



Catecholamine Coated Maghemite Nanoparticles for Asphaltene Adsorption/Desorption Process

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Received: 29 November 2020, Revised: 24 January 2021, Accepted: 26 January 2021

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Abstract

In this study, a nano-layer polydopamine (PDA) coated superparamagnetic maghemite nanoparticles (MNPs) was applied to investigate the asphaltene adsorption-desorption behavior using a model solution. In this process, Fourier-transform infrared spectrum (FT-IR) spectroscopy of the polydopamine/MNP core/shell (MNP@PDA) before and after asphaltene adsorption was indicated the attachment of asphaltene molecules on the polymeric nano-adsorbent surface. The isotherms and kinetics of the asphaltene adsorption process on the MNP@PDA were studied. The good prediction of asphaltene adsorption by the modified Langmuir model indicated that adsorption occurs on an MNP@PDA surface by multilayer adsorption. Also, within the kinetic models, the double-exponential model can fit the experimental data well. The obtained results revealed that about 90% removal for asphaltene happened within 30 min which can be acceptable. The results of this study showed that natural polymers can be used for surface modification of nanoparticles and use it successfully for asphaltene adsorption from prepared asphaltene-toluene solution.

Keywords:

Adsorption,
Asphaltene,
Maghemite,
Nanoparticles,
Polydopamine

Introduction

Heavy oil and bitumen reserves are the most unconventional available energy source that their utilization is expected to increase in the close future. The production of useful petroleum products from such residua requires new upgrading techniques [1]. Accordingly, the changes in pressure, temperature, and compositions may cause precipitation of heavy organic solids such as asphaltene, which decreases the efficiency of the good flow during different stages of hydrocarbons production [2, 3]. During the crude oil production and refining, at first, asphaltene precipitation/aggregation may be occurred and then deposited in the reservoir rock, wellbore, equipment, and transmission pipes [4]. Moreover, the viscosity of heavy and extra-heavy oils can dramatically increase because of the increase in the rate of asphaltene aggregation [5]. The adsorption of previously precipitated asphaltene onto mineral surfaces can also lead to formation damage in oil reservoirs by reducing the oil effective permeability [6]. Suppose the polar groups of the asphaltene molecular structure stick onto the rock surface. In that case, the rock wettability can be altered from water-wetting to oil-wetting, thereby affect the final oil recovery properties [6].

Recently, nanotechnology has been used as an interesting method to control the asphaltene deposition process[7-9]. Nassar studied asphaltene adsorption onto alumina nanoparticles. He showed that nanoparticles would remove asphaltene from the heavy oil rapidly and thus making the remaining fraction of oil transportable for conventional processing. Second, nanoparticles

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could be employed as catalysts for upgrading asphaltenes into light usable distillates [10]. Nassar et al. investigated the adsorption and catalytic oxidation of asphaltene using two different alumina particles (micro- and nano-particles) that have different particle sizes and surface acidities. The obtained results showed that alumina nanoparticles have an adsorption capacity for asphaltenes higher than that of alumina microparticles based on the surface area [11]. In other work, different metal oxide nanoparticles such as NiO, Co₃O₄, and Fe₃O₄ were applied for the adsorption/oxidation of Athabasca asphaltenes [12, 13]. Mirzayi and Shayan investigated the asphaltene adsorption and oxidation on synthesized maghemite nanoparticles [14]. The results of their study showed that the equilibrium condition for asphaltene adsorption onto maghemite nanoparticles is established in less than 2 h, and the adsorption kinetics data exhibited a good fit with the pseudo-second-order model. Additionally, their study confirmed that the adsorption isotherms are in good agreement with the Langmuir isotherm model. Franco et al. used fumed silica and nanoparticles of NiO and/or PdO supported on fumed silica for the adsorption and post-adsorption oxidation of n-C₇ asphaltene using different ratios of heptol (n-heptane and toluene) [15]. According to their results, the novel three-parameters solid-liquid equilibrium (SLE) model [16] was able to fit the adsorption isotherms of asphaltene from different solvents including different volume ratios of n-heptane/toluene. Dopamine (DA) is one of the catecholamine families, which could be polymerized to polydopamine (PDA) at a slightly alkaline pH. The polymerization process involves the reaction of dopamine hydrochloride in Tris-HCl solution as an alkaline buffer. Because of the different functional groups such as amino, imino, and catechol in their structure, the polydopamine coatings is one of the interesting topics in the last decade of studies [17]. The active surface of polydopamine enables it to adhere easily to the other material's surface for different purposes. For example, polydopamine coated nanoparticles have been used for environmental remediation [17], drug delivery [18, 19], biomedical [20], biosensors [21], etc.

