



## Removal of sodium dodecylbenzenesulfonate from aqueous solution using polyethyleneimine-modified bentonite clay

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### ABSTRACT

In this study, adsorption of the anionic surfactant, sodium dodecylbenzenesulfonate (SDBS), onto the cationic polymer, polyethyleneimine (PEI)-modified bentonite clay (PMB), was investigated. In order to prepare PMB, the bentonite suspension was mixed with PEI and the adsorption kinetic and adsorption capacities of the PMB suspensions were determined using SDBS as adsorbate. Fourier transform infrared spectroscopy (FTIR) was used in the characterization of PMB. The adsorption studies of PMB were carried out using batch adsorption methods. The equilibrium was achieved in 10 min. Adsorption data were applied with several kinetic (pseudo-first-order, pseudo-second-order, intra-particle, and Elovich) and isotherm (the Langmuir, Freundlich, Dubinin–Radushkevich (D–R), and Temkin) models. All the experimental data of SDBS on PMB were analyzed with error functions as correlation coefficient ( $R^2$ ) and the normalized standard deviation  $\Delta q$  (%). Results indicated that for the adsorption kinetics, the best fit was provided from the pseudo-second-order kinetic model with  $R^2$  value of 0.9999. For the adsorption isotherms, the best fit was obtained Langmuir and Temkin isotherms with  $R^2$  value of 0.9968 and 0.9865, respectively. High adsorption capacity ( $q_m$ ) as 443.1 mg·g<sup>-1</sup> was determined from the Langmuir isotherm.

*Keywords:* Bentonite clay; Adsorption; Kinetic; Cationic polyelectrolyte; Anionic surfactant

### 1. Introduction

Surfactants are the main substances found in the content of miscellaneous products used in industry and in the household, and they mix to the surface and underground waters, which results in ecological risks. Alkyl benzene sulfonates are the main components of detergents, mainly ranking from C10 to C16. Sodium dodecylbenzenesulfonate (SDBS) is a good representative of the whole group with C number 12. Various publications have reported on the removal of surfactants from waste waters [1–7]. The adsorption of SDBS on activated carbon [8–10], bentonite clays [11,12], and zeolites [13,14] have been investigated.

Bentonite is a clay mainly consists of montmorillonite, which has crystalline structure with three layers of one octahedral alumina sheet compressed between two tetrahedral silica sheets. Clay minerals are used as adsorbent for the

removal of pollutants from wastewater because of their high specific area and low cost. However, the surface of clay minerals is usually charged negatively. Thus, although bentonite did well in sorption of cationic species, it failed in sorption of anionic species. If the inorganic cations in the interlayer of clay are exchanged with certain organic cations, natural clay mineral converts into organo-clay, which has the capability to sorb anionic pollutants. For this purpose, the surface of clay is modified with cationic surfactants or polymers to enhance the removal efficiency of anions. Polyethyleneimine (PEI) is a non-toxic cationic polyelectrolyte, used often in water treatment and as transfection agents to deliver DNA to cells for therapeutic applications [15]. In our previous studies on the rheological behavior of PEI and clay suspension, we observed a high affinity of PEI towards bentonite clay [16–18]. So far, bentonite clays modified with PEI were not used for the adsorption of surfactants. In the present work, bentonite saturated with PEI

was studied as a composite adsorbent for the effective elimination of SDBS from aqueous solution. Adsorption kinetic and adsorption isotherms were introduced.

## 2. Experimental

### 2.1. Materials and methods

The natural Ca-bentonite (CaB) was procured from the Enez region of Turkey. The determination of the chemical and silica contents of the CaB was achieved by using an atomic adsorption spectrophotometer (Perkin Elmer 3030 model) and the gravimetric method, respectively. The composition of the natural clay is shown in Table 1. The mean grain size and specific area of clay are 366.8 nm and 47.36 m<sup>2</sup>·g<sup>-1</sup>, respectively [16].

