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Assessment on linear and non-linear analysis for the estimation of pseudo-second-order kinetic parameters for removal of dye using graphene nanosheet

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ABSTRACT

Graphene nanosheets were used as adsorbent for the removal of crystal violet dye from its aqueous solution. The equilibrium kinetic data were analyzed using pseudo-second-order kinetic model. A comparison between linear and non-linear methods of estimating the kinetic parameters was examined. Four pseudo-second-order kinetic linear equations have been discussed here. The coefficient of determination (r^2) and chi-square (χ^2) test were employed as methods of error analysis for determining the best-fitting equation. The results revealed that the non-linear method proved to be a significantly better alternative for obtaining the kinetic parameters in comparison with the linear form of the model. In addition, the (χ^2) test was found to be a better method for determination of the best-fitting model.

Keywords: Crystal violet (CV); Graphene; Nanosheets; Pseudo-second-order; Linear method; Non-linear method

1. Introduction

Every year a large volume of effluent rich in dyes and other colored pigments are generated from textile manufacturing and processing industries. These dyes when discharged through wastewater are highly undesirable and of enormous concern due to their ecotoxicological effect on surrounding environment and life. Several physical, chemical, and biological methods developed for remediation of this dye include coagulation, microfiltration, chemical precipitation, membrane filtration, solvent extraction, reverse osmosis, photocatalytic degradation, sonochemical degradation, micellar-enhanced ultrafiltration, cation-exchange membranes, electrochemical degradation, etc. [1–5]. But most of these techniques are difficult to implement on wide scale and not very efficient in some cases as well. Attempts have thus been made by various researchers to find alternative, convenient and cost-effective substitutes of these existence techniques. In order to develop alternative environmental remediation strategies, the process of adsorption is currently receiving great importance as the procedure incurs low initial cost, simplicity of design, and ability to reduce the pollution level to near totality. This process incurs passive uptake and binding of chemical species or ions to a solid surface. So the study of adsorption is of great

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significance for achieving maximum removal of toxic and hazardous environmental pollutants. [6–13].

Recently discovered graphene nanosheets have been reported to possess remarkable potential for environmental pollution control and abatement. Graphene is mainly a group of sp^2-hybridized carbon atoms packed into a two-dimensional honeycomb network. These carbon nanomaterials have well-defined uniform structure in comparison to that of activated carbon and hence have higher adsorption capacity. They also have varying molecular size and can be easily regenerated.

This one atom allotrope of carbon can be wrapped into zero-dimension buckyballs or rolled into one-dimension sheets. It is one of the strongest materials that possess both brittle and ductile properties. It is impermeable to even the smallest gas molecule. Each carbon atom of the lattice has π orbital which contributes a delocalized network of electrons rendering the structure of graphene as highly stable. Graphene offers a unique contribution of 3-D aspect ratio and large specific surface area as well. As a result, this carbon nanomaterial can be used in different fields, such as nanoelectronics, structural composites, conductive polymers, electrodes, etc. The application of graphene nanosheets as adsorbent depends on the homogeneous dispersion of the same in liquid phase for the removal of pollutants present in solution. But this material has a tendency to agglomerate and restack to form graphite during liquid processing [11,13–18].

In this study, an attempt has been made to synthesize graphene nanosheets as adsorbent material to reduce the effect of crystal violet (CV) from solution. The effects of CV dye include cancer, methemoglobinemia, severe inflammatory responses in GI tract, eye irritation, cytogenetic toxicity (acts as mitotic poison), etc. [6]. The present study includes analysis of adsorption kinetics, i.e. solute removal rate from solvent as it controls the residence time of sorbate in solid–solution interface. Kinetic study is also essential for selecting the optimum conditions for carrying out batch reactions. Out of the several available methods of adsorption kinetics, pseudo-first- and pseudo-second-order kinetics were estimated as they have been widely used in studies on various kinds of adsorbents and are regarded as most suitable for the study on kinetics of dye adsorption [20–24].

The results obtained from batch studies were thereby fitted using these two models. It was observed from the fitted rate constants and predicted equilibrium uptakes along with the corresponding correlation coefficients (Figure not shown) that the equilibrium uptake values predicted by the pseudo-first-order model were comparatively lower than the experimental values obtained. For the pseudo-second-order model, the correlation coefficients were found to be higher than 0.98 in most studies [25–28]. Moreover, the calculated equilibrium uptake values agreed very well with the experimental data.