Herein, in the present study, the surface of the synthesized maghemite nanoparticles (MNPs) was coated with a bio-based polymer (i.e. polydopamine, PDA), and the prepared polydopamine/MNPs core/shell (MNP@PDA) was examined to adsorb the asphaltene from a model solution for the first time. By conducting different batch-mode experiments, the kinetics, isotherm, and thermodynamic behavior of asphaltene adsorption onto MNP@PDA were investigated. The core/shell nanoparticles prepared in this research are proposed as potential candidates for removing asphaltene from crude oils.

Experimental

Materials and Methods

Tris (hydroxymethyl)-aminomethane (Tris) and 4-(2-aminoethyl) benzene-1,2-diol (dopamine, DA) were purchased from Sigma-Aldrich. Ammonia solution (25%), HCl, FeCl₃ and FeCl₂•4H₂O, toluene, and n-heptane were purchased from Merck. All the chemical reagents used in this work were of analytical grade and were used as received without further purification. The crude oil is produced from a reservoir located in the south of Iran. The asphaltenes content was extracted from the crude oil sample according to IP-143 standard. Maghemite nanoparticles (MNPs) were prepared by co-precipitation of ferric and ferrous salts. The obtained MNPs were washed with deionized water and ethanol [22].

Synthesis of Polydopamine Coated γ -Fe₂O₃ Nanoparticles (MNP@PDA)

The solution of FeCl₃ (1 M), FeCl₂•4H₂O (2 M), ammonia (2 M), and hydrochloric acid (2 M) was prepared and the ammonia solution was transferred into a reaction flask and iron salt solution was added dropwise under agitation by a mechanical overhead stirrer within 20 min.

The obtained MNPs were washed three times with deionized and ethanol. The prepared nanoparticles were separated by an external magnet and redispersed in 40 mL ethanol for later use. Then, the polydopamine coated maghemite nanoparticles (MNP@PDA) were prepared by adding dopamine (2 mg/mL) to the synthesized MNPs in Tris buffer solution (pH=8.5, 10 mM) [17, 23]. To remove the unreacted monomers and impurities, the synthesized product was washed several times with deionized water. Finally, the obtained product was dried at 40 °C in an oven to obtain the final powder. The schematic of polydopamine polymerization and surface coating of MNP has been illustrated in Fig. 1.

