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Research Article

Interaction and Adsorption of Hydrophobically Modified Polyacrylamide on Silica and Asphaltene Surfaces

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The adsorption of polymers affects the cost and oil recovery in oil reservoir exploitation and the flocculation effect in the treatment of oil sand tailings. The adhesion and adsorption of a hydrophobically modified polyacrylamide (HMPAM), i.e., P(AM-NaAA-C_{16}DMAAC), on silica and asphaltene were investigated using surface force measurements, thermodynamic analysis and quartz crystal microbalance with dissipation (QCM-D) measurement. Our study indicates that HMPAM polymer has strong interaction with both silica and asphaltene. The adhesion force of HMPAM on silica was stronger than that on asphaltene surface. Consistently, the adsorption of HMPAM was also greater on silica surface, with a more rigid layer formed on the surface. For HMPAM/silica system, the attractive interaction and the strong adhesion are mainly driven by the hydrogen bonding and electrostatic interaction. For HMPAM/asphaltene system, it is mainly due to hydrophobic interaction between the long hydrocarbon chains of HMPAM and asphaltene. Furthermore, continuous adsorption of HMPAM was detected and multiple layers formed on both silica and asphaltene surfaces, which can be attributed to the hydrophobic chains of HMPAM polymers. This work has illustrated the interaction mechanism of HMPAM polymer on hydrophilic silica and hydrophobic asphaltene surfaces, which provide insight into the industrial applications of hydrophobically modified polymer.

1. Introduction

Recently, special attention has been focused on the hydrophobically modified polyacrylamide containing relatively low amounts of hydrophobic monomers (~2 mol%). Different hydrophobic monomers were reported, such as di-alkyl substituted acrylamides di-*n*-propylacrylamide, di-*n*-octylacrylamide [1], *N,N*-dihexylacrylamide or *N,N*-diphenylacrylamide [2], *N*-octadecylacrylamide [3], sodium 2-acrylamido-tetradecane sulfonate [4], 3-acrylamido-2-hydroxypropyltriakylammonium chloride [5], poly(propylene glycol) monomethacrylate [6], isomeric 11-acrylamidoundecanoic acid [7], stearyl methylacrylate [8], sodium 9-(and 10)-acrylamidostearate [9], etc. Due to the intramolecular and intermolecular interaction between the hydrophobic groups, the enlargement of hydrodynamic volume of polymers increases the viscosity of hydrophobically modified polyacrylamide aqueous solutions, and intermolecular association may further enhance the viscosity [10]. The aqueous solution of these polymers is expected to show special properties, including temperature, salt, and shear resistances [11, 12]. The viscosity of hydrophobically modified polyacrylamide solution does not decrease at high salinity when its concentration is larger than the critical aggregation concentration [13]. For example, a kind of hydrophobically modified polyacrylamide based novel functional polymer (RH-4) at the concentration of 2000 mg/L has lower the apparent viscosity as NaCl concentration increase until ~2000 mg/L but it increased with the NaCl concentration at 2000–8000 mg/L [14]. In a semi-dilute solution of polyacrylamide with hydrophobic *t*-octylacrylamide group (0.5 wt.%), the zero-shear viscosity decreases with salt concentration at low salt conditions (0~0.3 M KCl), while it displayed a monotonically increase with salt concentration at high salt conditions (0.3~4 M KCl) [15]. NaCl was found to promote the association and adsorption of a hydrophobically modified polyacrylamide on kaolin particles, resulting in higher flocculation efficiency [16]. The adsorption isotherms of a hydrophobically associating polyacrylamide on K-montmorillonite and on siliceous minerals, has shown different behaviors compared with a nonassociating