The kinetic equation, yielding a coefficient of determination closest to unity, was considered as the best-fitting equation. However, an interest in the utilization of non-linear optimization modeling has been noted in recent studies. A probable reason is that such transformation of non-linear equations to linear forms alter the error structure and may also disregard the error variance and normality assumptions of standard least squares [21,25]. As a result, one may obtain variable kinetic parameters when using different forms of a kinetic model for a given adsorption process. Besides, the non-linear method for analyzing the experimental data also provides a more complex mathematical method for determination of kinetic parameters and is conducted on the same abscissa and ordinate, thus avoiding the drawbacks of linearization [25–30].

In this present study, a comparison of linear least-square method and non-linear method for estimating the kinetic parameters of the pseudo-second-order kinetic model were examined based on the experiment of CV adsorption from its aqueous solution onto graphene nanosheet. The problems associated with linearization have also been discussed. It is to be noted that the results obtained in the present study is limited to the specific graphene nanosheet/CV system. However, the general trend may still be valid.

2. Materials and methods

2.1. Synthesis of graphene oxide and graphene

Graphene oxide was synthesized by the modified Hummers' method [19]. The synthesis was performed by exfoliating graphite powder in the presence of potassium permanganate ($KMnO_4$) and concentrated sulfuric acid (H_2SO_4)[g]. The product solution was sonicated and filtered in vacuum filter by repeated washing with distilled water and 10% (HCl) to remove metal ions. Graphene oxide obtained as residue on the filter paper was dried in hot air oven at 60˚C for 48 h [19].

For synthesis of graphene nanosheets, 3.0 g of graphene oxide in 100 mL of distilled water was taken and heated in microwave oven at a temperature around 45˚C. Then, hydrazine hydrate (H_2O_4) was added to the solution and the color of solution changed from brown to black. The solution was put in a shaker at 120 rpm, 35℃ for 150 min. After this the solution was sonicated and filtered with vacuum filter, the cake which was formed was Graphene. It was again dried at 60℃ for 48 h.

2.2. Batch studies of adsorption

All the experiments of adsorption were performed in 250 mL Erlenmeyer flasks under continuous stirring and constant temperature. The dye concentration was varied from 10 to 300 mg/L and the stock solution of CV dye was prepared separately for each set of experiment. In each set of experiments, four different concentrations (25, 50, 75, and 100 mg) of adsorbent was taken separately and added in different conical flasks containing 100 mL of dye solution keeping dye concentration in solution constant. The flasks were agitated at a constant speed of 150 rpm and incubated at 35 ± 1℃ in an incubator shaker (Model Innova 42, New Brunswick Scientific, Canada) for 3 h. Each flask was capped to avoid evaporation at high temperature. Aliquots were sampled at regular time intervals and centrifuged to settle the particles present in the samples, which were collected. The initial and final concentration of CV in the solution was analyzed using UV/VIS spectrophotometer (Model Hitachi—2800) at $\lambda_{\max}$ of 589 nm.

The amount of dye adsorbed at equilibrium q_e (mg g^{-1}) was calculated by:

$$q_e = \frac{(C_i - C_e)V}{m} \tag{1}$$

where C_i is the initial dye concentration (mg L^{-1}), C_e is the equilibrium dye concentration in solution (mg L^{-1}), V is the volume of the solution (L), and m is the mass of the adsorbent used (g).

In order to ensure the accuracy, reliability, and reproducibility of the collected data, all experiments were carried out thrice and the mean values were considered.

3. Results and discussion

3.1. XRD analysis of graphene oxide and graphene

The XRD analysis was done to investigate the interlayer spacing of the synthesized graphene oxide and graphene (Fig. not shown). Two peaks were observed at $2\theta = 11.1$ and at $2\theta = 42.62$, respectively. Typical (002) and (100) peaks of graphene oxide were observed at $2\theta = 11.1$ and 42.62, respectively, which could be indexed to the characteristic peaks (002) and (100) plane reflections shows with an inter layer spacing of 0.796 nm and 0.211 nm which is similar to the reported value of graphene oxide $2\theta = 13.2$ [6], $2\theta = 13.5$ [7], $2\theta = 10.8$ [2,3], and $2\theta = 11.36$ [1]. Two peaks were observed for graphene at 2θ= 26 and 43, which could be indexed to the characteristic peaks (002) and (100) plane reflections of graphite from the graphene [11].