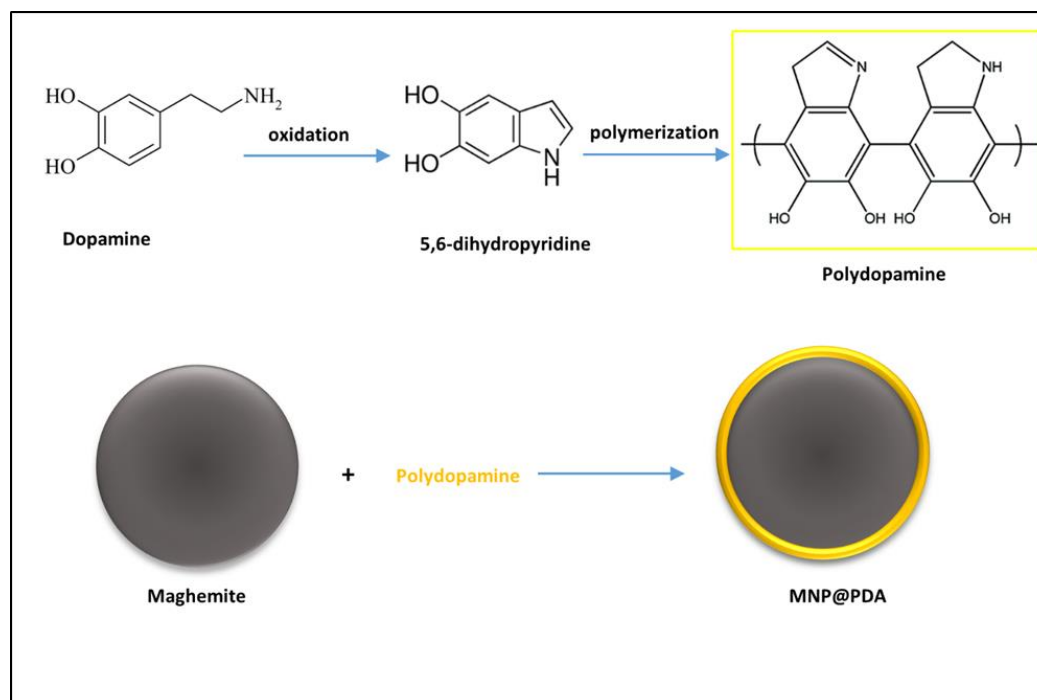


Fig. 1. Schematic representation of polydopamine polymerization and surface coating of MNP

Adsorption Experiments

Adsorption of asphaltene onto MNP@PDA was performed at temperatures 25, 40, and 60 °C. Batch adsorption experiments were carried out by adding a certain mass of MNP@PDA to the asphaltene-toluene solution so that a ratio of 10:1 (g/L) was achieved. The vials containing the samples were shaken at 250 rpm in an incubator at 25, 40, or 60 °C for 24 hours. Then the nanoparticles containing adsorbed asphaltene were separated via a strong magnetic field followed by applying centrifugal force at 2500 rpm for 10 min. The concentration of the remaining asphaltene in the supernatant solution was evaluated using a UV-vis spectrophotometer at a wavelength of 297 nm. The amount of asphaltene adsorbed onto nanoparticles, q_t (mg/g), was calculated using the mass balance according to Eq. 1:

$$q_t = \frac{(C_o - C_t) V}{m} \quad (1)$$

where V is the adsorption medium volume (L), C_o is the initial concentration of asphaltene in the solution (mg/L), C_t is the concentration of asphaltene in the solution at time t (mg/L), and m is the mass of the nanoparticles (g). To confirm the trends of obtained results, all the experiments were performed three times and the average of the data was applied.

Adsorption Isotherms Models

Base on the trend of experimental data and the literature the adsorption isotherm models were chosen. Therefore, the isotherms for the asphaltene adsorption onto MNP@PDA were obtained using several models such as the Langmuir, Freundlich, modified Langmuir, and solid–liquid equilibrium (SLE) models. The expression of the models was illustrated in Table 1.

Table 1. Isotherm models expression

Isotherm model	Expression	Parameters
Freundlich	$q_e = K_F C_e^{1/n}$	$n, K_F ((\text{mg/g})(\text{L/mg})^{1/n})$
Langmuir	$q_e = q_m \frac{K_L C_e}{1 + K_L C_e}$	$q_m (\text{mg/g}), K_L (\text{L/mg})$
Modified Langmuir	$q_e = q_m \frac{K_s C_e^x}{1 + K_s C_e^x}$	$x, q_m (\text{mg/g}), K_s ((\text{L/mg})^{1/x})$

Adsorption Kinetics Models

The adsorption of asphaltene onto MNP@PDA was monitored for 12 h at an initial asphaltene concentration of 500 mg/L, and a temperature of 25 °C. Using the obtained data, the adsorption kinetics were modeled using several commonly used adsorption models such as pseudo-first-order, pseudo-second-order models, and double-exponential models (DEM) [24, 25]. The efficiency of the grafted adsorbent was evaluated by studying the adsorption kinetics. The studied kinetics models were summarized in Table 2.