Molecular mass of used PEI ranges from 6 × 10<sup>5</sup> to 1 × 10<sup>6</sup> and was acquired from Fluka (Buchs, Switzerland). SDBS were purchased from Aldrich (St. Louis, MO, USA). All other reagents were analytical grade.

The SDBS concentration of the solutions was determined by UV–Vis spectrophotometer (Shimadzu UV-1800) at 224 nm. The Fourier transform infrared spectroscopy (FTIR) spectra of samples were measured by FTIR spectrometer (Perkin Elmer spectrometer 65, Waltham, MA, USA). All solutions were prepared with ultrapure water purifier by an Elga Purelab Option-7-15 filtration system (Elga, UK). pH measurements were carried out by using an Orion Dual Star pH-ISE meter (Thermo Fisher Scientific Beverly, MA, USA) with combined glass pH electrode.

### 2.2. Preparation of PEI-modified bentonite adsorbent

Bentonite was modified with PEI by the adsorption procedure described in our previous study [16]. The adsorption capacity has been reported as 145 mg PEI per gram bentonite clay. For this purpose, an amount of PEI equal to five times of the adsorption capacity of the clay was added to the bentonite clay suspension. The suspensions were stirred for 24 h at 25°C and centrifuged. The excess of PEI was removed by washing of solid product with deionized water. The solids were dried at room temperature. The PEI-modified bentonite (PMB) sample was gently ground and sieved to 90 μm particle size before SDBS adsorption studies.

Table 1  
Chemical composition (% w/w) of natural Ca-bentonite (CaB) sample

Sample	CaB
SiO <sub>2</sub>	60.18
Al <sub>2</sub> O <sub>3</sub>	18.49
Fe <sub>2</sub> O <sub>3</sub>	5.65
Na <sub>2</sub> O	1.41
CaO	4.60
K <sub>2</sub> O	2.32
MgO	2.40
MnO	0.11
TiO <sub>2</sub>	0.68
P <sub>2</sub> O <sub>5</sub>	0.37

### 2.3. Adsorption studies

The batch equilibrium method was used in adsorption experiments that were carried out in triplicate using 0.02 g PMB in 20 mL of SDBS solutions of several concentrations (0–3.5 g·L<sup>-1</sup>). Solutions were agitated for 2 h at 25°C. After centrifugation at 5,000 rpm for 2 h, supernatants were filtered using a 0.45-μm membrane filter.

Adsorption kinetics was followed by measuring decreasing SDBS concentration with time. A UV absorption spectrophotometer was used to detect SDBS concentrations of solutions. From adsorbed contents, adsorption kinetic calculations were made.

The adsorption rate was obtained by the addition of 0.2 g of adsorbent to 200 mL of 175 mg·L<sup>-1</sup> SDBS containing solution. As the solutions were mixed at 420 rpm, aliquots of 5 mL were taken at a certain time intervals (1, 2, 3, 4, 5, 10, 15, 20, 30, 60, 120, and 1,440 min), centrifuged at 5,000 rpm for 2 h, filtered using a 0.45-μm membrane filter. The concentrations of SDBS in the filtrates were determined. The adsorbed amount was calculated from the difference between the initial and equilibrium concentrations. Experimental data were analyzed by using following kinetic and isotherm models.

### 2.4. Kinetic models

#### 2.4.1. The pseudo-first-order model

The linear form of the pseudo-first-order model of Lagergren can be expressed as [19]:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (1)$$

where  $q_e$  and  $q_t$  (mg·g<sup>-1</sup>) are the adsorbed amounts at equilibrium and at time  $t$ , respectively.  $k_1$  (min<sup>-1</sup>) is the rate constant of first-order.

#### 2.4.2. The pseudo-second-order model

The pseudo-second-order model can be indicated as follows [20]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where  $k_2$  (g·mg<sup>-1</sup>·min<sup>-1</sup>) is the rate constant of second-order.

