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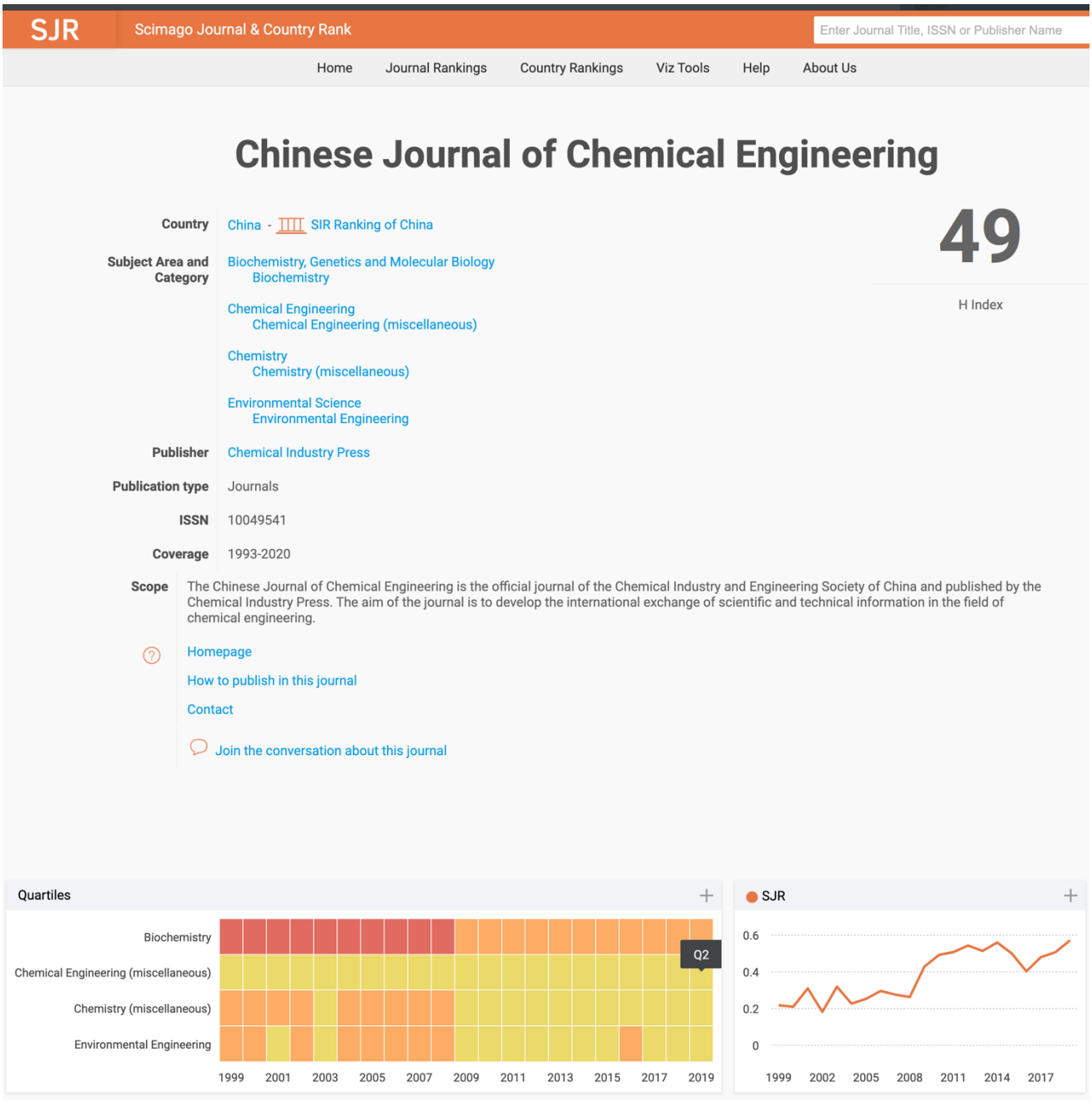

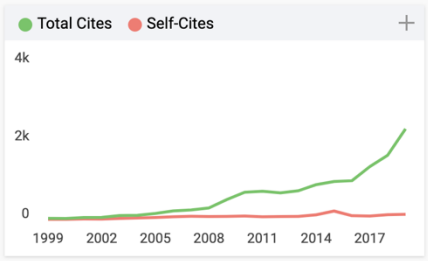

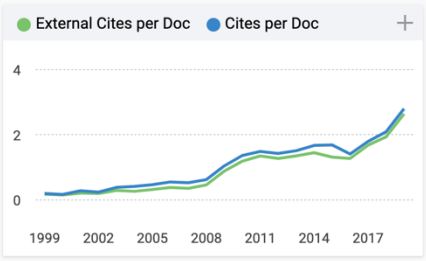

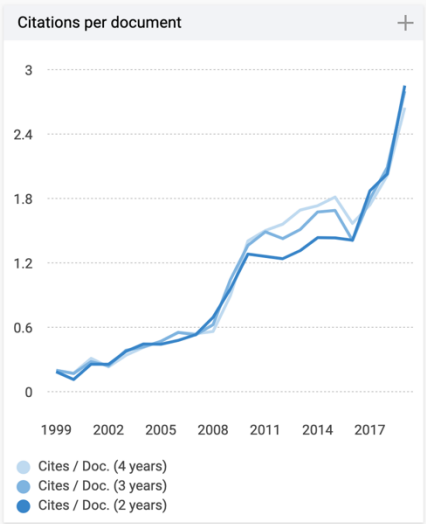

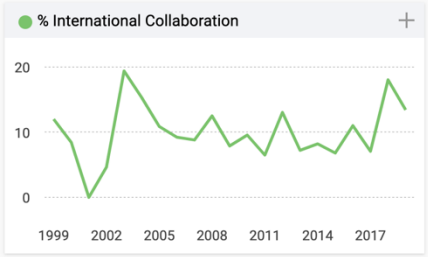

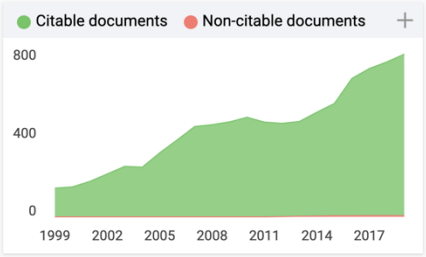

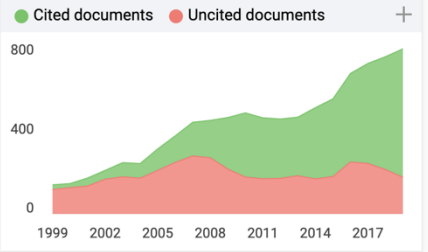

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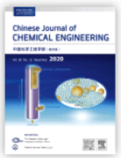

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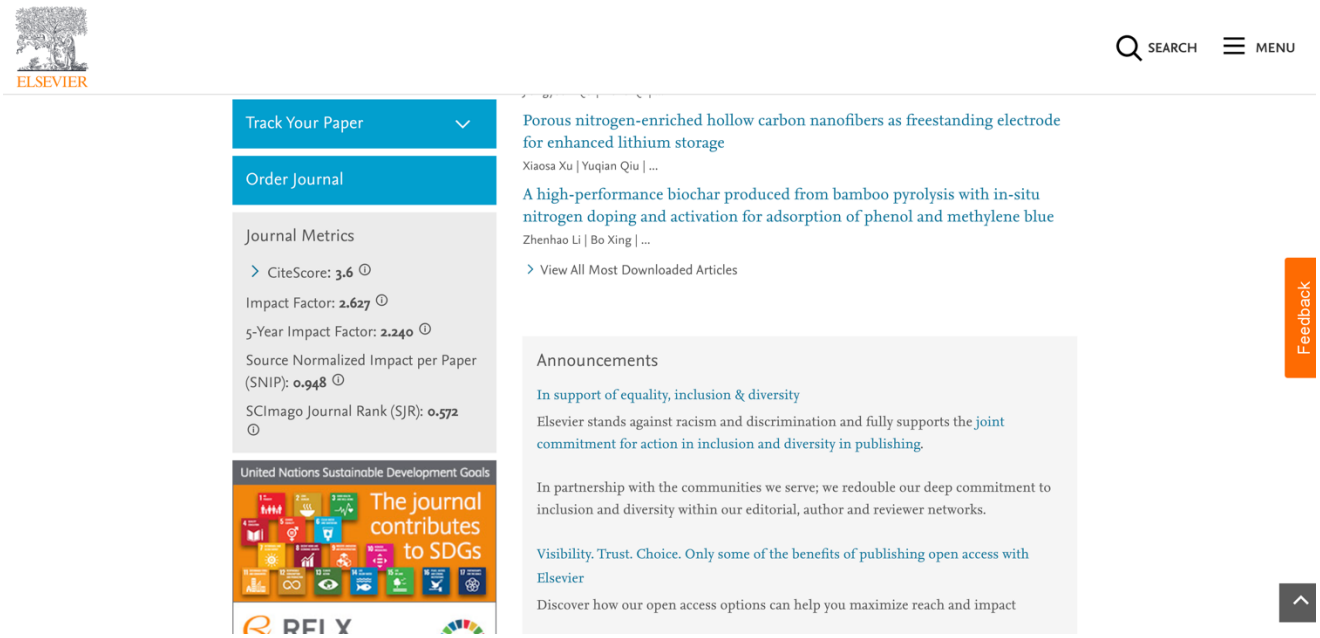

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This paper reports the potential of spent tea leaves (STL) treated with polyethyleneimine (PEI) as adsorbent of Reactive Black 5 (RB5) and Methyl Orange (MO) (anionic dyes) from simulated wastewater. STL surface was modified by attachment of PEI surfactant via simple step, and the process reduced the STL surface area. Adsorption capacities of 71.9 mg/g (RB5) and 62.11 mg/g (MO) on PEI-STL were recorded. The adsorption performance of PEI-STL increased with contact time until 100 min, when it became nearly constant. Increasing pH resulted in lower adsorption performance of PEI-STL towards RB5 and MO. A positive correlation between adsorbent dosage and adsorption performance of PEI-STL was also observed. Higher temperature promoted the adsorption performance of RB5 dyes, but reduced the adsorption performance of MO. This study demonstrates the superior property of PEI-STL in adsorption of RB5 and MO dyes without carbonization and activation.

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Adsorption of Anionic Dyes on Spent Tea Leaves Modified with Polyethyleneimine (PEI-STL)

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Abstract

This paper reports the potential of spent tea leaves (STL) treated with polyethyleneimine (PEI) as adsorbent of Reactive Black 5 (RB5) and Methyl Orange (MO) (anionic dyes) from simulated wastewater. STL surface was modified by attachment of PEI surfactant via simple step, and the process reduced the STL surface area. Adsorption capacities of 71.9 mg/g (RB5) and 62.11 mg/g (MO) on PEI-STL were recorded. The adsorption performance of PEI-STL increased with contact time until 100 min, when it became nearly constant. Increasing pH resulted in lower adsorption performance of PEI-STL towards RB5 and MO. A positive correlation between adsorbent dosage and adsorption performance of PEI-STL was also observed. Higher temperature promoted the adsorption performance of RB5 dyes, but reduced the adsorption performance of MO. This study demonstrates the superior property of PEI-STL in adsorption of RB5 and MO dyes without carbonization and activation.

Keyword: Adsorption, Spent Tea Leaves, Polyethyleneimine, Reactive Black 5, Methyl Orange

1.0 Introduction

Water is vital for all aspects of life. Unfortunately, the availability of clean and fresh water is critical, due to urbanization and industrialization. Among all industrial sectors, the textile industries are major the water consumer for fabric processing purpose. It was estimated that ~12% of the synthetic dyes used in the fabric processing are lost, and one fifth of the lost dyes find their ways to natural water bodies [1], causing significant threats to the environment and ecosystem, which in turn affect human wellbeing. Dye molecules are resistant to biodegradation and photo-degradation owing to the complex and large chemical structures, therefore conventional wastewater treatment which relies on aerobic biodegradation is less effective on dyes removal from wastewater [2]. Thus, appropriate treatments on dyes-bearing effluents are required prior to the effluent discharge into water bodies [3]. Among several possible methods to remove contaminants from textile wastewater, adsorption is more viable than others due to process simplicity and cost-effectiveness [4].