Table 2. Kinetics models expression

Kinetics model	Expression	Parameters
Pseudo-first-order	$q_t = q_e (1 - e^{-K_1 t})$	$q_e (\text{mg/g}), K_1 (1/\text{min})$
Pseudo-second-order	$q_t = (K_2 q_e^2 t) / (1 + K_2 q_e t)$	$q_e (\text{mg/g}), K_1 (\text{g/mg} \cdot \text{min}^{-1})$
Double-exponential	$q_t = q_e - \frac{D_1}{m_a} \exp(-K_1 t) - \frac{D_2}{m_a} \exp(-K_2 t)$	$q_e (\text{mg/g}), K_1, K_2 (1/\text{h}), D_1, D_2 (\text{mg/g})$

Recycling Experiment

For desorption of asphaltene adsorbed on the surface of MNP@PDA, the nanoparticles were dispersed into 10 mL of chloroform/methanol (70/30 Vol. %) solution and shaken at 300 rpm for 4 h. After spending the desired time, the nanoparticles were collected using an external magnetic field (1.2 T) and washed repeatedly with an excessive amount of the solution. The concentration of asphaltene in the residual solution was determined using UV–vis spectrophotometer. After drying the nanoparticles in a vacuum oven at 40 °C, they were re-dispersed in a known concentration of asphaltene solution to investigate the adsorption ability of the retrieved removal agent. The amount of asphaltene desorbed during any elution process was determined by performing an appropriate mass balance. The recovery rate was calculated according to the following equation:

$$RE(\%) = \frac{C_r}{C_0 - C_e} \times 100 \quad (2)$$

where C_0 is the initial concentration of asphaltene in solution (mg/L), C_e is the equilibrium concentration of asphaltene (mg/L), and C_r is the concentration of asphaltene (mg/L) in eluent solution.

Results and discussion

Characterization of MNP@PDA

The chemical structure, morphology, size, and specific surface area of MNP@PDA could be found in the previous work [23]. Briefly, the average size of MNP@PDA was determined using TEM analysis to be about 10 nm, and the theoretical surface area was about 30 m²/g.