#### 2.4.3. The intraparticle diffusion model

This model describes the diffusion of the adsorbate within the adsorbent. An intraparticle diffusion model is used to describe the rate-determining step in the adsorption process. This intraparticle diffusion model is represented as follows [21]:

$$q_t = k_{id} t^{0.5} \quad (3)$$

where  $q_t$  is the adsorbed amount (mg·g<sup>-1</sup>),  $t$  is the time (min),  $k_{id}$  (mg·g<sup>-1</sup>·min<sup>-0.5</sup>) is the rate constant of intraparticle

diffusion. If the plot is linear, intraparticle diffusion is the rate-controlling step of adsorption. If the plot is non-linear, it is not rate-controlling step. In that case, adsorption may be controlled by multiple processes.

#### 2.4.4. The Elovich model

The linearized the Elovich model is expressed by the following equation [22]:

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln(t) \quad (4)$$

where  $q_t$  is the adsorbed amount at time  $t$  ( $\text{mg}\cdot\text{g}^{-1}$ ),  $\alpha$  is defined as the initial sorption rate ( $\text{mg}\cdot\text{g}^{-1}\cdot\text{min}^{-1}$ ) and  $\beta$  is a constant connected the activation energy for chemisorption ( $\text{g}\cdot\text{mg}^{-1}$ ), and  $t$  is the time (min).

### 2.5. Adsorption models

#### 2.5.1. The Langmuir isotherm

According to the Langmuir isotherm model, adsorption occurs on the adsorbent with homogeneous surface. The Langmuir isotherm is applied successfully to monolayer adsorption. The linear form of the Langmuir isotherm model is represented as follows [23]:

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m} \quad (5)$$

where  $C_e$  ( $\text{mg}\cdot\text{L}^{-1}$ ) is the equilibrium concentration,  $q_e$  ( $\text{mg}\cdot\text{g}^{-1}$ ) is the amount adsorbed at equilibrium,  $q_m$  ( $\text{mg}\cdot\text{g}^{-1}$ ) and  $b$  ( $\text{L}\cdot\text{mg}^{-1}$ ) are the Langmuir constants.  $q_m$  is the monolayer adsorption capacity and  $b$  is relevant to the heat of adsorption.

#### 2.5.2. The Freundlich isotherm

The Freundlich isotherm is generally used for multilayer adsorption on a heterogeneous surface. The linear form of Freundlich equation is expressed as [24]:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (6)$$

where  $K_F$  and  $n$  are constants of the Freundlich isotherm relevant to adsorption capacity and adsorption intensity, respectively.

#### 2.5.3. The Temkin isotherm

The linearized Temkin isotherm model is given by the following equation [25]:

$$q_e = B \ln A + B \ln C_e \quad (7)$$

where  $A$  ( $\text{L}\cdot\text{mg}^{-1}$ ) and  $B$  ( $RT/b$ ) are constants, and  $B$  is related to the heat of adsorption.

#### 2.5.4. The Dubinin–Radushkevich isotherm

The linear form of the Dubinin–Radushkevich (D–R) isotherm model is expressed as [26,27]:

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (8)$$

where  $q_m$  is the D–R monolayer adsorption capacity constant ( $\text{mg}\cdot\text{g}^{-1}$ ),  $\beta$  is the constant related to adsorption energy, and  $\varepsilon$  is the Polanyi potential which is given as

$$\varepsilon = RT \ln \left[ 1 + \frac{1}{C_e} \right] \quad (9)$$

The mean free energy of adsorption,  $E$  ( $\text{kJ}\cdot\text{mol}^{-1}$ ) is calculated from  $\beta$  constant:

$$E = \frac{1}{\sqrt{2\beta}} \quad (10)$$

The value of  $E$  distinguishes between physical or chemical adsorption.

### 2.6. Validity of models and statistical evaluation

The validity of each model was tested by using two error analysis methods, the correlation coefficient ( $R^2$ ) of lines in the graphs and the normalized standard deviation  $\Delta q$  (%) which was calculated by following Eq. (11)

$$\Delta q(\%) = 100 \times \sqrt{\frac{\sum [(q_{\text{exp}} - q_{\text{cal}})/q_{\text{exp}}]^2}{(n-1)}} \quad (11)$$

where  $q_{\text{exp}}$  and  $q_{\text{cal}}$  are the experimental and calculated adsorption amount of SDBS with related model, respectively, and  $n$  is the number of data points.