Therefore, main research focus in wastewater treatment by adsorption is to replace costly commercial activated carbon with cheap adsorbents with satisfactory adsorption performance. To date, a great number of reports were published on synthesis of activated carbons from various biomass waste [5, 6], as well as carbon-rich waste materials [7, 8]. Such adsorbents are considered cheap and renewable, and conversion of these wastes into activated carbons (ACs) serves as a solution to waste management. Another research direction lies in the modification of biomass precursor surface chemistry with surfactants which results in satisfactory pollutants adsorption without application of high energy and chemicals input as in preparation of ACs (carbonization, activation, washing etc). As pointed out by Alhashimi *et al.* [9], proper modification of surface functional group on adsorbent can have equal importance on adsorption performance. One of the widely studied surfactants, polyethyleneimine (PEI), is known to contain a great number of nitrogen-containing functional groups (i.e.–NH and $-NH_2$ groups) [10], thus it is an ideal adsorbent of anionic pollutants through electrostatic interaction. Nevertheless, PEI is soluble in water, hence a solid support is required for attachment of PEI to enable the adsorption property. Various adsorbents were synthesized from organic molecules modified with PEI, including magnetic nanoparticles Fe3O4@catechol/PEI polymer [11], graphene oxide/PEI hydrogel [12] with satisfactory performance in adsorption of anionic dyes. Despite the low cost of biowaste as solid support for PEI, only few reports were reported on the synthesis of such materials.

Sajab *et al.* [13] reported high adsorption capacity of phenol red by PEI modified oil palm empty fruit bunch (171 mg/g). Another research group by Sadaf *et al.* [14] successfully synthesized adsorbent based on treatment of sugarcane bagasse with PEI, with appreciable removal of direct yellow 50 dye (10.4 mg/g). In another work, Deng and Ting (2005) successfully removed Cr (VI) anions from solution by PEI-modified fungal biomass as biosorbent, and electrostatic interaction between adsorbent and adsorbate was the expected driving force in this adsorption processes [15].

However, up to date, there is no investigation on the potential of STL modified with cationic surfactant, polyethyleneimine (PEI) in removal of cationic yes. Application of spent tea leaves (STL) as an adsorbent precursor is widely reported, due to the popularity of tea as beverage all around the globe [16]. In view of the abundance of STL, it is important to study the potential of PEI-modified STL (PEI-STL) in removal of cationic dyes. The paper reports the investigation on the effects of several parameters, namely contact time, pH, temperature, adsorbent dosage and initial dye concentration, on removal efficiencies of RB5 and MO (both anionic dyes) from simulated wastewater onto PEI-STL, followed by isothermal and kinetic study on the adsorption behaviour. The changes on adsorbents' textural properties and surface functional groups after dyes adsorption were also studied.

2.0 Methodology

2.1 Materials

STL contained in teabags were collected from local households in Johor, Malaysia. The leaves were extracted from the teabags and cleaned by boiling with water until the filtrate turned colourless. After that, the washed STL were then dried in an oven, then ground and sieved to obtain particles in uniform size of ~500 µm. Surface modification was performed on STL according to the procedure described by Deng *et al.* [17]. STL were treated with PEI solution (5% w/v) at temperature 65 ˚C for 6 hours in a water bath, followed by washings and filtrations. Finally, the mixture was then dried for 24 hours at 50 ˚C. The treated sample is termed as PEI-STL in this manuscript. Three PEI-STL samples with different ratios between PEI and STL (2:1, 1:1, and 1:2) were prepared for initial screening purpose. Modified STL before and after adsorption experiments were characterized using the Fourier Transform Infrared (FTIR) Spectrophotometer (Thermo Fisher Scientific, model Nicolet IS5) with Attenuated Total Reflectance (ATR) technique. Small amount of sample was placed on the sample holder for FTIR scanning from 400-4000 cm^{-1}. The data were

analysed using OMNIC 8.3.103 software. The specific surface areas of the adsorbents were quantified using NOVA Surface Area Analyser (model 2200, Quantachrome Instruments).

RB5 and MO dyes in powder forms were purchased from Sigma-Aldrich (M) Sdn Bhd. Analyses of the stock dye solutions (by dissolution of dye powder in distilled water) with the UV-VIS spectrophotometer revealed characteristic peaks at 597 nm for RB5 and 506 nm for MO, therefore the absorbance values at these wavelengths were used to produce calibration curves for each dye as shown in Fig. 1. The concentration of remaining dyes in the solution after adsorption study was measured using UV-Vis Spectrophotometer (Aquamate, v4.60) at 597 nm (for RB5) and 506 nm (for MO). The values of initial and final concentrations of RB5 and MO dyes before and after adsorption were determined using standard calibration curves of the respective solutions (Fig. 1). The determined values were used to calculate the percentage removal of RB5 and MO according to Eq. (1) [18]. The amount of RB5 and MO adsorbed at equilibrium, q_e (mg/g), were calculated using Eq. (2).

$$\text{Percentage removal (\%)} = \frac{C_o - C_e}{C_e} \times 100 \quad (1)$$

$$q_e = \frac{(C_o - C_e)\ V}{W} \quad (2)$$

Where,

q_e : amount of dyes adsorbed on the sample at equilibrium

C_o : initial concentration of RB5 and MO (before adsorption) (mg/L)

C_e : final concentration of RB5 and MO (after adsorption) (mg/L)

V : dye solution volume (L)

W : adsorbent mass (g)

Fig. 1. Calibration curves that relate absorbance to concentrations of (a) RB5 and (b) MB dye solutions.

2.2 Adsorption Study

The adsorption study was conducted to analyse the effects of contact time (5-200 min), temperature (35-60 °C), adsorbent dosage (0.01-0.15g), pH (7-11), and initial dye concentration (50-100 mg/L) on removal of RB5 and MO dyes by PEI-STL. The procedure of batch adsorption study was adopted from the steps described by Su *et al.* [19] with several adaptations. The batch test was performed in 250 mL Schott bottles, each holding 50 mL of dye solution mixed with predetermined amount of adsorbent. The bottles were shaken at 190 rpm using a mechanical shaker at room temperature for a specific period. After that, the solutions in the Schott bottles were filtered (Whatman filter paper, 125mm) to remove the adsorbents from the solution, and the remaining solution was analysed using the UV-Vis to determine the amount of residual dyes. Sodium hydroxide (0.1M) and hydrochloric acid (0.1M) were used to modify the pH values of samples in this study.

3.0 Results and Discussion

3.1 Characterizations of Adsorbents

3.1.1 FTIR Analysis

The surface chemistry of PEI-STL was checked via FTIR analysis. Fig. 2 represents the adsorption spectra of raw STL, PEI-STL before adsorption while Fig. 3 represents the transmittance spectra of PEI-STL before adsorption and after adsorption of RB5 and MO, respectively. The spectra of four samples (raw STL, PEI modified STL before and after adsorption of RB5 and MO) revealed the presence of several peaks related to different functional groups on the adsorbent surface. Shifting and disappearance of these peaks after adsorption are observed, together with formation of new peaks. In both figures, the samples show a stretch bandwidth around 3600-3200 cm^{-1} signifying the presence of free –OH overlapped with N-H groups even when located at different bandwidth [20]. After modification of raw STL with PEI, intensity of the mentioned band was reduced due to attachment of PEI amine groups on STL surface, as well as formation of hydroxyl groups via crosslinking process [17]. The appearance of strong and sharp peaks in 2919-2851 cm^{-1} indicate C-H stretching of lignocellulosic components present in alkyl groups such as in methyl and methylene groups [21, 22]. However, the reduction in the intensity of peak for PEI-STL sample when compared with raw STL suggests the presence of abundant PEI molecules on the surface of raw STL [23]. The characteristic peaks in the range of 2361-2320 cm^{-1} indicate C-H stretching for all the samples [21]. The appearance of the band at 1733-

1728 cm^{-1}, reveals absorptions that may be ascribed to olefinic C=C vibrations in the aromatic region for the spent tea similar with the results obtained by Pretsch *et al.* [24]. The adsorption located around 1650-1621 cm^{-1} indicates the C=O stretch non-conjugated ketones, carbonyls and in ester groups [20, 25]. The imine group formed in the crosslinking reaction has the characteristic peak at around 1645-1630 cm^{-1} [26], showing a strong peak at 1632 cm^{-1}. The bands that appeared around 1521-1423 cm^{-1} are due to the N-O stretching [27]. According to Pretsch *et al.* [24], the broad band at 1045-1001 cm^{-1} can be attributed to some molecules containing sulphur/oxygen (S=O) bonds.

Fig. 2. FTIR spectra of (a) raw STL and (b) PEI-STL before adsorption

Fig. 3. FTIR spectra of (a) PEI-STL before adsorption; (b) PEI-STL with RB5 and (c) PEI-STL with MO

PEI contains large amount of–NH and $-NH_2$ groups. Fig. 3(a) illustrates the intensity reduction for peaks at 3600 – 3200 cm^{-1}, indicating involvement of N-H bonds in adsorption of RB5 and MO. Meanwhile, C-H group at position 2321 cm^{-1} is more intense than PEI-STL before adsorption (Fig. 3(b)). PEI-STL after RB5 adsorption had its N-O asymmetric stretch situated at 1516 cm^{-1} slightly changed which involved in adsorption. The peak at 1037 cm^{-1} is shifted from 1031 cm^{-1} due to the reduction of S=O indicating RB5 adsorption takes place. Overall, the band intensities for RB5 decreased after the adsorption process indicating RB5 molecules being adsorbed by PEI-STL.

As seen in Fig. 3(c), the intensity of the peak which assigned to the C=O group appears at 1633 cm^{-1} reduced indicating the participation of PEI-STL surface functional groups in the adsorption reaction. After MO uptake, the absorption band at 1517 cm^{-1} represents N-O stretching vibrations shifting to 1517 cm^{-1}. The peak at 1030 cm^{-1} is attributed to S=O stretching became lower compared to PEI-STL and this indicates the SO_3^- is involved in MO adsorption. These changes confirmed the participation of PEI-STL surface functional groups with group of MO molecules. The assignments of each peak for all samples are summarized in Table 1.

Table 1. Spectra of basic structure for spent tea using FTIR

Wavelength (cm^{-1})	Functional groups

STL	PEI-STL before adsorption	STL-RB5	STL-MO	
3333	3290	3293	3307	-OH stretch overlapped N-H stretch [20]
2919	2918	2919	2919	Aliphatic C-H stretching [21, 22]
2851	2851	2850	2851	
2361	2321	2321	2350	C-H stretching [21]
1732	1732	1732	1733	C=C vibration [24]
1624	1633	1630	1629	C=O stretching [20, 25]
1516	1517	1516	1517	asymmetric N-O stretching [27]
1028	1031	1037	1030	S=O stretching [24]

3.1.2 Surface Area

Table 2 shows the specific surface area of the prepared adsorbent determined by BET surface area analyser. After modification with PEI, the surface area of STL slightly decreased from 3.23 m^2/g to 2.33 m^2/g which may be ascribed to the introduction of PEI into spent tea surface area was successful. After the removal of RB5 and MO from the aqueous solution, the surface areas become 0.90 m^2/g and 1.98 m^2/g, respectively. This observation verified the adsorption of RB 5 and MO onto PEI-STL.