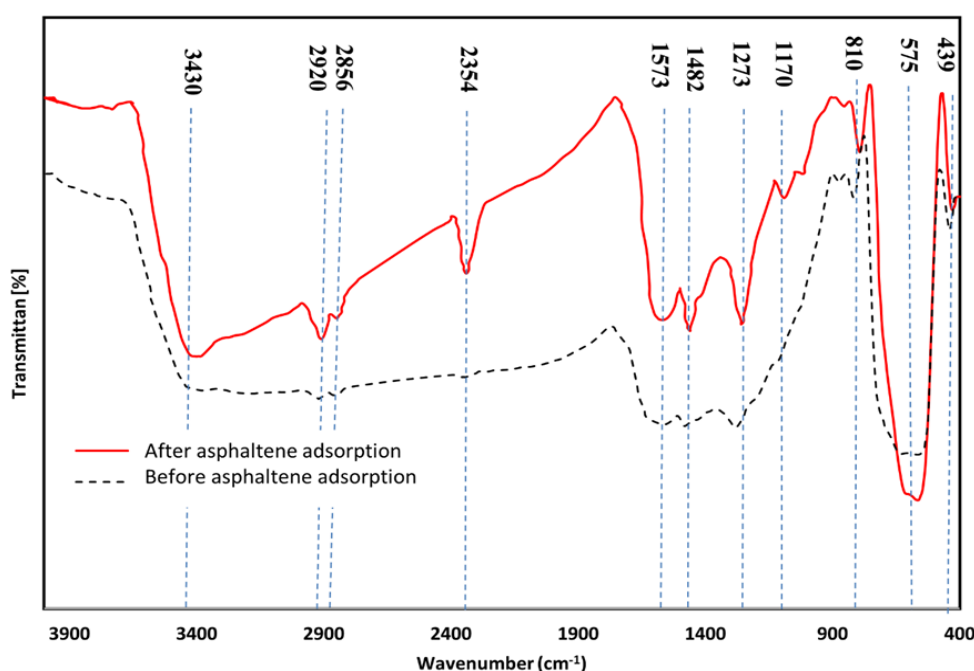


Fig. 2. FTIR spectra for MNP@PDA before and after asphaltene adsorption

The Fourier transform infrared (FT-IR) spectroscopy was also applied for revealing the presence of chemical interactions between the maghemite nanoparticles (MNPs) and polydopamine (PDA) [17, 23]. Therefore, in the present work, the characterization of the adsorbed material onto MNP@PDA was accomplished by FT-IR spectroscopy. FT-IR analysis of MNP@PDA [23], before and after asphaltene adsorption was conducted and shown in Fig. 2. According to the FT-IR spectrum of MNP@PDA before asphaltene adsorption, the peaks at wavenumbers 575 and 439 cm⁻¹ are attributed to the stretching vibration of Fe-O band in the MNP structure and the peak at wavenumber 3430 cm⁻¹ attributed to the absorption of water and hydroxyl groups on the surface of the nanoparticle [17]. The peaks at wavenumbers 1624, 1490, and 1292 cm⁻¹ are attributed to the overlapping of the resonance vibration of C-C in the aromatic ring with N-H bonding, the N-H stretching vibration, and stretching-bonding vibration C-O-H in polydopamine structure, respectively. Several new peaks have been created in the FT-IR spectrum of MNP@PDA after asphaltene adsorption, which is as follows: (1) the peak at 1273 cm⁻¹ is related to connectors ether and ester in the surface of nanoparticles, (2) the peak at 1460 cm⁻¹ belongs to the existence bending of the CH₃ and CH₂, (3) the peak at 2354

cm^{-1} absorption bands is related to C=O and C=N=O bonds is in conjunction with carboxylic connections or strong bond C=O, (4) the peak at 2856 cm^{-1} is attributed to the C-H bond, and (5) the peaks at 2920 and 3403 cm^{-1} are related to $\text{CH}_3\text{-CH}$ and N-H bonds, respectively, that the latter may be attributed to the sharing of electron between nitrogen atoms available in the asphaltene structure and hydrogen atoms located on the surface of the nanoparticles.

Adsorption isotherms

To obtain the maximum amount of asphaltene adsorbed onto MNP@PDA and determine the isotherm model constants, the adsorption experiments were carried out at different temperatures. The parameters of the models and obtained R^2 values from adsorption isotherms at different temperatures are reported in Table 3. The results show that the modified Langmuir type isotherm could cover the experimental data well with R^2 values of greater than 0.999. This result proves that the adsorption of asphaltene onto MNP@PDA occurs by multilayer adsorption. The adsorption isotherms of asphaltene according to the modified Langmuir model are shown in Fig. 3. There is a good agreement between the model predictions and the experimental data.

Table 3. Parameters of isotherms for asphaltene adsorption

Isotherm model	T (°C)	Model parameters		R^2		
		$1/n$	$K_F (\text{mg/g})(\text{L/mg})^{1/n}$			
Freundlich	25	0.3369	5.0292	0.9744		
	40	0.3377	5.1572	0.9736		
	60	0.3428	5.5842	0.9867		
Langmuir		$q_m (\text{mg/g})$	$K_L (\text{L/mg})$			
	25	50.13	0.0128	0.9942		
	40	51.25	0.0133	0.9949		
	60	56.17	0.0142	0.9883		
Modified Langmuir		x	$q_m (\text{mg/g})$	$K_S (\text{L/mg})^{1/x}$	R^2	
	25	0.7738	56.82	0.0253		0.9994
	40	0.7722	58.66	0.0254		0.9995
	60	0.6162	78.09	0.0356	0.9991	