## 3. Results and discussion

PEI is a water-soluble polyamine, and its chemical structure is given in Fig. 1. The positive charge on PEI occurs as a consequence of protonation of the imine groups on PEI. The pH of the solution and molecular weight of PEI strongly influence the charge density of PEI. At low pH values due rising the degree of protonation, the positive charge of the polymer increases. The PEI used in this study has a high molecular weight and has positive charge on a wide pH interval (3 to 11) [28]. The pH of PEI-clay suspension during the preparation of PMB adsorbent was measured as around 8.5. Therefore, at this pH, PEI positively charged and interacts with the negatively charged clay surface.

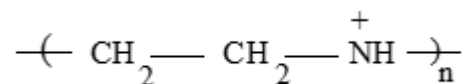


Fig. 1. The structural units of PEI.

Fig. 2 shows FTIR spectra of CaE (spectrum a) and PMB (spectrum b). For CaE, the bands at  $3,631\text{ cm}^{-1}$  (O–H stretching),  $1,635\text{ cm}^{-1}$  (H–O–H bending in water), and the bands between  $600$  and  $1,128\text{ cm}^{-1}$  (Si–O–Si, Al–Al–OH, Al–Si–O, and Si–O) were observed.

In PMB, the representative peaks between  $3,000$  and  $2,800\text{ cm}^{-1}$  corresponding to –C–H stretching and between  $1,500$  and  $1,300\text{ cm}^{-1}$  corresponding to –N–H bending and –C–H bending are clearly separated in the spectrum of PMB from the spectrum of CaE.

### 3.1. Adsorption kinetics

Kinetic studies describe absorption rates of SDBS on PMB, which can identify the mechanism of adsorption. Fig. 3 shows absorption rates of SDBS on PMB at  $25^\circ\text{C}$ . As seen from the figure, for PMB, the adsorption rates of SDBS are fast and equilibrium is reached in 10 min. The adsorption process has two stages. The reaction stage is between 0 and 10 min and the equilibrium stage is after 10 min. Adsorption data were analyzed by using pseudo-first-order, pseudo-second-order, intraparticle diffusion, and Elovich models.

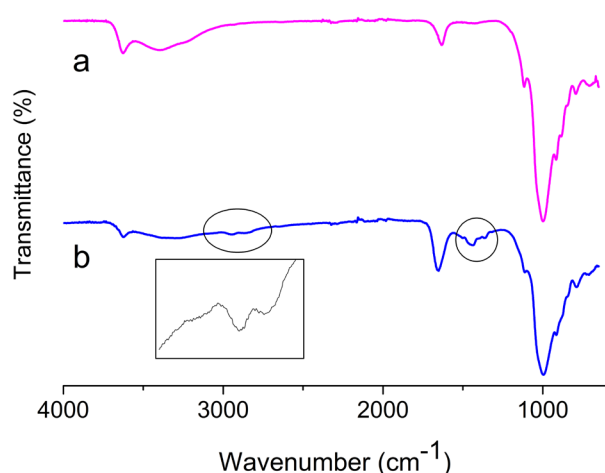


Fig. 2. FTIR spectra of CaE (a) and PMB (b).

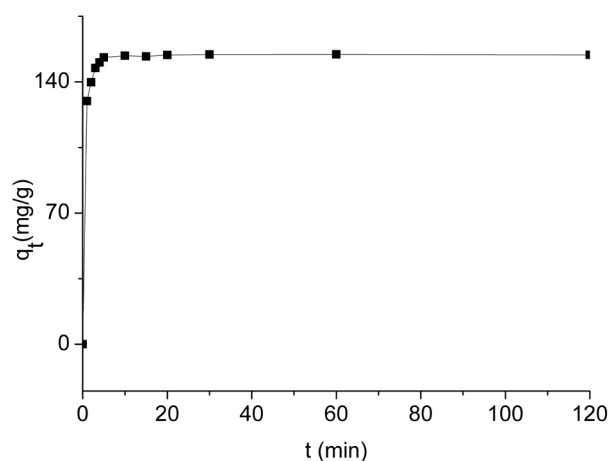


Fig. 3. Effect of contact time on SDBS absorption at  $25^\circ\text{C}$ .