3.2. Pseudo-second-order kinetic model

The pseudo-second order kinetic model was proposed by Ho [20], and is derived on the basis of the sorption capacity of the solid phase. The differential form of the model is expressed as [21,22]:

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

where q_t and q_e are the amounts of dye adsorbed at time t and at equilibrium (mg g^{-1}) and k_2(g mg^{-1} min^{-1}) is the pseudo-second-order rate constant for the adsorption process. Integrating and applying boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$, Eq. (2) becomes

$$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t} \tag{3}$$

Eq. (3) can be linearized to at least four different forms [21]. The different linearized forms of the pseudo-second-order equation are given in Table 1. The most popular linear form used is category 1 [9].

For category 1 model, q_e and k_2 can be calculated using the expression:

$$q_e = \frac{1}{slope}; \quad k_2 = \frac{slope^2}{intercept} \tag{4}$$

For category 2, the q_e and k_2 can be calculated by:

$$q_e = intercept; k_2 = \frac{-1}{(intercept\ x\ slope)} \tag{5}$$

For category 3, the q_e and k_2 can be calculated by:

$$q_e = \frac{-intercept}{slope}; k_2 = \frac{slope^2}{intercept} \tag{6}$$

For category 4, the q_e and k_2 can be calculated by:

Table 1
Different linearized forms of the pseudo-second-order equation

Category	Linear form of pseudo-second-order model	Plotting
Category 1	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$	t/q_t vs. t
Category 2	$q_t = q_e - \frac{1}{k_2 q_e} \frac{q_t}{t}$	q_t vs. q_t/t
Category 3	$\frac{q_t}{t} = k_2 q_e^2 - k_2 q_e q_t$	q_t/t vs. q_t
Category 4	$\frac{1}{q_t} = \frac{1}{q_e} + \frac{1}{k_2 q_e^2} \frac{1}{t}$	$1/q_t$ vs. $1/t$

$$q_e = \frac{1}{intercept}; \quad k_2 = \frac{intercept^2}{slope} \tag{7}$$

3.3. Error analysis

To evaluate the fit of the equations to the experimental results, error functions are required. In the present study, the coefficient of determination (r^2) and the chi-square (χ^2) tests were used in order to compare the applicability of each model quantitatively [23–26]:

$$r^2 = \frac{\left(q_{e,meas} - \overline{q_{e,cal}}\right)^2}{\sum \left(q_{e,meas} - \overline{q_{e,cal}}\right)^2 + \left(q_{e,meas} - q_{e,cal}\right)^2} \tag{8}$$

$$\chi^2 = \sum \frac{\left(q_{e,meas} - q_{e,cal}\right)^2}{q_{e,cal}} \tag{9}$$

where $q_{e,meas}$ and $q_{e,cal}$ (mg g^{-1}) are the measured and calculated adsorbate concentration at equilibrium, and $\overline{q_{e,cal}}$ (mg g^{-1}) is the average of $q_{e,cal}$.

3.4. Linear and non-linear method

The experimental data were used for pseudo-first- and pseudo-second-order kinetic models and it was observed that pseudo-second-order expression could represent the experimental data better than pseudo-first-order kinetic.

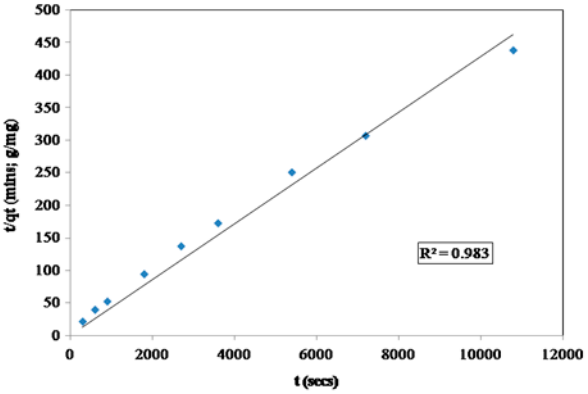

Fig. 1. Pseudo-second-order kinetics obtained using linear method for the sorption of CV onto graphene nanoparticle using category 1.