polymer [17]. Another kind of hydrophobically modified polyacrylamide with monomers of acrylamide and 2-(methacryloyloxyl) ethylhexadecyldimethylammonium bromide displays the adsorption of multilayer on natural sand [18]. Guo et al., systematically studied the flow behavior through porous media and microdisplacement performances of hydrophobically modified partially hydrolyzed polyacrylamide (HMHPAM) [19–21]. They found that hydrodynamic sizes of HMHPAMs were much more sensitive to the polymer concentration, filtration pressure, and associating monomer content than partially hydrolyzed polyacrylamide, and had better an efficiency than glycerol and HPAM for displacing the residual oil trapped in the "dead" ends of flow channel at the same viscosity. With the excellent properties, hydrophobically modified polyacrylamide polymers have been applied in enhanced oil recovery (EOR) of oil reservoir exploitation [7, 22–26], emulsified oil removal from aqueous solution [27] and oil sand tailings treatment [28].

To be noted, the adsorption behavior of polyacrylamide directly affects its adsorption loss and the related cost [29, 30]. The adsorption retention loss of polymer solution in reservoir pores should not be too large, so that the polymer solution in deep reservoir can keep sufficient concentration, and the reservoir near the injection well will not be blocked by large amount of polymer adsorption, which will affect the oil recovery. Also, the adsorption of flocculants would influence the flocculation efficiency in oil sand tailings treatment. Therefore, the adsorption and interaction mechanism of polymer at different solid/liquid interfaces are important for the application of hydrophobically modified polyacrylamide.

In this work, a hydrophobically modified polyacrylamide (HMPAM) was synthesized with a hydrophobic monomer hexadecyl dimethyl allyl ammonium chloride (~0.3 mol%). The interaction and adsorption behaviors of HMPAM on silica and asphaltene surface were investigated using colloidal probe atomic force spectroscopy, thermodynamic analysis of interaction energy, and quartz crystal micro-balance with dissipation (QCM-D) monitoring technique. Our work revealed the fundamental interaction mechanisms between HMPAM and silica and asphaltene surface, which will benefit the development of polymers in oil reservoir exploitation and oil sand tailings treatment.

2. Experimental

2.1. Materials. HMPAM were synthesized using the procedures described in our previous work [31, 32]. Diiodomethane, glycerol, hydrogen peroxide and sulfuric acid were purchased from Fisher Scientific (China). Ethanol, methylbenzene, Sodium dodecyl sulfate (SDS) and sodium hydroxide (NaOH) were purchased from Shanghai Aladdin Bio-Chem Technology Co., Ltd. Toluene was purchased from Nanjing Reagent (China). Octadecyltrichlorosilane (OTS, ≥90%) was purchased from Sigma Aldrich, China. Water was purified by Milli-Q system. Nitrogen at 99.99+% purity levels was used to dry the surfaces. Silica sensors were purchased from Q-sense (Gothenburg, Sweden).

2.2. Preparation of Asphaltene Surfaces. Asphaltene was extracted from vacuum distillation feed Athabasca bitumen following the previous procedure [33, 34]. The asphaltene sample was dissolved in toluene at the concentration of 0.5 wt.%. The asphaltene-toluene solution was sonicated for 10 min and filtered through a 0.2 μm polytetrafluoroethylene filter (Nalgene) before use. Subsequently, the silica wafer was immersed in the piranha solution (7 : 3 vol./vol. concentrated H_2SO_4 : 30% H_2O_2) for 20 min and washed with ethyl alcohol. Then, the silica surface was rinsed thoroughly with copious amount of Milli-Q water and blown dry by nitrogen gas. Then the cleaned silica wafer was immersed in dilute asphaltene-toluene solution (0.005 wt.%) for 12 h at 25°C. Finally, the prepared asphaltene surface was washed by toluene and dried by nitrogen gas before use.