Table 2. The surface area for raw ST, PEI-STL and PEI-STL after adsorption

	Adsorbent samples	Surface area (m^2/g)
Present study	Raw STL	3.23
	PEI-STL (before adsorption)	2.33
	STL-RB5 (after RB5 adsorption)	0.90
	STL-MO (after adsorption)	1.98
Bajpai and Jain (2012)	Raw STL	1.41

3.2 Effects of Reaction Parameters on dyes adsorption

3.2.1 Effect of Contact Time

Fig. 4 shows the effect of contact time (5 -200 min) on the percentage removal of both anionic dyes onto PEI-STL. It may be observed from the figure that the maximum percentage

removal taking place within 120 min and became gradual thereafter. High adsorption rate was observed at initial stage, and equilibrium state was achieved around 120 minutes. For RB5, optimum adsorption performance was attained at 100 min with 99% removal. Whereas, for MO the percentage removal also increased with time with the maximum percentage of 88.1% at equilibrium after 120 min. Hamzeh *et al.* [28] had a similar observation while investigating the removal of RB5 and AO7 when canola stalks adsorbent was utilized. It is understood that during the initial adsorption stage, all vacant sites on the adsorption surface are available for attachment of dye molecules. As the process continues, the number of vacant sites decreases, and repulsion exists between the adsorbate molecules attached to the adsorbent and in bulk phase. The percentage removal by RB5 was found to be slightly more than MO. This situation is similar with the previous study by Oei *et al.* [29] who reported that different dye investigated gave different percentage removal using the same adsorbent. This trend was observed because different dyes in the case of RB5 and MO exhibit different structural characteristics. It is also based on assumptions in view of their mechanism where PEI-STL surfaces cannot admit MO molecules deeper as compared to RB5 molecules.

Fig. 4. Effect of contact time on the removal efficiency of RB5 and MO onto PEI-STL. (0.05 g/L of dye solution; 0.1 g of adsorbent; 120 min of reaction time; room temperature; pH 7)

3.2.2 Solution pH

The effect of pH (2-10) on the percentage removal of RB5 and MO is shown in Fig. 5. It is known that RB5 and MO dyes molecules form anions in aqueous solution. As charged species, the rates of adsorption of these dyes molecules are highly affected by solution pH depending on the adsorbent surface charge, degree of ionization and dissociation of functional groups of adsorbate species. As a result, the dye exhibited different process behaviour. A higher removal was found to be at pH 3 for both dyes, indicating strong dependency of adsorption performance on pH. The adsorption process showed no significant improvement beyond pH 7. At pH 2-3, presence of amine groups (as a result of protonation) led to positive charge on STL surface, thus producing significant electrostatic attraction with negatively charged anionic dyes. Thus, the optimum pH for RB5 and MO adsorption is pH3. Such result is supported by another adsorption study on RB5 and MO adsorption onto sunflower seeds [30] and cationic surfactant wheat straw [19], respectively.

Fig. 5. Effect of solution pH on the removal efficiency of RB5 and MO onto PEI-STL. (0.05 g/L of dye solution; 0.1 g of adsorbent; 120 min of reaction time; room temperature)

3.2.3 Effect of Adsorbent Dosage

Proper selection of initial adsorbent dosage is crucial because the available surface area and binding sites on its surfaces control adsorption efficiency [31]. The effect of PEI-STL dosage (0.01-0.15g) on the removal of RB5 and MO from the aqueous solution is shown in Fig. 6. The results revealed that the percentage removal increased with adsorbent dosage. The optimum amount of the adsorbent is 0.1 g and no significant improvement was observed beyond the optimum dosage. This is due to the fact that availability of more surface active sites at the PEI-STL surface increased resulting from the increased dosage [32]. The RB5 and MO percentage removal increased from 55.3% to 99.1% and from 45.5% to 88% respectively. Similar result was reported in adsorption of RB5 onto canola straw [28] and MO onto bottom ash [33].

Fig. 6. Effect of the adsorbent dosage on the removal efficiency of RB5 and MO onto PEI-STL. (0.05 g/L of dye solution; 120 min of reaction time; room temperature; pH 7)

3.2.4 Temperature

In an adsorption process, temperature affects the diffusion of dye molecules at dye external boundary layer interface, and also inside the adsorbent pores [34]. Fig. 7 illustrates the percentage removal of RB5 and MO onto PEI-STL in the range of 25-60 °C. RB5 adsorption exhibits a significant percentage removal even at low temperature which is 97.9%. The adsorption slightly increased with temperature to a small extent, suggesting the adsorption is an endothermic process. This observation is similar with the study on RB5 adsorption onto CPC modified barley straw [29]. For MO, the removal percentage decreased from 88.1% to 61% at elevated temperatures, thus the reaction is said to be exothermic. Such observation is related to the weakening of the physical bonding between dye molecules and the sorption sites of the PEI-STL at higher temperature. On the other hand the increased dye solubility leads to stronger interactions between dye molecules and solvent. Similar trend have been reported for the removal of MO by cationic surfactant-modified wheat straw [19].

Fig. 7. Effect of temperature on the removal efficiency of RB5 and MO onto PEI-STL. (0.05 g/L of dye solution; 0.1g of adsorbent; 120 min reaction time; pH 7)

3.2.5 Initial Dye Concentration

The effect of RB5 and MO initial concentration on the adsorption (50-100 mg/L) onto PEI-STL is presented in Fig. 8. It can be seen that the percentage removal of RB5 (from 98.7% to 43.5%) and MO (from 88.7% to 32.7%) decreased when higher initial dye concentration was used. Increasing initial concentration of RB5 and MO enhances the driving force of film mass transfer thereby accelerating the movement of dye molecules from bulk solution to film zone near PEI-STL surface. At the early stage of adsorption, where concentration was low, the removal was rapid due to the higher number of unoccupied sites available. In contrast, at higher initial concentration of dyes, the available sites became saturated. Therefore, there are limited binding sites for dyes molecules, and some remained in the solution without being adsorbed. These are consistent with studies reported by RB5 removal by CPC modified barley straw [29] and MO adsorption onto de-oiled soya adsorbent [33].

Fig. 8. Effect of the initial dyes concentration on the removal efficiency of RB5 and MO onto PEI-STL. (0.1g of adsorbent; 120 min of reaction time; room temperature; pH 7)

3.3 Process Modelling

3.3.1 Adsorption Isotherm

The adsorption isotherm is the basic requirement in designing the adsorption system [35], as it indicates the characteristics of the adsorbate layer/s on the adsorbent surface at adsorption equilibrium. Isotherms help in providing information about the optimum adsorbent dosage in the adsorption process. For further interpretation on adsorption behaviours of investigated dyes RB5 and MO, the experimental adsorption data is fitted to the Langmuir and Freundlich isotherm models, which are the most widely models in adsorption study.

Figs. 9 and 10 represent the linearized Langmuir and Freundlich isotherm plots for RB5 and MO adsorption onto PEI-STL. The adsorption nature for both investigated dyes is determined by comparing the R^2 of the Langmuir and Freundlich isotherms. Experimental data of PEI-STL gave the best fitting to the Langmuir model as verified by the higher correlation coefficient, R^2 value, for RB5 (0.9505) and MO (0.9271) compared to R^2 value obtained from Freundlich isotherm model. The isotherm constants and correlation coefficients were calculated and represented in Table 3.

PEI-STL adsorption had equal activation energy for both dye molecules and this indicates the homogenous nature of the adsorbent surface. This result also implies the presence of monolayer coverage of RB5 and MO dye molecules on the outer surface of the PEI-STL. The value of R_L was calculated in order to evaluate whether the adsorption is favourable. Calculated R_L is found to be 0.2273 (RB5) and 0.258 (MO), respectively, therefore PEI-STL is favourable for adsorption RB5 and MO.

Table 3. The adsorption isotherm parameters for RB5 and MO

Adsorption Isotherm Models	**Parameter Value**	
	RB5	**MO**
Langmuir		
q_m (mg/g)	71.94	62.11
K_L (L/mg)	0.0680	0.0576
R^2	0.9505	0.9271
Freundlich		
K_F (mg/g)	1.569	1.507
n	1.569	1.081
R^2	0.8519	0.8983

Fig. 9. Linear plots of the Langmuir isotherm for adsorption of (a) RB5 and (b) MO

Fig. 10. Linear plots of the Freundlich isotherm for adsorption of (a) RB5 and (b) MO

3.3.2 Adsorption Kinetics Study

Kinetic studies are carried out to better understand the mechanisms of adsorption process. The pseudo-first order and pseudo-second order kinetic models were employed to analyse the RB5 and MO dye adsorption mechanism in this study. The adsorption data is fitted to the mentioned models in order to select the best model to describe the adsorption kinetic. The graphs of pseudo-first order and pseudo-second order equations for the PEI-STL are shown in Figs. 11 and 12. The plotted graph reveals that the pseudo-first order did not fit well as compared to the pseudo-second order.

Fig. 11. Pseudo-first order kinetics of (a) RB5 and (b) MO adsorption onto PEI-STL

Fig. 12. Pseudo-second order kinetics of (a) RB5 and (b) MO adsorption onto PEI-STL

Values of the predicted dye uptake capacity (q_e), rate constants along with correlation coefficients were analysed from the plot and are presented in Table 4. The high discrepancies between the calculated values from pseudo-first order and experimental values indicate the inadequacy of the model in description of the process. On the other hand, small differences are observed between the q_e values predicted from pseudo-second order model and values calculated based on experimental data. The small difference is most possibly due to the existence of boundary layer effects during the adsorption. This confirms the adequacy of the pseudo-second order model for the verification of RB5 and MO removal using PEI-STL, and the adsorption process is controlled by chemisorption.

Table 4. Adsorption kinetic of RB5 and MO using PEI- STL

Kinetic Models	**Value**	
	RB5	**MO**
q_e (exp)(mg/g)	45.04	44.78
Pseudo-first order		
K_1 (min^{-1})	0.0326	0.0359
q_e (calc) (mg/g)	35.37	33.15
R^2	0.9391	0.9264
Pseudo-second order		
K_2 (min^{-1})	0.0002	0.0001
q_e (calc) (mg/g)	42.19	43.64
R^2	0.9731	0.9609

4.0 Conclusion

This study explores the potential of adsorbent produced from STL impregnated with PEI in removal of RB5 and MO from simulated wastewater. Impregnation of PEI produced

positive charge on the adsorbent surfaces, resulting in strong attraction towards anions. The operating conditions varied were contact time, solution pH, adsorbent dosage, temperature and initial dye concentration. It was found for each controlled operating parameter; the surfactant modified spent tea exhibited higher adsorption towards RB5 than MO**.** The adsorption isotherms were also determined for the dyes adsorption. For both dyes tested, the equilibrium data agreed well with the Langmuir isotherm model with the maximum monolayer adsorption capacity for RB5 and MO were 71.94 and 62.11 mg/g, respectively. The kinetics of both dyes adsorptions on PEI-STL followed the pseudo-second-order model. This study demonstrates the potential of PEI-STL in dyes adsorption under laboratory conditions, and more studies are needed to investigate the prospect of such adsorbent in industrial textile effluent treatment.