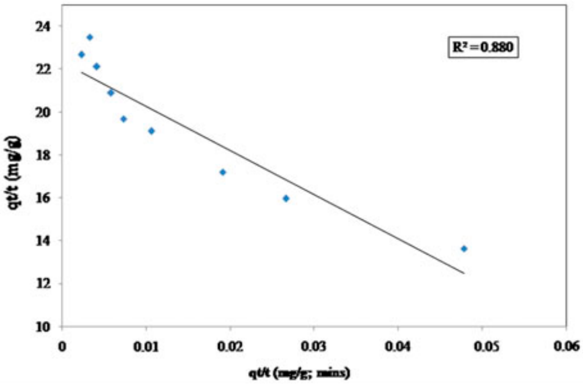

Fig. 2. Pseudo-second-order kinetics obtained using linear method for the sorption of CV onto graphene nanoparticle using category 2.

Method of least square has been used to find out the parameters of kinetics models. For this study, four different linearized forms of pseudo-second-order kinetics were used (Table 1). For category 1 linearized form, theoretical q_e and kinetic constant k_2 were calculated from the plot of t/q_t vs. t (Fig. 1). q_e was calculated using $1/slope$ and k_2 using: $slope^2/intercept$. Similarly, q_e and k_2 were determined for category 2, category 3, and category 4 expressions, respectively (Fig. 2–4; Eqs. 5–7). The detailed values of k_2 and q_e and the error function values have been provided in Table 2. It has been observed that the calculation of kinetic constant and q_e, obtained from the pseudo-second-order expression were totally different. Values of r^2 obtained were lower and χ^2 was higher for category 2, category 3, and category 4 models, indicating

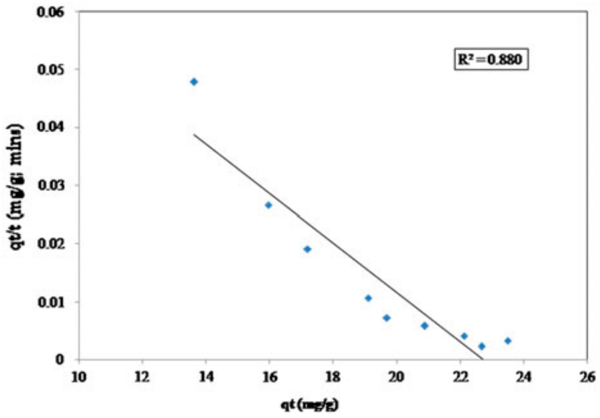

Fig. 3. Pseudo-second-order kinetics obtained using linear method for the sorption of CV onto graphene nanoparticle using category 3.

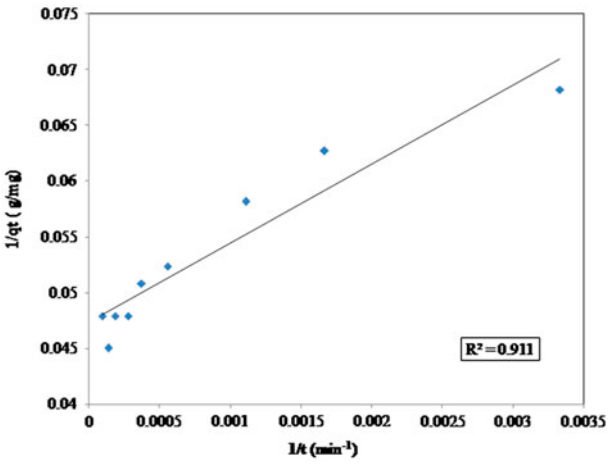

Fig. 4. Pseudo-second-order kinetics obtained by using linear method for the sorption of CV onto graphene nanoparticle using category 4.

that these expressions poorly fitted the pseudo-second-order model. On the other hand, category 1 expression of linearized form suggested that this model could appropriately represent the phenomenon of adsorption of CV using graphene nanosheets and was hence considered valid for representing the kinetic model.