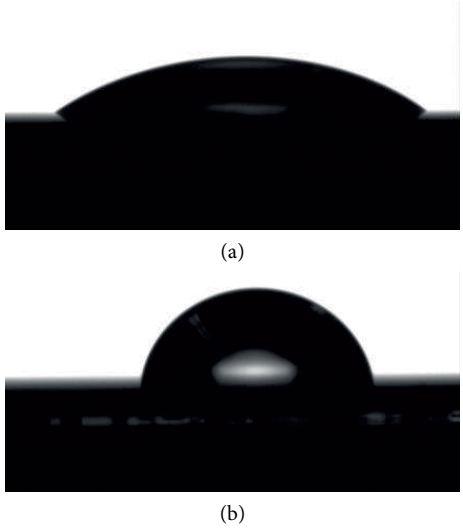
The asphaltene sensors for QCM-D were prepared by spin-coating asphaltene solution onto OTS-treated silica sensors following the procedures shown in the literature [31]. Briefly, silica sensor was washed with 2% SDS, milli-Q water and UV/ozone followed the cleaning protocols provided by the Biolin Scientific. OTS was deposited on silica wafer by the vapor deposition as reported [33, 35]. Several drops of asphaltene were spin-coated on the OTS-treated silica sensor. Then the sensor was placed in vacuum overnight to remove any residual toluene.

2.3. AFM Force Measurements. The interaction forces measurements between HMPAM and silica or asphaltene surface were conducted using an MFP-3D AFM instrument (Asylum Research, Santa Barbara, USA). The HMPAM functionalized silica probes were prepared under the catalysis of EDC/NHS as described in the literature [36]. The force measurements were conducted, also, as in the literature [36]. The aqueous solution for the AFM force measurements was 100 mM KCl. Force mapping mode was conducted to analyze the interactions for at least 100 times at more than 3 different areas of the silica or asphaltene surfaces.

2.4. Characterization. The surface morphologies of silica and asphaltene surface before and after adsorption of HMPAM were imaged by the tapping mode. The surface wettability was characterized by KRUSS DSA100 (Hamburg, Germany) instrument. The errors of the contact angles at more than 3 different areas were less than ±2%, and the average contact angles were used. The QCM-D analysis was carried out to measure the in-situ adsorption kinetics of HMPAM using a Q-sense E1 system (Q-sense, Gothenburg, Sweden).

3. Results and Discussion

Figure 1 shows the different wettability of silica and asphaltene surfaces. The water contact angles of silica and asphaltene surfaces were 36.7 ± 1.5° and 82.2 ± 2.1°. Asphaltene is more hydrophobic than silica surface. Both silica and asphaltene surfaces were submerged in 20 mg/L HMPAM of 100 mM KCl solution. After 3 hours, the surfaces were washed with 100 mM KCl solution, then the morphology of the surfaces was observed after being dried by nitrogen gas. In Figure 2, the



(a)

(b)

Figure 1: Water contact angle on (a) silica and (b) asphaltene surfaces.

AFM imaging shows silica exhibits a smooth and uniform morphology, and the root-mean-square surface roughness (R_q) is only 0.06 nm. After adsorption of HMPAM, the R_q of silica surface increases to 0.75 nm. Differently, asphaltene surface is rougher with the R_q of 0.68 nm. After adsorption of HMPAM, the asphaltene surface becomes slightly smoother with R_q of 0.61 nm, which might be due to the large amount of HMPAM adsorption.

To measure the interaction between HAMPAM and silica and asphaltene, force measurement was carried out as shown in Figure 3. It shows an attraction between HMPAM and silica surface at up to 10 nm distance during approaching in 100 mM KCl solution (Figure 3(a)). It is also attraction between HMPAM and asphaltene surface (Figure 3(b)). But the attraction distance on asphaltene is longer up to 20 nm, which could attribute to the larger roughness of asphaltene surface and the hydrophobic interaction between the hydrophobic asphaltene surface and HMPAM. During retraction, jump-out behaviors were detected on both silica and asphaltene surfaces, indicating the adhesion between HMPAM and silica/asphaltene surface. Force mapping was carried out on silica and asphaltene surfaces to statistically obtain the adhesion forces. In Figure 3(c), the F_{adh} of HMPAM on silica surface was 3.30 ± 0.56 nN, slightly larger than on asphaltene surface at 2.96 ± 0.41 nN. This result indicates that the adhesion between HMPAM and silica surface is stronger than that between HMPAM and asphaltene surface.