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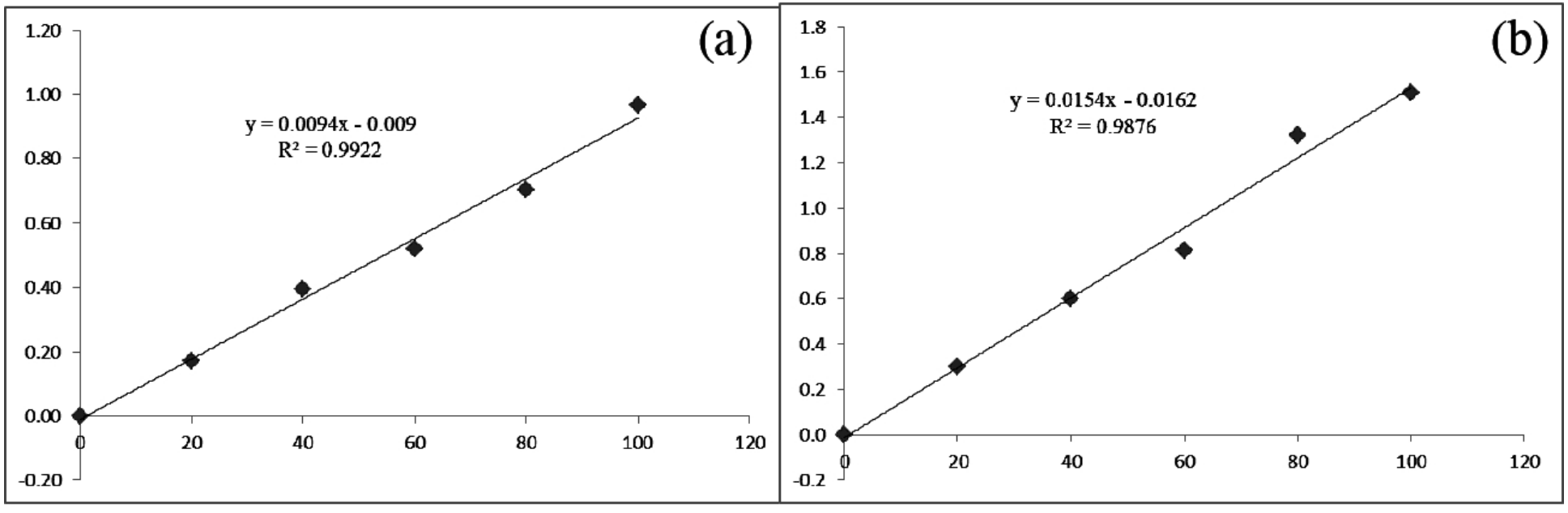
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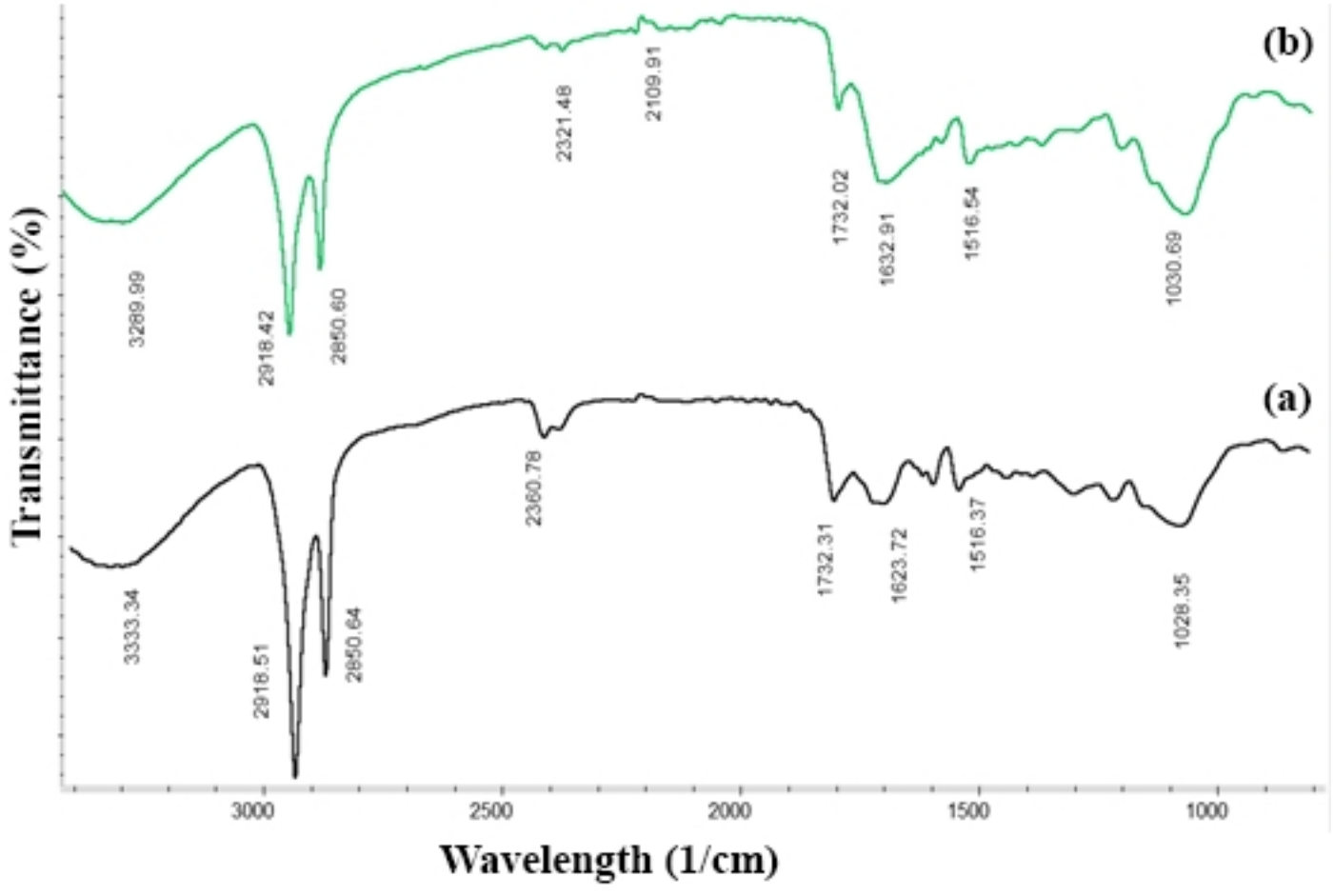
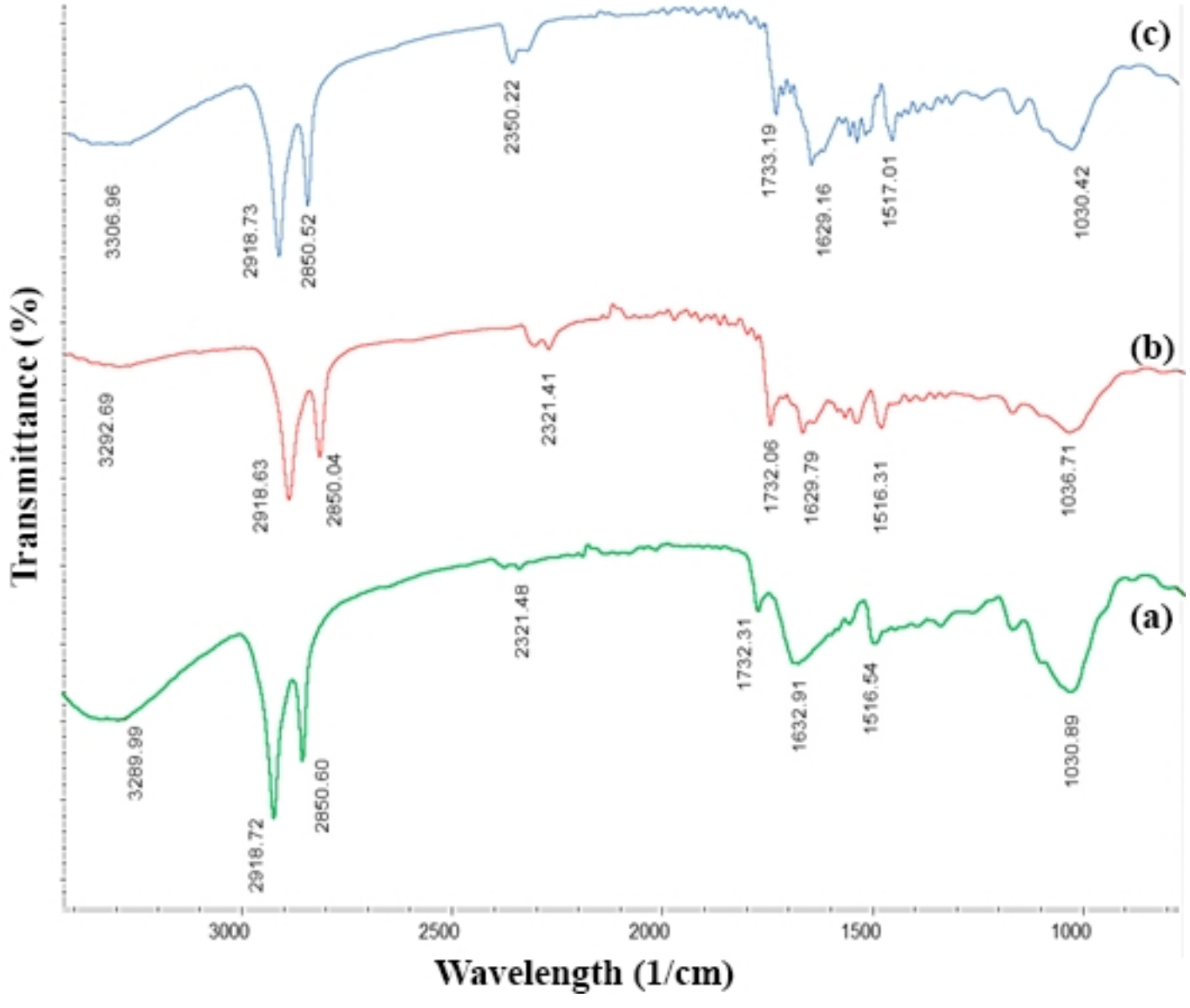
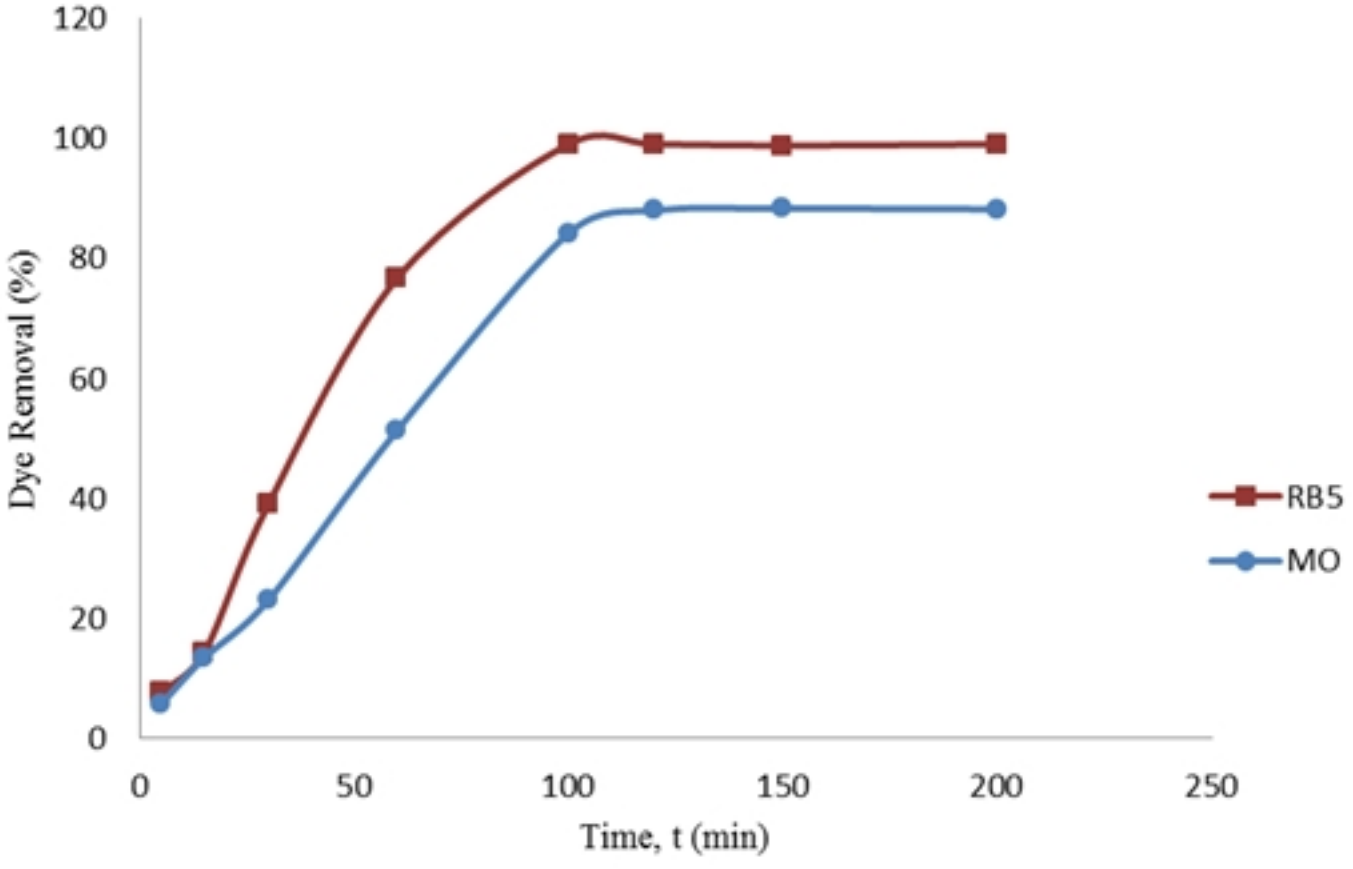