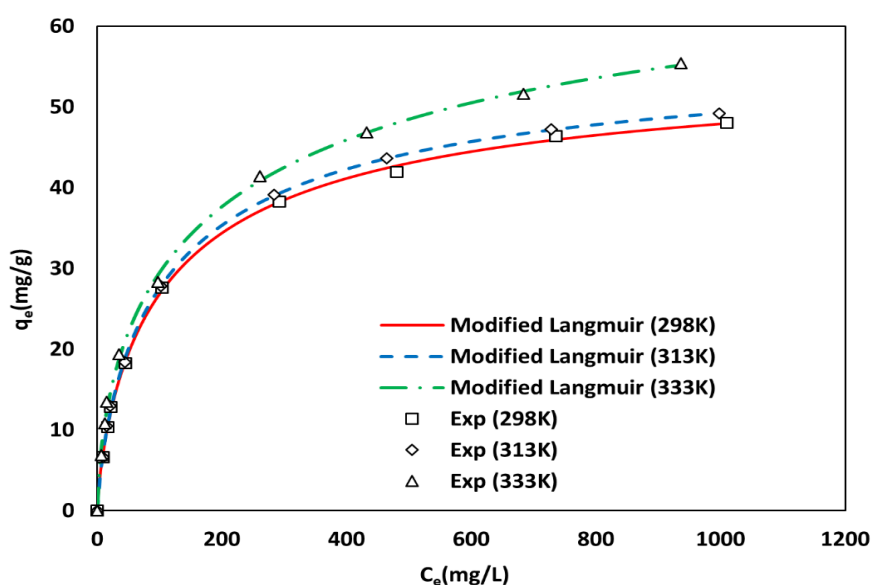


Fig. 3. Modified Langmuir model adsorption isotherm of asphaltene

Adsorption kinetics

The kinetics of the asphaltene adsorption onto MNP@PDA were modeled and the efficiency of the grafted adsorbent was evaluated using the three adsorption kinetics models. Fig. 4 shows the changes in the amount of asphaltene adsorbed onto MNP@PDA as a function of contact time. It can be concluded that the adsorption is very fast so that the equilibrium was achieved in less than 30 min. According to the obtained results, about 90% of removal of asphaltene happened within 30 min. The results (Fig. 4) demonstrate that the experimental data are fitted well by the double-exponential model. The double-exponential model describes well the mechanism of adsorption and mass transfer diffusion through the two-step kinetics of the adsorption: the rapid and the slow step. In this model, the parameters with subscripts 1 and 2 represent the rapid and slow steps, respectively. During the rapid step, most of the adsorbate diffuses toward the adsorbent, and adsorption happens within a few minutes, whereas on the second step, adsorption is more slowly. Based on the adsorption mechanism of DEM the rapid step involves both external and internal diffusion, however, the slow step is controlled only by the intraparticle diffusion.