The curved probe surface and flat substrate can be correlated according to the Johnson–Kendall–Roberts contact mechanics model [37, 38] as follows.

$$F_{adh} = 1.5\pi R W_{adh}, \tag{1}$$

where W_{adh} is the adhesion energy (mJ/m^2), R the radius of the probe (m).

The measured adhesion energies of HMPAM are 0.350 mJ/m^2 on silica and 0.278 mJ/m^2 on asphaltene, respectively. Generally, the adhesion interaction can be ascribed to van der Waals force, electrostatic interaction, hydrogen-bonding and hydrophobic interaction. The van der Waals interaction is generally weak in the aqueous medium and only works in a few nanometers of the two surfaces. The Debye length, describing the range of the electric double layer was 0.96 nm in 100 mM KCl. Thus, for hydrophilic silica surface, the adhesion can be induced by both electrostatic interaction between amine group in HMPAM and silica surface, and hydrogen bonding interaction between the C=O, NH_2 groups of HMPAM and the –OH group on silica surface. Besides, the silica surface can be mildly hydrophobic with water contact angle of $36.7 \pm 1.5°$, according to the literature [39, 40]. There could be hydrophobic attraction between HMPAM and silica surface. For asphaltene surface, hydrophobic interactions may dominate the adhesion, which is between the hydrophobic domains on asphaltene surface and the long hydrocarbon chains of HMPAM. It indicates that HMPAM has the capability of attracting with both silica and asphaltene by various interactions.

Surface thermodynamic characterization of the HMPAM coated silica and asphaltene surfaces has been assessed to better understand the interaction of HMPAM on these surfaces. According to the Van Oss-Chaudhury-Good theory, the surface free energy of solid is composed of the non-polar part and polar part [41, 42]. The surface energy of solid can be calculated as follows.

$$\gamma_s = \gamma_s^{LW} + \gamma_s^{AB} = \gamma_s^{LW} + 2\left(\gamma_s^+ \gamma_s^-\right)^{1/2}, \tag{2}$$

where γ_s (mJ·m^{-2}) is the total surface free energy of solid, γ_s^{LW} (mJ·m^{-2}) the nonpolar part, referring to LIfshitz-van der Waals, γ_s^{AB} (mJ·m^{-2}) the polar part, referring to short range acid-base interaction forces, γ_s^+ (mJ·m^{-2}) the acid part and γ_s^- (mJ·m^{-2}) the base part. The interface free energy of solid and liquid is expressed:

$$\gamma_{sl} = \gamma_s + \gamma_l - 2\left[\left(\gamma_s^{LW}\gamma_l^{LW}\right)^{1/2} + \left(\gamma_s^+\gamma_l^-\right)^{1/2} + \left(\gamma_s^-\gamma_l^+\right)^{1/2}\right]. \tag{3}$$

Young's equation [43] is as follows:

$$\gamma_s = \gamma_{sl} + \gamma_l \cos\theta, \tag{4}$$

$$\gamma_{sl} = \gamma_s^+ \gamma_s^- W_a, \tag{5}$$

where γ_l (mN/m) is the surface tension of liquid, γ_{sl} (mN/m) the interfacial tension of solid and liquid, γ_l^+ (mN/m) the acid part of liquid, γ_l^- (mN/m) the base part of liquid, $\theta(^\circ)$ the contact angle, the W_a (J) is a adhesion work.