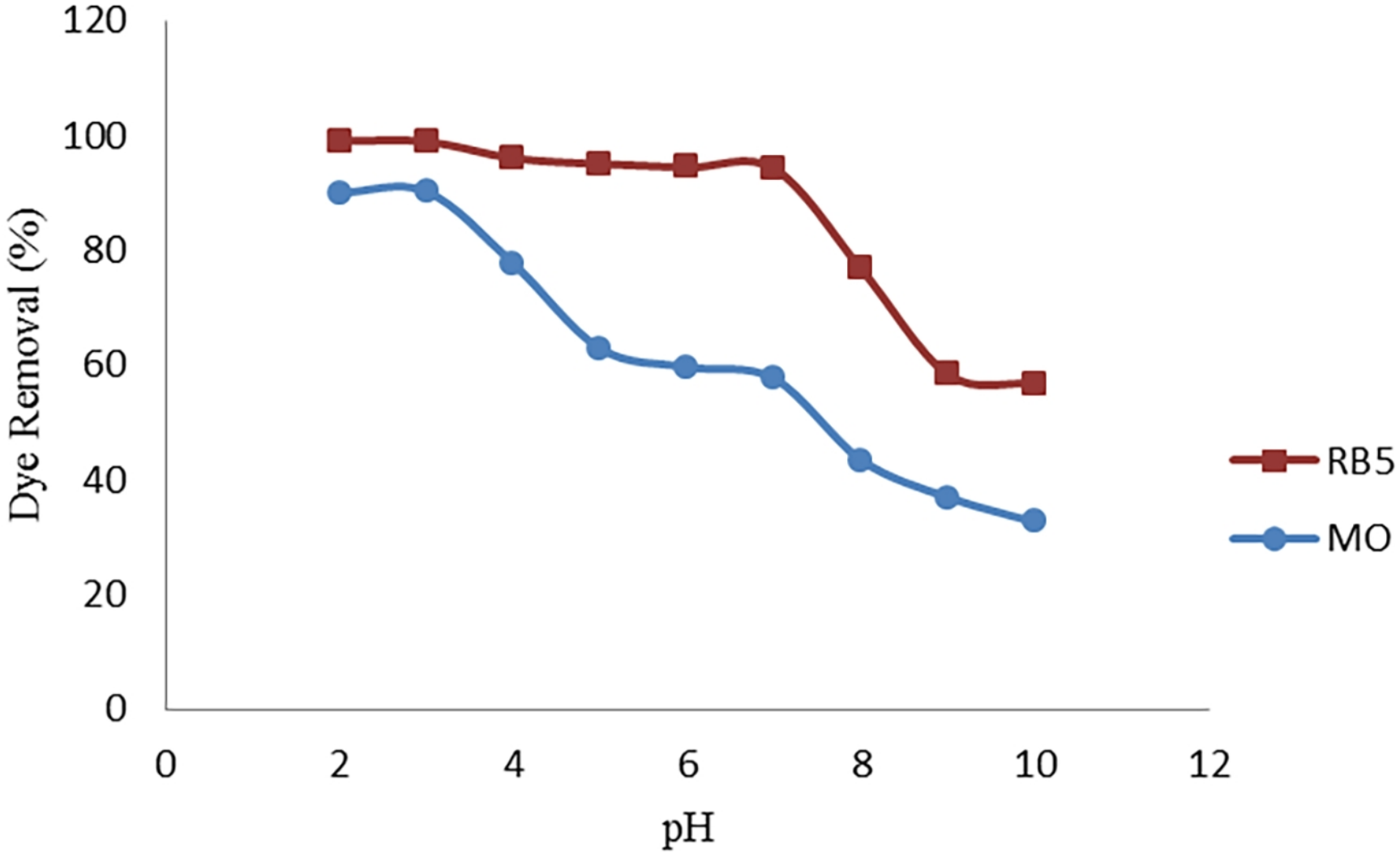
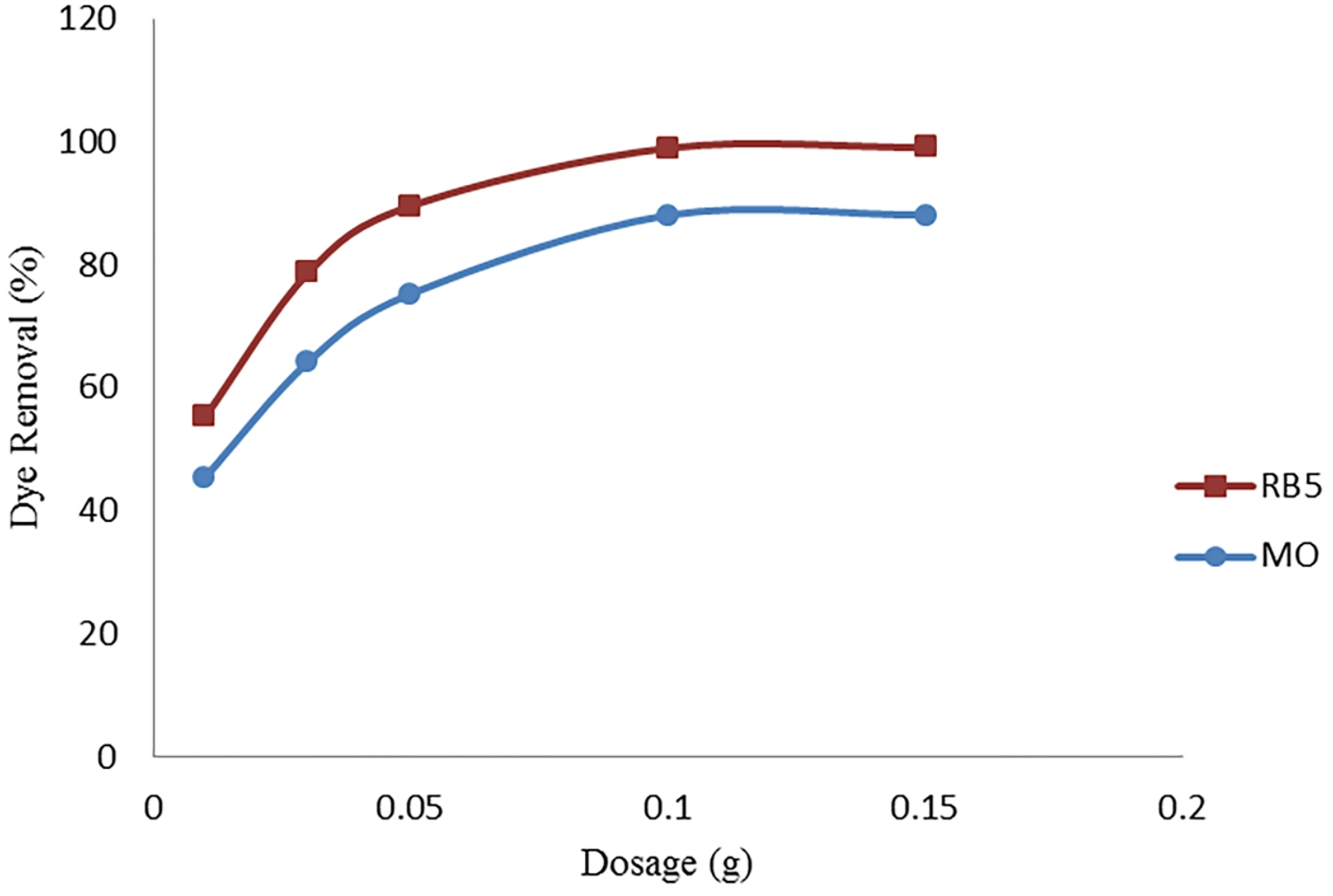
1.00
0.80
0.60
0.40
0.20
0.00
-0.20
0
20
40
60
80
100
120
(b)
y = 0.0154x - 0.0162
R² = 0.9876
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-0.2
0
20
40
60
80
100
120