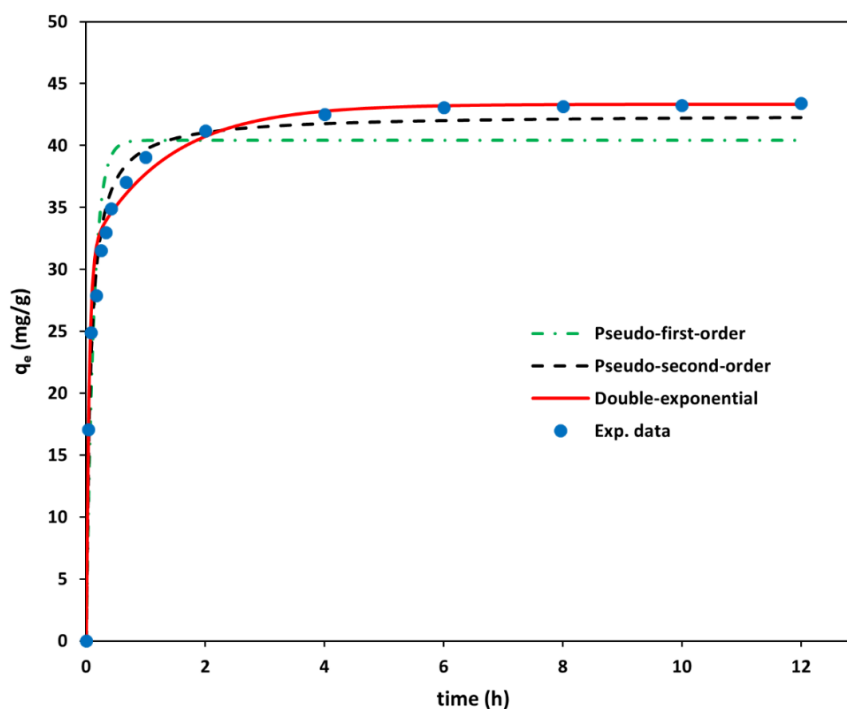


Fig. 4. Evaluation of kinetic models for the asphaltene adsorption onto nanoparticles

By using the experimental kinetic adsorption data (q_t vs. t) and applying the mathematical curve fitting method, the parameters of the model (D_1 , D_2 , K_{D1} , and K_{D2}) were determined at the optimum dose of nanoparticles ($m_a=10$ g/L). The models' parameters and R^2 -values are also reported in Table 4. By using the calculated parameters and derivation form of the DEM equation, the overall initial mass transfer rate (r_0) can be determined.

Desorption and reusability

The variation of adsorption and desorption efficiency concerning the cycle number is depicted in Fig. 5. As it can be seen in this figure, the adsorbed/desorbed cycle does not remarkably change during three cycles. Consequently, the synthesized nanoparticles could be

successfully reused at least three times only with a slight loss of their original adsorption efficiency. The result of the recovery test indicates that we can use polymer at the surface of the nanoparticle as recyclable adsorbents for asphaltene removal.

Table 4. Kinetic models parameters for asphaltene adsorption

Kinetic model	Parameters						R^2
	q_0 (mg/g)	m_0 (g/L)	K_1 (1/min)	D_1 (mg/L)	K_2 (1/min)	D_2 (mg/L)	
Double-exponential	43.3283	10	0.3409	307.739	0.0128	120.732	0.9881
Pseudo-second-order	q_e (mg/g)		K_2 (g/mg.min)				
	42.51		0.0021				0.9826
Pseudo-first-order	q_e (mg/g)		K_1 (1/min)				
	40.4171		0.1377				0.9097

