiency by treatment with 1.0 M  $\text{HNO}_3$  solution. The present study concludes that the exchanger will be a potential candidate for the removal of heavy metal ions such as lead and organic dyes like crystal violet from aqueous solution.

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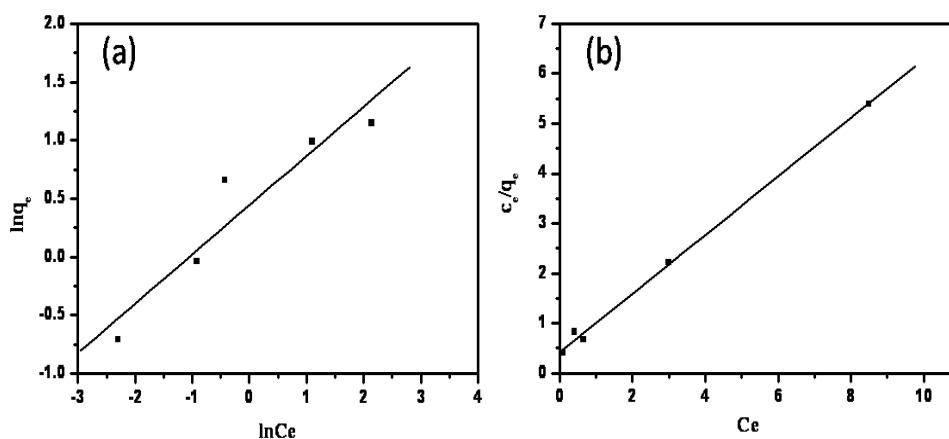


Figure 7: (a) Freundlich and (b) Langmuir adsorption plot

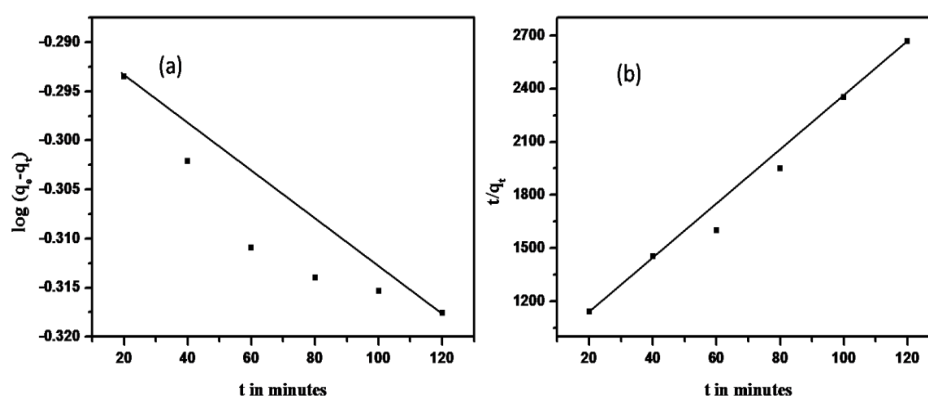


Figure 8: (a) Test of the pseudo-first-order adsorption kinetics for CV adsorption on SnMoV and (b) test for the pseudo-second-order adsorption kinetics for CV adsorption on SnMoV

Table 2  
Removal of Crystal Violet from the waste water by tin(IV) molybdovanadate

Samples	pH	Initial concentration of CV (mgL <sup>-1</sup> )	Final concentration of CV (mgL <sup>-1</sup> )	% Removal
1	7.45	0.99	0.093	70
2	7.32	0.95	0.089	72

\*Amount of tin(IV) molybdovanadate: 2.0 g; Amount of waste water: 100 mL; Standing time: 2 hrs

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