(b)
3289.99
2918.42
2850.60
2321.48
2109.91
1732.02
1632.91
1516.54
1030.69
(a)
3333.34
2918.51
2850.64
2360.78
1732.31
1623.72
1516.37
1028.35
Transmittance (%)
3000
2500
2000
1500
1000
Wavelength (1/cm)


(c)
3306.96
2918.73
2850.52
2350.22
1733.19
1629.16
1517.01
1030.42
(b)
3292.69
2918.63
2850.04
2321.41
1732.06
1629.79
1516.31
1036.71
(a)
3289.99
2918.72
2850.60
2321.48
1732.31
1632.91
1516.54
1030.89
Transmittance (%)
3000
2500
2000
1500
1000
Wavelength (1/cm)


120
100
80
60
40
20
0
Dye Removal (%)
0
50
100
150
200
250
Time, t (min)
RB5
MO


120
100
80
60
40
20
0
Dye Removal (%)
0
2
4
6
8
10
12
pH
RB5
MO


120
100
80
60
40
20
0
Dye Removal (%)
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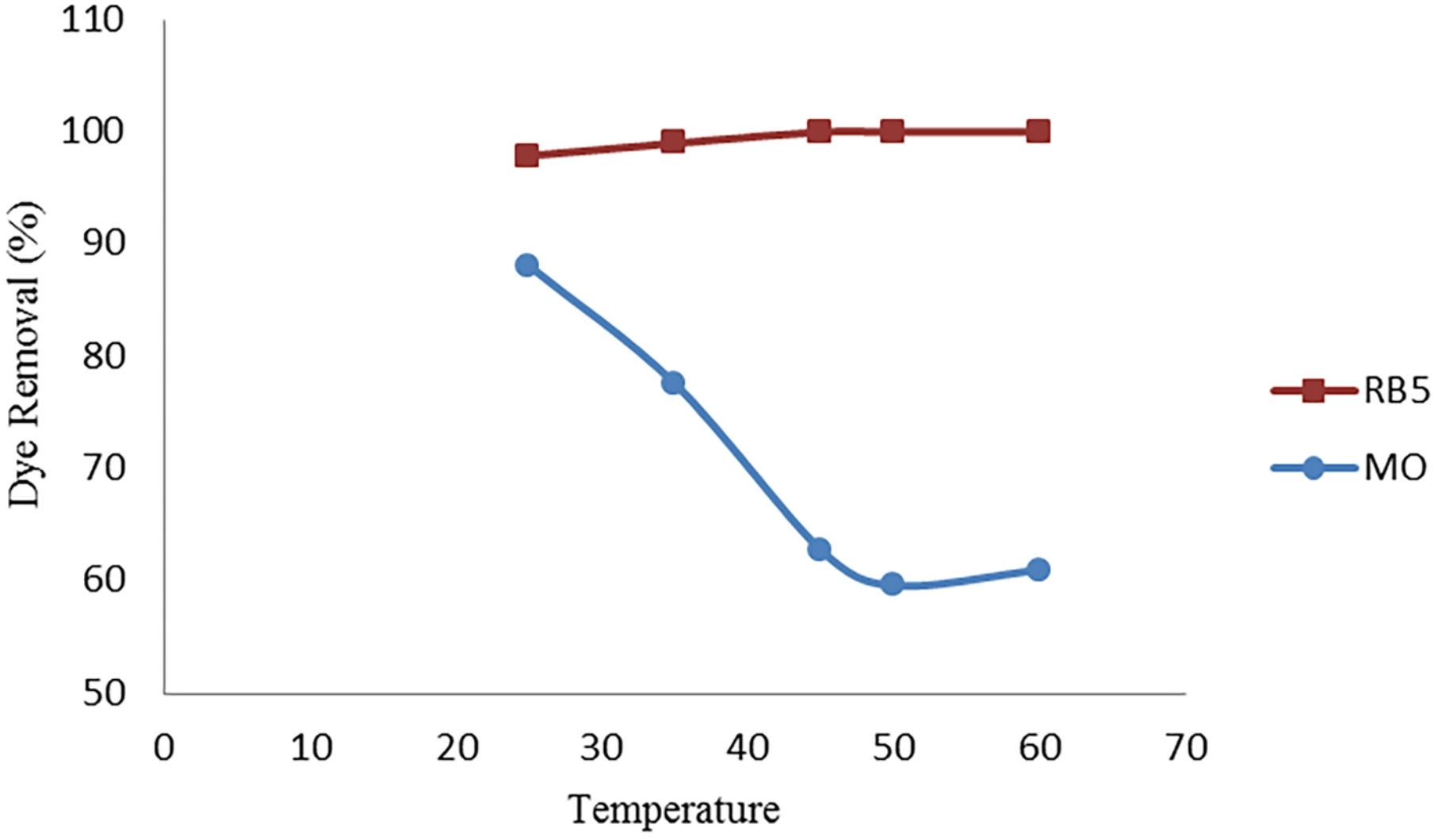
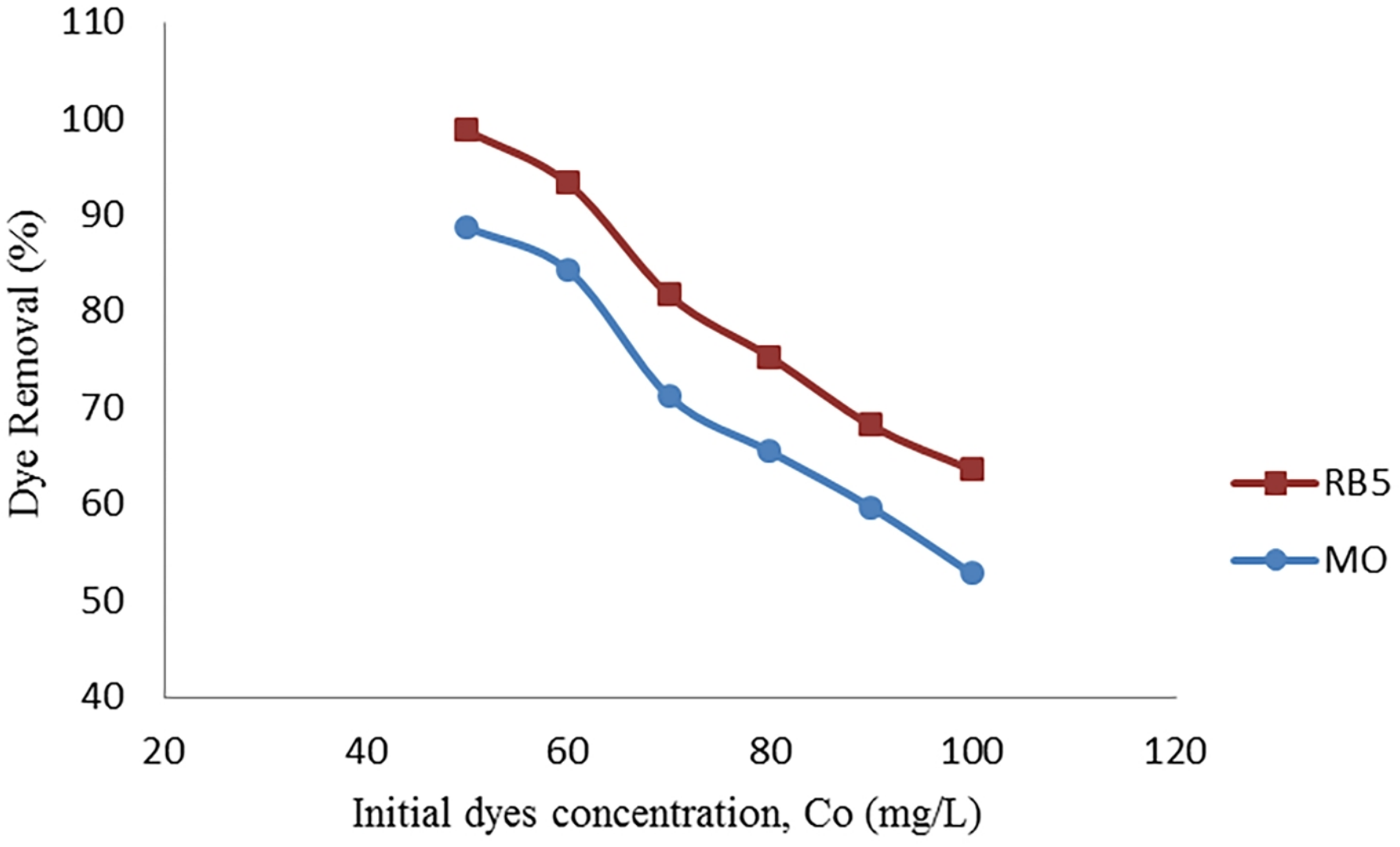
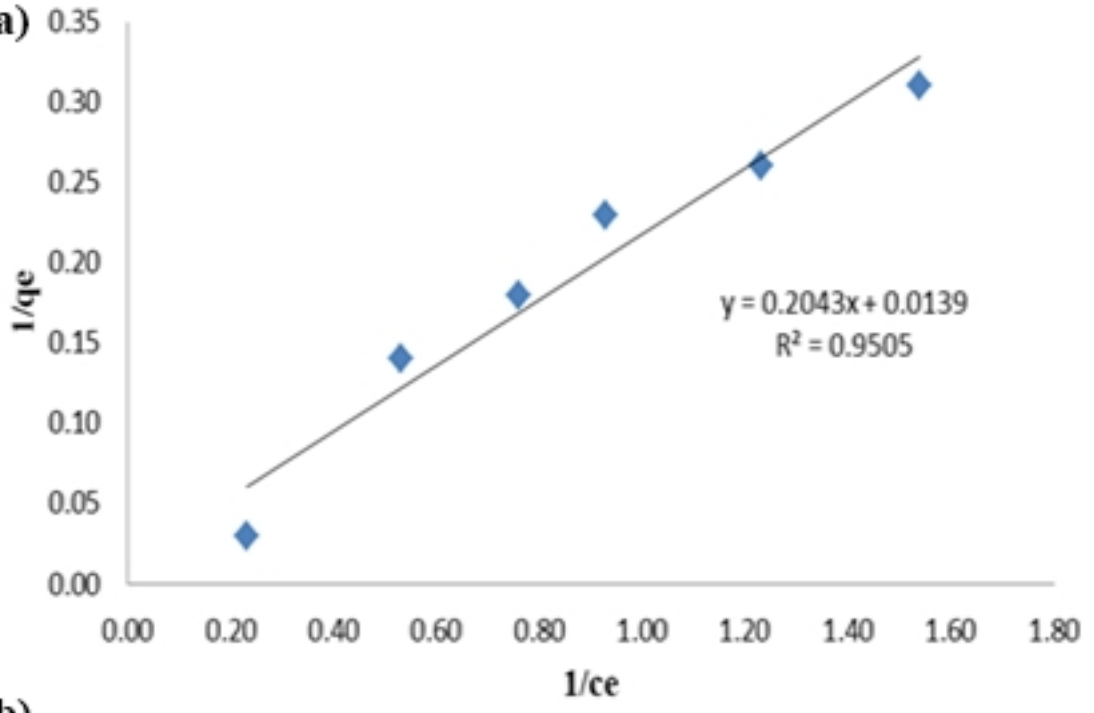
0.1
0.15
0.2
Dosage (g)
RB5
MO

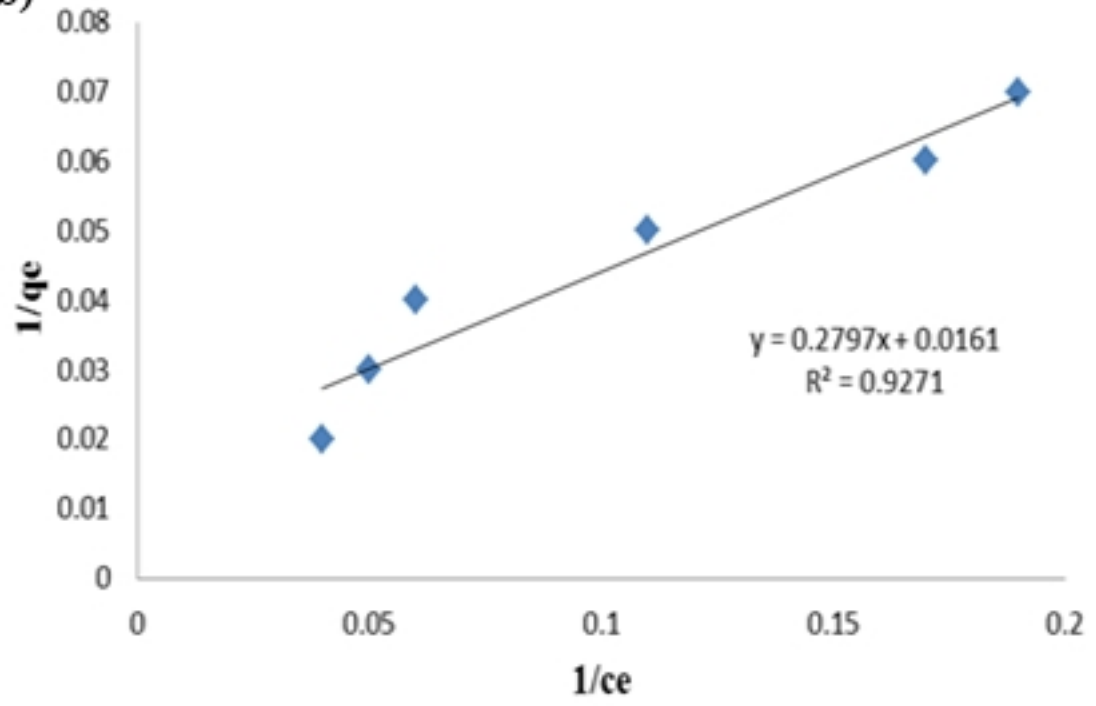

110
100
90
80
70
60
50
Dye Removal (%)
0
10
20
30
40
50
60
70
Temperature
RB5
MO


110
100
90
80
70
60
50
40
Dye Removal (%)
20
40
60
80
100
120
Initial dyes concentration, Co (mg/L)
RB5
MO

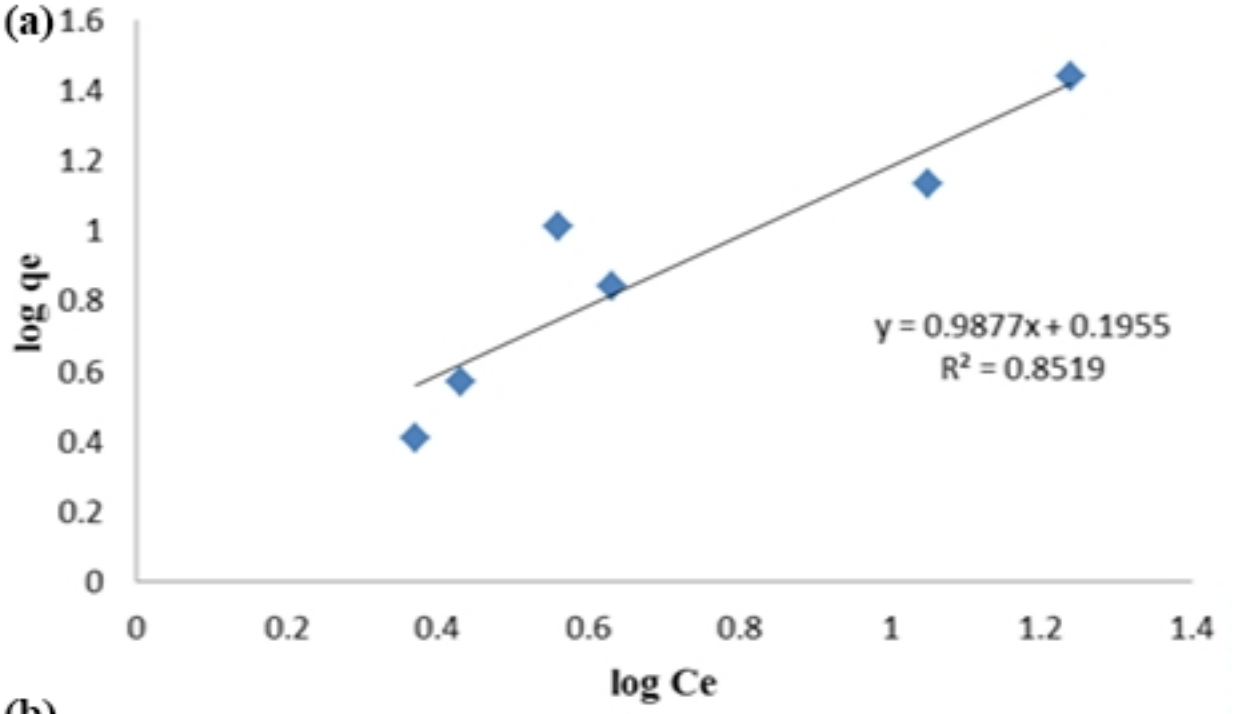
(a)