Fig. 4 shows the plots of four kinetic models at  $25^\circ\text{C}$ . The types of the adsorbent and the surfactant mostly influence the kinetics of adsorption. Generally, a very fast adsorption rate occurs if the adsorbate and the adsorbent are oppositely charged. Since PEI and SDBS are oppositely charged, due to the electrostatic interaction, equilibrium state is reached in only 10 min.

The suitability of the models depends on the values of correlation coefficient ( $R^2$ ) and calculated  $q_e$ . Table 2 lists the parameters of kinetic models and coefficients. As seen from the values of  $R^2$ , the experimental data are matched with the kinetic models studied, except for the pseudo-first-order kinetic model. Among three models, the value of  $R^2$  for the pseudo-second-order kinetic model was very high, so that this model explains the adsorption mechanism. The calculated  $q_e$  value from the pseudo-second-order kinetic model is compatible to the experimental  $q_e$  value. The pseudo-second-order model supposes that chemisorption is the rate-controlling mechanism [20], which takes place due to the electrostatic attraction force between SDBS and PMB. Furthermore, the Elovich equation was also available to describe the kinetics of the adsorption of SDBS, which suggests a chemisorption mechanism.

The pseudo-second-order model cannot describe the diffusion mechanisms in the adsorption process; therefore, the intraparticle diffusion model was also checked by using Eq. (3). If two or more parts occur in the plots, the intraparticle diffusion model is not linear. The first part is surface adsorption and other part is also intraparticle diffusion. If the plot of the model is linear and it also passes through the origin, intraparticle diffusion is the rate-controlling step. In this study, the plot was not linear in all time range, so that intraparticle diffusion was not only rate-controlling step. The rate constant of intraparticle diffusion was calculated as  $18.93\text{ mg g}^{-1}\text{ min}^{-0.5}$ .

### 3.2. Adsorption isotherms

Fig. 5 shows the adsorption isotherm of SDBS on PMB at  $25^\circ\text{C}$ . This adsorption isotherm indicates high affinity type, where adsorption values first start with a sharp increase and later become smooth. As can be seen from Fig. 5, the saturation adsorption values for PMB are higher than around  $400\text{ mg SDBS}\cdot\text{g}^{-1}\text{ PMB}$ . The adsorption of SDBS on PMB particles was analyzed using the Langmuir, Freundlich, Temkin, and D–R models.

Fig. 6 shows the Langmuir, Freundlich, Temkin, and D–R isotherms. Table 3 gives the parameters and coefficients of these isotherms. The Langmuir and Temkin isotherms fit better than Freundlich and D–R isotherms. The values of  $R^2$  for Langmuir and Temkin isotherms are higher than other isotherms. Adsorption capacity was calculated as  $443.1\text{ mg}\cdot\text{g}^{-1}$  from the Langmuir isotherm. This value is matched with experimental data, which indicates monolayer surface adsorption.

According to Eq. (12), from the  $b$  constants of the Langmuir isotherm, the separation factor or equilibrium parameter ( $R_L$ ) can be calculated, which shows whether an adsorption system is favorable or unfavorable [11,29]:

$$R_L = \frac{1}{1 + bC_0} \quad (12)$$

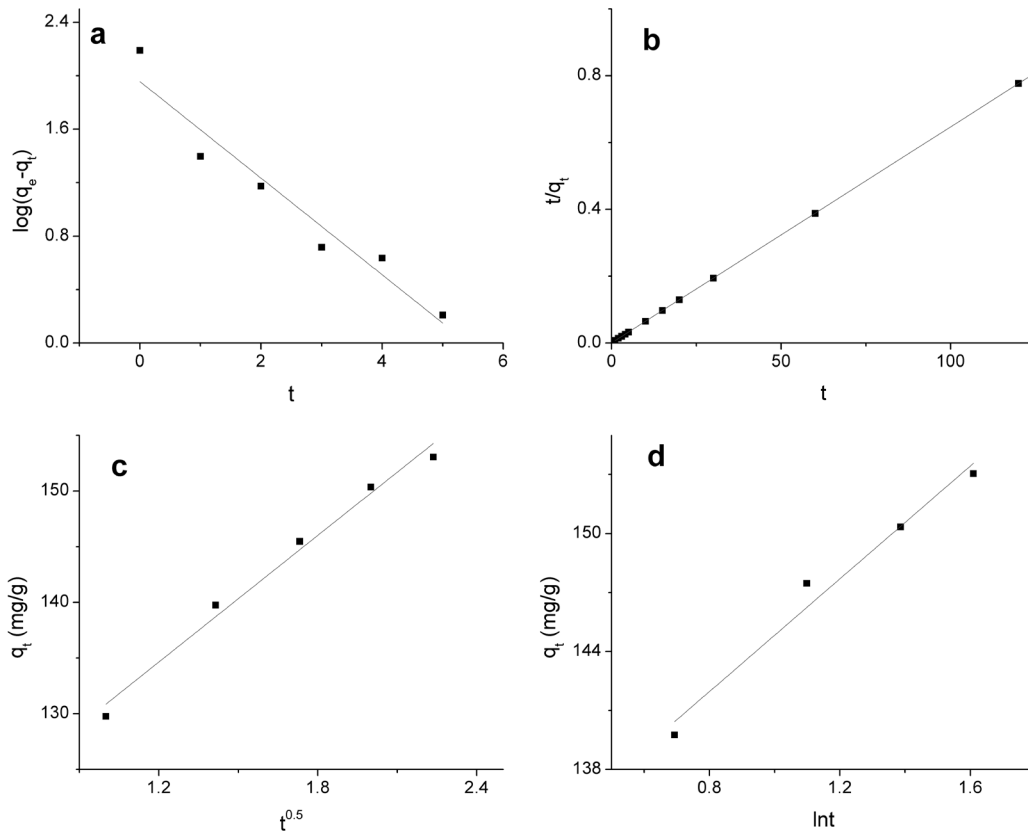


Fig. 4. Pseudo-first-order (a), pseudo-second-order (b), intraparticle diffusion (c), and Elovich (d) kinetic models fit for SDBS sorption onto PMB particles.

Table 2  
Parameters of the kinetic models

Kinetic models	Parameters
Pseudo-first-order kinetic	
$q_e$ (mg·g <sup>-1</sup> )	93.23
$k_1$ (min <sup>-1</sup> )	0.8823
$R^2$	0.9418
% $\Delta q$	1.198
Pseudo-second-order kinetic	
$q_e$ (mg g <sup>-1</sup> )	155.9
$k_2$ (g mg <sup>-1</sup> ·min <sup>-1</sup> )	0.0379
$R^2$	0.9999
% $\Delta q$	3.262
Intraparticle diffusion model	
$k_{id}$ (mg g <sup>-1</sup> min <sup>-0.5</sup> )	18.93
$R^2$	0.9865
% $\Delta q$	0.7591
Elovich model	
$A$	$1.25 \times 10^5$
$B$	0.06950
$R^2$	0.9711
% $\Delta q$	1.100

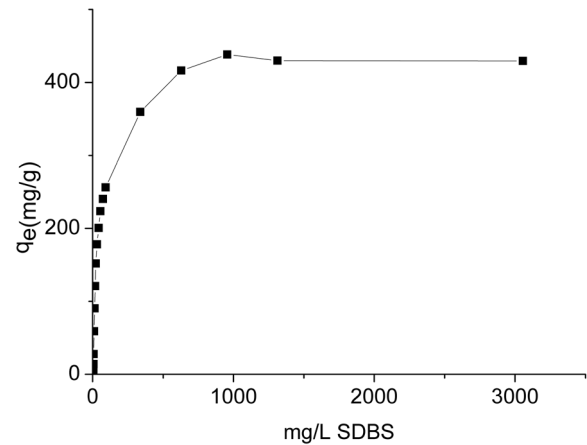


Fig. 5. Adsorption isotherm of SDBS on PMB at 25°C.