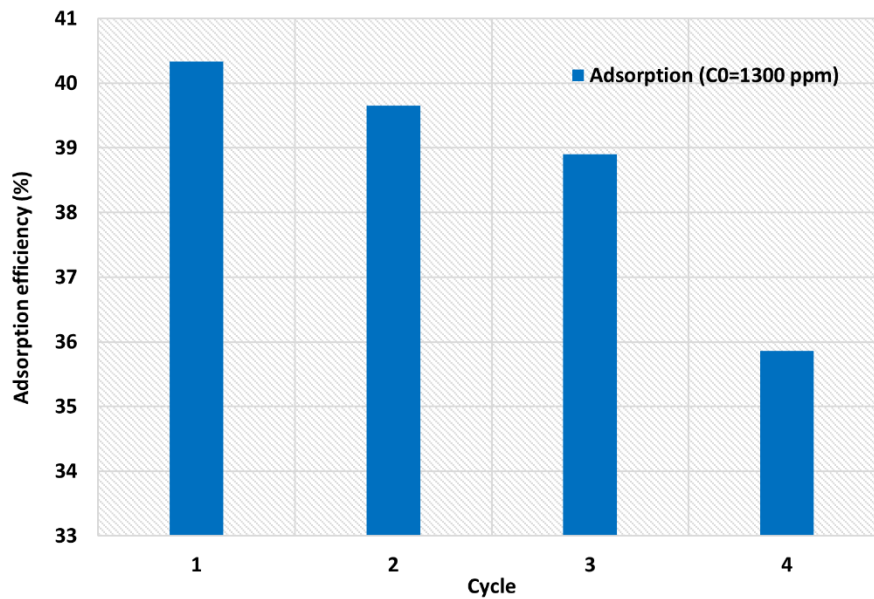


Fig. 5. Adsorption efficiency of asphaltene in adsorption/desorption cycles

Thermodynamic studies

The study of asphaltene adsorption onto nanoparticles from a thermodynamics point of view helps us to better understand the temperature effect on this process. By using Van't Hoff equations (Eqs. 3 and 4) thermodynamic studies of the adsorption process can be evaluated [26]:

$$\Delta G_{ads}^o = -RT \ln(K) \quad (3)$$

$$\ln(K) = -\frac{\Delta H_{ads}^o}{RT} + \frac{\Delta S_{ads}^o}{R} \quad (4)$$

where R is the universal ideal gas constant ($=8.314 \text{ J/mol.K}$), T is the temperature (K), and K is the adsorption equilibrium constant (dimensionless). The constant, K , can be expressed as $K_L C_s$, where K_L is the equilibrium Langmuir constant (L/mmol) and C_s is the solvent molar concentration (mM). The solvent molar concentration can be calculated from the density and molecular mass of toluene. The molecular mass of asphaltene in the range of 750-5000 g/mol was used to determine the thermodynamic parameters and also the equilibrium Langmuir constant. The calculated thermodynamic parameters for the sorption of asphaltene onto the

nanoparticle are given in Table 5. The calculated values of ΔG_{ads} are negative which indicates that the adsorption of asphaltene molecules on the nanoparticle surface is a spontaneous process. The positive values of the enthalpy indicated that asphaltene adsorption onto nanoparticles is endothermic whereas the positive ΔS° values confirmed the increased randomness at the solid–solute interface during adsorption.

Thermodynamic studies also showed that as temperature and asphaltene molecular mass increase, the standard Gibbs free energy decreases.

Table 5. Calculated parameters of van't Hoff equation for the adsorption of asphaltene onto nanoparticles at different temperatures

M_w (g/mol)	T (K)	K	$-\Delta G$ (kJ/mol)	$-\Delta H$ (kJ/mol)	ΔS (J/mol.K)	R^2
800	298	96687.61	28.45	-2.47	103.12	0.9926
	313	99709.10	29.96			
	333	106507.45	32.06			
2000	298	241719.04	30.72	-2.47	111.3	0.9926
	313	249272.76	32.35			
	333	268157.06	34.62			
4500	298	543867.84	32.73	-2.47	118.06	0.9926
	313	560863.71	34.46			
	333	603353.38	36.86			

Conclusion

In this work, kinetic and isotherm studies of asphaltene adsorption onto maghemite nanoparticle modified with natural polymers nano-layer were investigated experimentally. Characterization of the adsorbed asphaltene was accomplished using FT-IR. The FT-IR spectrum revealed the adsorption of asphaltene onto the modified nanoparticles. Among the adsorption isotherm models, the modified Langmuir type isotherm showed the best fitting to the adsorption experiment which indicated that the adsorption of asphaltene onto MNP@PDA occurs in monolayer. To study the kinetics of the adsorption of asphaltene onto MNP@PDA three models namely, pseudo-first- and -second-order model and double-exponential model were used. The results showed that the double-exponential model fits well with the experimental data. The obtained results also revealed that about 90% removal of asphaltene happens within 30 min. the reusability of MNP@PDA was examined using chloroform/methanol as stripping solution. The results showed successful reusability of the natural polymer-coated maghemite nanoparticle at least three times, for asphaltene adsorption from the prepared asphaltene-toluene solutions. Furthermore, in terms of thermodynamic, calculation of thermodynamics parameters (Gibbs free energy, entropy, and enthalpy) of asphaltene adsorption onto modified nanoparticles showed: (1) conceivability and spontaneity of adsorption process, (2) endothermic nature of the process for this nanoparticle.

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