0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
1/qe
y = 0.2043x + 0.0139
R² = 0.9505
0.00
0.20
0.40
0.60
0.80
1.00
1.20
1.40
1.60
1.80
1/ce

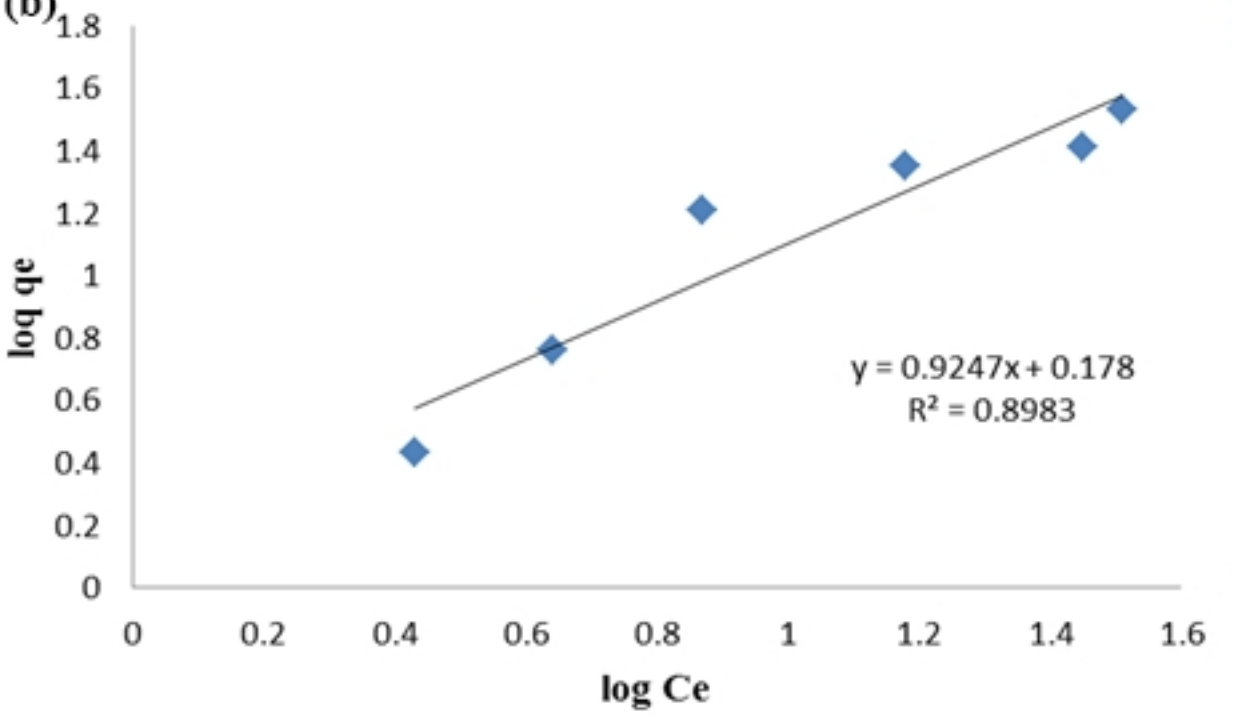
(b)

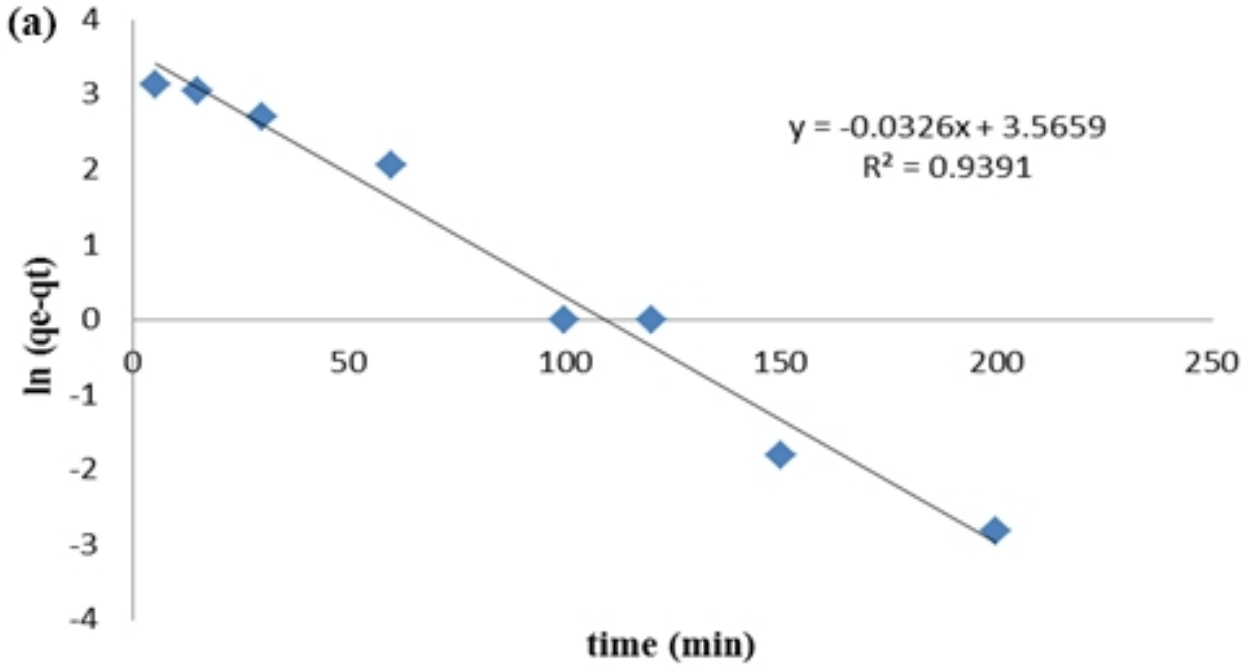

0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
1/qe
y = 0.2797x + 0.0161
R² = 0.9271
0
0.05
0.1
0.15
0.2
1/ce

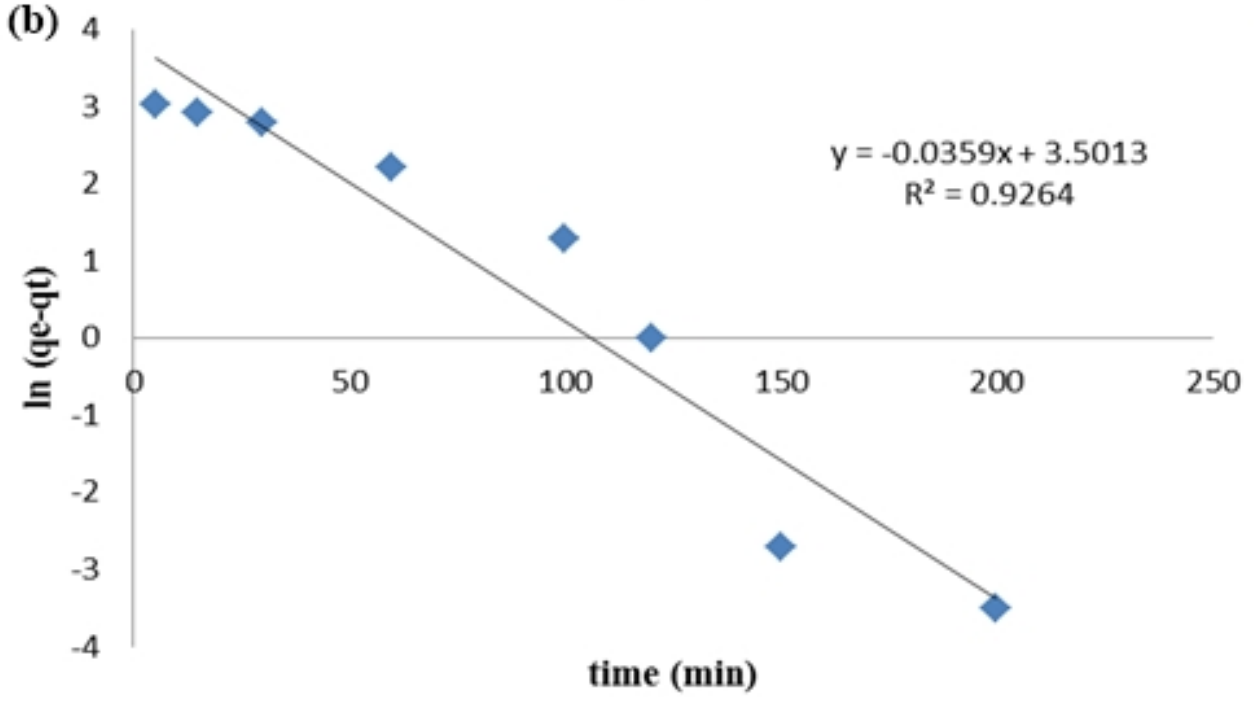
(a)


1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
log qe
0
0.2
0.4
0.6
0.8
1
1.2
1.4
log Ce
y = 0.9877x + 0.1955
R² = 0.8519

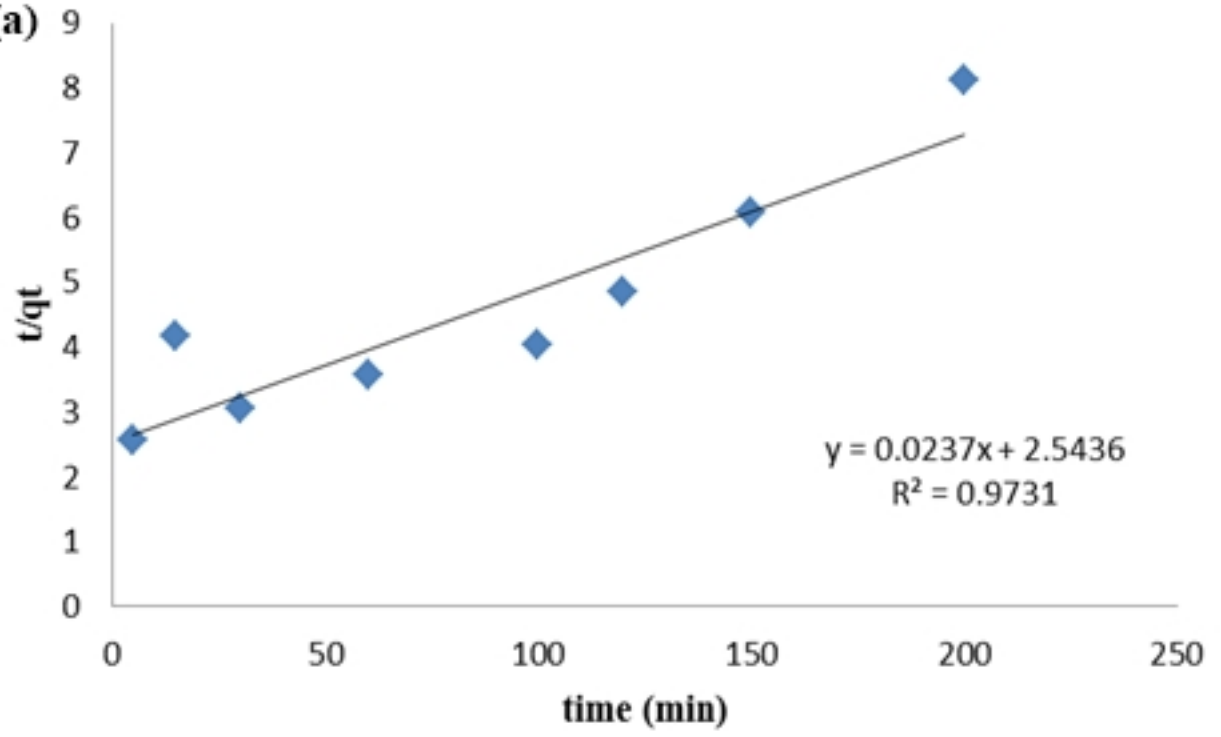
(b)