If  $R_L$  is between 0 and 1, adsorption is accepted as favorable. In this study, the values of  $R_L$  for all  $C_0$  values were between 0.01 and 0.78. According to these results adsorption of SDBS on PBM is favorable. This parameter obtained from the Langmuir isotherm indicates that PMB is a convenient adsorbent for adsorption of SDBS from aqueous solution.

The value of  $R^2$  for the Temkin isotherm is smaller than that for the Langmuir isotherm. This isotherm gives

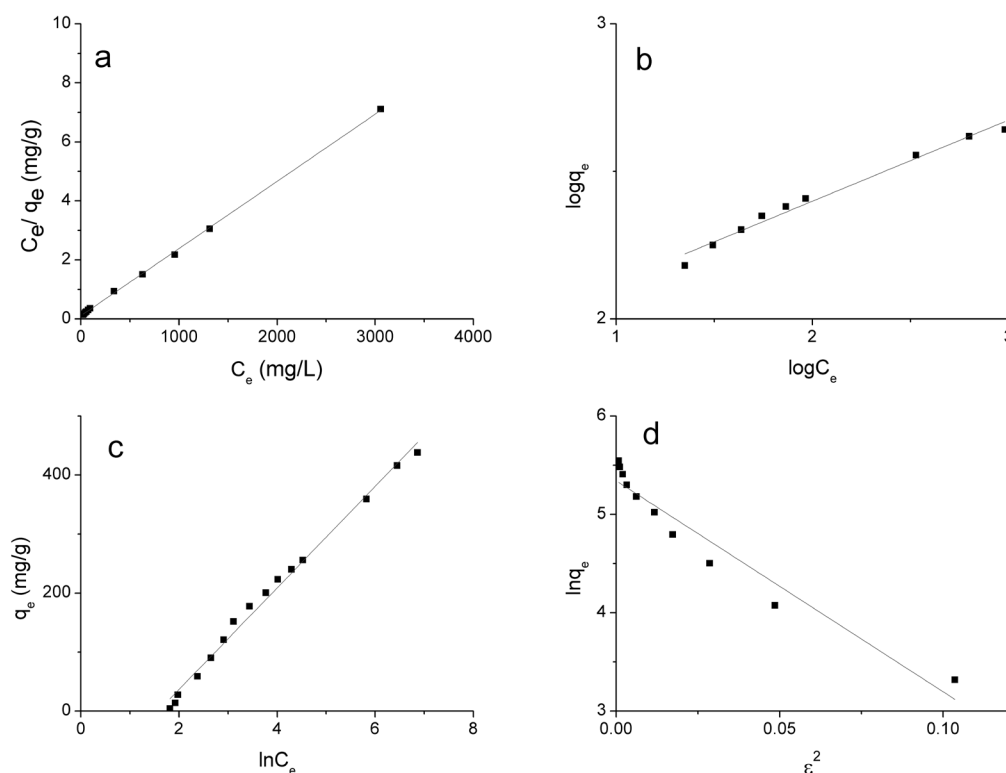


Fig. 6. Langmuir (a), Freundlich (b), Temkin (c), and D–R (d) isotherm models for adsorption of SDBS on PEI-bentonite particles at 25°C.