These different observations showed the complexities of sorption kinetics experienced in practical application of different models and thereby helped to conclude that the resulting phenomenon was due to the alteration of error structure which varied as a result of conversion from non-linear form to linear form. This conversion might be held responsible for the violation of certain assumptions considered at the time of non-linear model formation. During the conversion, assumption of normality of least square analysis experienced distortion as most of the kinetic models were non-linear due to the different mechanisms of operation. For the same reason, non-linear expressions produced a poor fit to experimental data, after transformation. Hence, results revealed that category 3 and category 4 of pseudo-second-order expressions produced a poor fit to experimental data obtained as category 1 expression. Due to the variations in axial settings, the result of non-linear regression yielded better results in comparison to the linearized form. This linear form reported only the slope and intercept for linear trend lines that could only predict the y value for a given x. Hence, it was evident that the linear method was not appropriate for predicting best fit kinetics to understand the adsorption kinetics of the present study. This drawback of the linear form could be compensated using non-linear regression as the latter was conducted on the same abscissa and ordinate values, resulting in same error distribution and structure. Therefore, further studies were performed using non-linear regression method. Similar findings were also reported in previous studies by the authors [27–30].

Experimental values and the value of pseudo-second-order kinetic using non-linear method are

Table 2
Pseudo-second-order kinetic parameters obtained using the linear and non-linear methods

Kinetic models	q_e, exp (mg g^{-1})	q_e (mg g^{-1})	k_2 (g mg^{-1} min^{-1})	r^2	χ^2
Linear type 1	24.67	25.64	0.0043	0.983	0.036
Linear type 2		22.32	0.013	0.880	0.2474
Linear type 3		24.0	0.01	0.880	0.0187
Linear type 4		21.276	0.0187	0.911	0.542
Non-linear		24.089	0.055	0.994	0.014

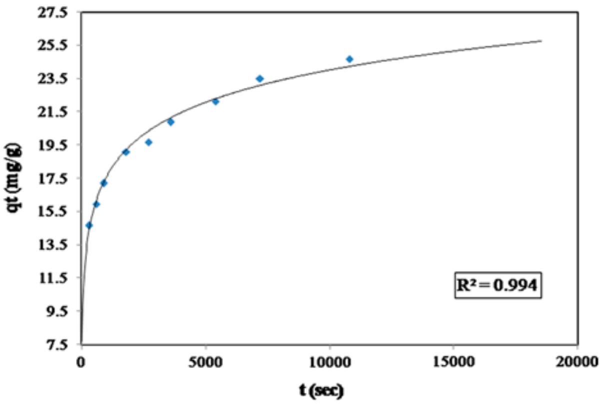

Fig. 5. Pseudo-second-order kinetics obtained using non-linear model for the sorption of CV onto graphene nanoparticle.

given in Fig. 5. The resulting k_2 and q_e have been listed in Table 2. High r^2 (0.994) and low χ^2 (0.014) values, calculated in case of non-linear expression, confirmed that non-linear expression represented the best fit to the pseudo-second-order kinetic expression than linear form with the error function distribution remaining unaltered. Further, for linear models, category 1 expression gave the best fit among others owing to the highest coefficient of determination obtained from the given equation. Consequently, it can be inferred that it would be most reliable to interpret the adsorption kinetic data through a non-linear regression analysis.

4. Conclusion

The adsorption kinetics of CV dye using graphene nanosheets were analyzed using pseudo-second-order kinetic models. The kinetic models were transformed to four linear forms and a comparative assessment was performed. From high r^2 and low χ^2 values of non-linear expression, it was suggested that the non-linear expression represented the best fit expression to the pseudo-second-order kinetic expression than other linear forms. The following conclusions were also obtained:

- Use of linear form of regression models for determining the kinetic parameters was not appropriate due to alterations in the error distribution, which had resulted from transformations.
- This drawback could be minimized using non-linear expression of pseudo-second-order model. It was observed that non-linear kinetic model provided the best description of the kinetics of CV adsorption onto graphene nanosheets.

Nomenclature

C_e	[mg L^{-1}]	—	equilibrium dye concentration in the solution
C_i	[mg L^{-1}]	—	initial dye concentration in the solution
k_2	[g mg^{-1} min^{-1}]	—	pseudo-second-order rate constant
M	[g]	—	mass of the adsorbent
$q_{e,cal}$	[mg g^{-1}]	—	calculated values of the equilibrium adsorbate concentration in solid phase
$\overline{q_{e,cal}}$	[mg g^{-1}]	—	average of $q_{e,cal}$
q_e	[mg g^{-1}]	—	amount of dye adsorbed at equilibrium
$qe, meas$	[mg g^{-1}]	—	measured values of the equilibrium adsorbate concentration in solid phase
q_t	[mg g^{-1}]	—	amount of dye adsorbed at time t
r^2	–	—	coefficient of determination
V	[L]	—	volume of the solution
χ^2	–	—	χ^2

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