1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
log qe
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
log Ce
y = 0.9247x + 0.178
R² = 0.8983


(a)
y = -0.0326x + 3.5659
R² = 0.9391
ln (qe-qt)
time (min)
4
3
2
1
0
-1
-2
-3
-4
0
50
100
150
200
250


(b)
y = -0.0359x + 3.5013
R² = 0.9264
ln (qe-qt)
time (min)
4
3
2
1
0
-1
-2
-3
-4
0
50
100
150
200
250

(a)


t/qt
time (min)
y = 0.0237x + 2.5436
R² = 0.9731
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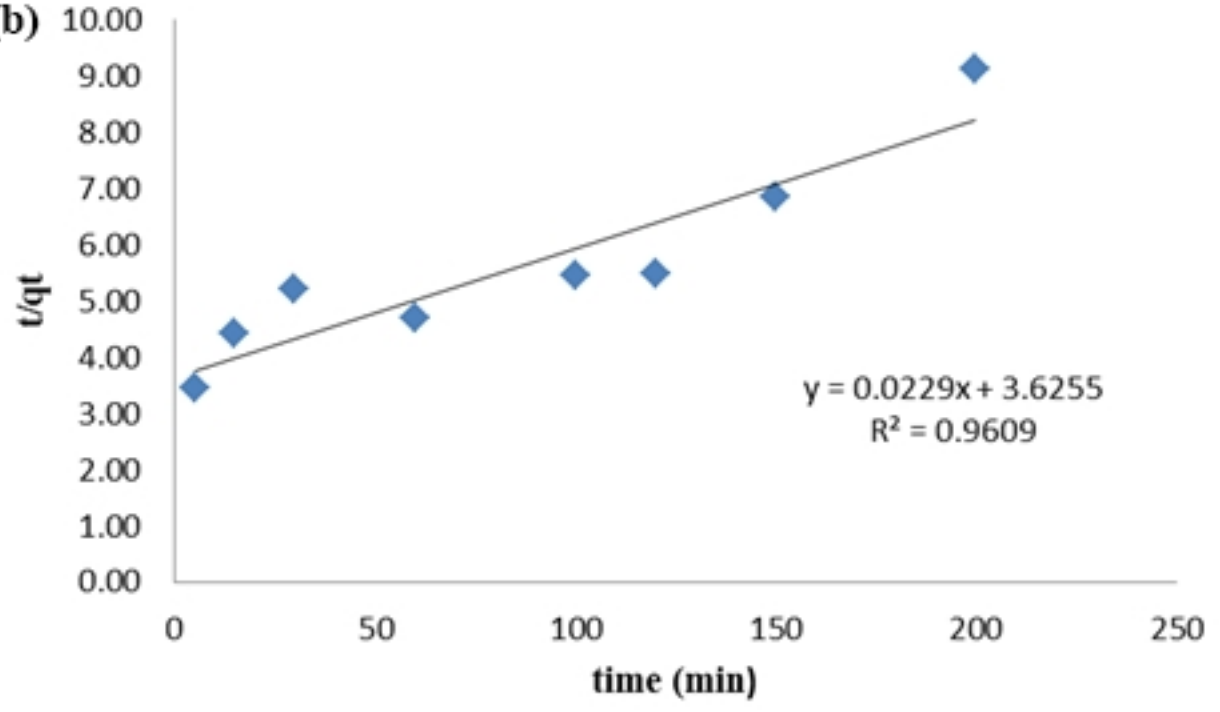
1
2
3
4
5
6
7
8
9
0
50
100
150
200
250

(b)


t/qt
time (min)
y = 0.0229x + 3.6255
R² = 0.9609
0.00
1.00
2.00
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00
0
50
100
150
200
250

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3. Bukti reminder to complete review

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Widi Astuti <widi_astuti@mail.unnes.ac.id>

Reminder to complete review for submission to Chinese Journal of Chemical Engineering

1 message

Chinese Journal of Chemical Engineering <EviseSupport@elsevier.com> Fri, Dec 1, 2017 at 9:19 PM
Reply-To: cjche@elsevier.com
To: widi_astuti@mail.unnes.ac.id

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Ref: CJCHE_2017_1493
Title: Adsorption of Anionic Dyes on Spent Tea Leaves Modified with Polyethyleneimine (PEI-STL)
Journal: Chinese Journal of Chemical Engineering
Corresponding Author: Corresponding Author: Norzita Ngadi
Co-authors: Co-authors: Hasnaa H. Tumari, Syieluing Wong, Onn Hassan

Dear Professor Astuti,

On 24/Nov/2017 you kindly agreed to review the above-referenced manuscript. This is a friendly reminder that your review of this manuscript is due by 08/Dec/2017.

As timely reviews are of utmost importance to authors, I would be grateful if you could submit your review as soon as possible via EVISE® at: http://www.evise.com/evise/faces/pages/navigation/NavController.jspx?JRNL_ACR=CJCHE.

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If your review will be delayed for any reason, please let me know when I can expect to receive your comments.

Kind regards,

Guohua Chen
Associate editor
Chinese Journal of Chemical Engineering

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4. Bukti thank you for reviewing manuscript

UNNES
UNIVERSITAS NEGERI SEMARANG

Widi Astuti <widi_astuti@mail.unnes.ac.id>

Thank you for reviewing manuscript Adsorption of Anionic Dyes on Spent Tea Leaves Modified with Polyethyleneimine (PEI-STL)

2 messages

Chinese Journal of Chemical Engineering <EviseSupport@elsevier.com> Mon, Dec 4, 2017 at 4:39 PM
Reply-To: cjche@elsevier.com
To: widi_astuti@mail.unnes.ac.id

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Ref: CJCHE_2017_1493
Title: Adsorption of Anionic Dyes on Spent Tea Leaves Modified with Polyethyleneimine (PEI-STL)
Journal: Chinese Journal of Chemical Engineering

Dear Professor Astuti,

Thank you for your review for the above-referenced manuscript. I greatly appreciate the commitment of your time and expertise. Without the dedication of reviewers like you, it would be impossible to manage an efficient peer review process and maintain the high standards necessary for a successful journal.

When a final decision has been reached regarding this manuscript you will be able to view this decision, as well as reviews submitted by any other reviewers, at: http://www.evise.com/evise/faces/pages/navigation/NavController.jspx?JRNL_ACR=CJCHE. You can also access your review comments here, at any time.

I hope that you will consider Chinese Journal of Chemical Engineering as a potential journal for your own publications in the future.

Kind regards,

Guohua Chen
Associate editor
Chinese Journal of Chemical Engineering

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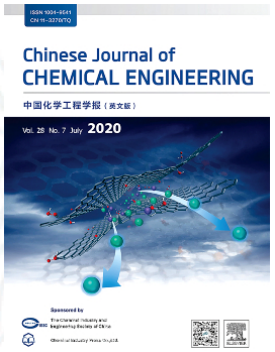

Chinese Journal of Chemical Engineering

Certificate of Reviewing

Awarded since December 2017 (1 review)
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WIDI ASTUTI

in recognition of the review contributed to the journal

The Editors of Chinese Journal of Chemical Engineering

Elsevier Reviewer RECOGNITION

5. Bukti revise decision

Widi Astuti <widi_astuti@mail.unnes.ac.id>

Revise decision made on CJCHE_2017_1493

1 message

Chinese Journal of Chemical Engineering <EviseSupport@elsevier.com> Tue, Dec 12, 2017 at 2:56 PM
Reply-To: system@evise.com
To: widi_astuti@mail.unnes.ac.id

Ref: CJCHE_2017_1493
Title: Adsorption of Anionic Dyes on Spent Tea Leaves Modified with Polyethyleneimine (PEI-STL)
Journal: Chinese Journal of Chemical Engineering

Dear Professor Astuti,

Our records indicate that you reviewed a version of this manuscript. This is to inform you that the editor has requested that the author revise the current version of this manuscript.

Reviewer and Editor comments to the author can be found below.

I appreciate your time and effort in reviewing this paper and greatly value your assistance as a reviewer for Chinese Journal of Chemical Engineering.

I hope you enjoyed using Scopus and that it helped you to review this article. If you have not yet used your 30-day full access to Scopus, you can still do so. Your Scopus access is valid starting from the day you agreed to review the article.

Kind regards,

Guohua Chen
Associate editor

Editor and reviewer comments:

--**Reviewer 1**

- The manuscript is interesting, however need some revision and clarification on some points. Need check english.

-Reviewer 2

-

This manuscript is about the removal of toxic dyes using spent Tea leaves modified with PEI-STL. The paper is poorly written and this is a routine work. So, the manuscript needs major revision before its acceptance in this Journal. Below are some of the suggestions:

1. There are thousands adsorbents available for the removal of these dyes. Then, how the presented work is better than the previously published similar work in the terms of cost and performance.

2. There are several typographical and grammatical mistakes which should be corrected

3. The characterization part is very weak. The authors must include the SEM, before and after adsorption

4. The quality of the Figures is very poor so it should be improved. No need to give Figure 1. I am very surprised that the authors did not even add the Figure number then how the reviewer will understand about the Figures. I think this paper is not written by an experienced person. The figures for all parameters (Ph, time, dose and temp) should be merged in one Figure.

5. The regeneration study is very important which should be performed by using different solvents.

6. A mechanism for the adsorption of dyes on the adsorbent should be given as a figure.

7. The introduction and result and discussion part should be improved. The authors gave several old references which should be deleted and the following latest references may be included:

Chemical Engineering Journal 330, 1351-1360, 2017; *ACS Appl. Mater. Interfaces*, 2017, 9 (41), pp 36026–36037; Process Safety and Environmental Protection, 109, Pages 301-310, 2017; Journal of Photochemistry and Photobiology A: Chemistry 347, 235-24, 2017; Chemical Engineering Journal 307, 264-272, 2017; Journal of Chemical & Engineering Data 61 (11), 3806-3813, 2016; Journal of Photochemistry and Photobiology A: Chemistry 329, 61-68, 2016; Toxicological & Environmental Chemistry 97 (5), 526-537, 2015; Journal of Molecular Liquids 242, 478-483, 2017

-Reviewer 3

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The work investigated the adsorption performance of polyethyleneimine (PEI) treated spent tea leaves (STL) for Reactive Black 5 (RB5) and Methyl Orange (MO) from simulated wastewater. The kinetic of adsorption have been probed. The effects of PH, temperature and adsorbent dosage on the adsorption performance of adsorbent were also reported. I am regret the work can not be published in the present form. The following issues must be addressed:

1. In line 13 page 12, why do you think the adsorption process is controlled by chemisorption? Chemisorption should be decide by the interaction of adsorbent and adsorbate, not the adsorption kinetic.

2. Page 7, Table 2, the surface area of STL and PEI-STL are too low (2~3 m2/g, and can be in the error range) to be suitable for adsorption. Certain pretreatment may be made to enlarge the porosity before introducing PEI.

3. In line 30 page 3, the ratios between PEI and STL (2:1, 1:1, and 1:2) means mass ratio, or volume ratio? Please give the exact meaning.

4. In line 6 page 4, the description of UV-VIS spectrophotometer should be consistent in the manuscript.
5. In line 21 page 5, there were only three samples in Fig 3, not four.
6. It would be better that citation of the different characteristics of MO and RB5 molecules in line 14 page 8.
7. In line 22 page 9, the label of temperature is wrong.
8. In line 1 page 11, only heat of adsorption can be estimated through the adsorption mechanism, not the activation energy.