Table 3  
Parameters of isotherm models for adsorption of SDBS on PMB at 25°C

Isotherms	Parameters
Langmuir	
$q_m$ (mg·g <sup>-1</sup> )	443.1
$b$ (L mg <sup>-1</sup> )	0.02796
$R^2$	0.9968
% $\Delta q$	6.755
Freundlich	
$K_f$ (mg·g <sup>-1</sup> )	1.423
$1/n$	0.4605
$R^2$	0.8026
% $\Delta q$	83.12
Temkin	
$A$ (L·g <sup>-1</sup> )	0.2068
$B$ (J mol <sup>-1</sup> )	80.14
$R^2$	0.9865
% $\Delta q$	10.30
D–R	
$q_m$ (mg·g <sup>-1</sup> )	209.0
$B$	-21.46
$R^2$	0.9427
% $\Delta q$	17.45

information about adsorption heat of adsorbents. The value of  $E$  was determined by the D–R isotherm as 0.15 kJ·mol<sup>-1</sup>. Because this value is smaller than the value of 8 kJ mol<sup>-1</sup>, adsorption is basically physical [30].

The adsorption has the characteristics of strong, rapid, and high capacity. The experimental results and high adsorption rates suggest that the adsorption is due to mainly to chemisorption and partially physisorption. The chemisorption occurs with electrostatic attraction between the negative head groups of SDBS and positively charged PMB surfaces. Physisorption can take place also due to hydrophobic interaction between the hydrophobic tail of the surfactant and the polymer.

Table 4 gives the literature values of adsorption capacities of bentonite- and zeolite-based adsorbents, and also activated carbons for SDBS. As seen from Table 4, the adsorption capacity of PMB used in this study is higher than those of bentonite- and zeolite-type adsorbents. Several studies were reported related to the adsorption of SDBS on bentonite, kaolinite, and montmorillonite [31–33]. For anionic compounds, clays were not efficient adsorbents due to the negative charge of the clay surface that repels the anionic species. The adsorption capacity of SDBS for bentonite, kaolinite, and montmorillonite were reported as about 191.7, 8.78, and 60 mg g<sup>-1</sup>, respectively, which are all much smaller than the adsorption capacity of PMB (443.1 mg·g<sup>-1</sup>) reported in this study.

As seen from Table 4, in order to increase adsorption capacities of bentonites, a modification step is necessary. This step aims to change the surface charge of bentonite



Table 4  
Comparison of adsorption capacity of PMB with bentonite, zeolite, and activated carbon-based adsorbents reported in the literature for SDBS

Adsorbent	Adsorption capacity (mg·g <sup>-1</sup> )	References
PMB	443.1	This study
HCl-activated bentonite	298.5	[11]
CTAB-modified bentonite <sup>a</sup>	216	
TAB-modified bentonite <sup>b</sup>	170	[12]
BTC-modified bentonite <sup>c</sup>	216	
Zeolites	0.767–130	[13]
CTAB-modified zeolite <sup>a</sup>	30.7	[14]
Activated carbons	260–470	[8]
Activated carbons	109.0–232.3	[9]
Lignin-based activated carbon	658.6–802.5	[10]
Bentonite	191.7	[31]
Kaolinite	8.78	[32]
Montmorillonite	60	[33]

<sup>a</sup>Hexadecyltrimethylammonium bromide.

<sup>b</sup>Tetramethylammonium bromide.

<sup>c</sup>Alkylbenzyltrimethylammonium chloride.

from negative charge to positive charge, with the help of acid or cationic surfactants. Activated carbons are known with high adsorption capacities in many adsorbents. Adsorption capacities of activated carbons used for SDBS adsorptions are lower or compatible with our results found with PMB. With only lignin-based activated carbon, higher adsorption capacity was obtained. However, for this adsorbent, 1–2 weeks were reported for contact time depending of samples.

#### 4. Conclusions

A novel adsorbent was prepared by modification of bentonite with PEI. It was found that PMB can be effectively utilized for removal of SDBS ions from aqueous solution. The preparation method of adsorbent is very simple and the contact time is very fast. It reached equilibrium in only 10 min. A high adsorption capacity as 443 mg SDBS·g<sup>-1</sup> was obtained. This amount is compatible with those found for active carbons. Moreover, both bentonite and PEI are non-toxic materials and both are used separately in water treatment. Consequently, PMB adsorbent could be proposed for effective removal of anionic surfactants from aqueous medium.

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