Thus, combining the Equations (3), (4), and (5), there would be Equation (6):

$$W_a = \gamma_l(1 + \cos\theta) = 2\left[\left(\gamma_s^{LW}\gamma_l^{LW}\right)^{1/2} + \left(\gamma_s^+\gamma_l^-\right)^{1/2} + \left(\gamma_s^-\gamma_l^+\right)^{1/2}\right]. \tag{6}$$

Surface free energy can be calculated based on contact angles of three model liquids by the Equation (6). The surface tensions of three liquids, i.e., diiodomethane, glycerol and deionized water, were shown in Table 1. Contact angles of the model liquids on silica and asphaltene surfaces before and after the

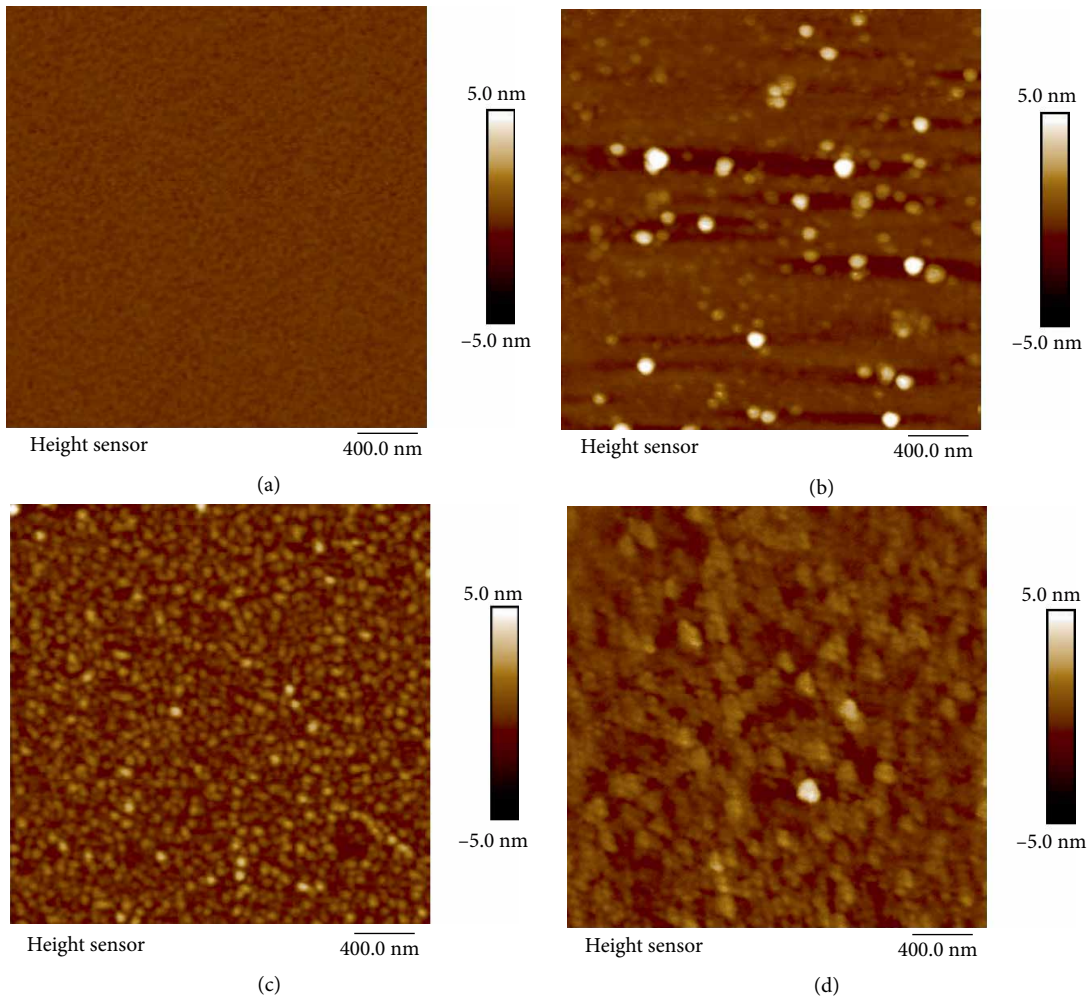


Figure 2: AFM images of different samples: (a) silica, (b) silica after adsorption of HMPAM, (c) asphaltene, and (d) asphaltene after adsorption of HMPAM.

adsorption of HMPAM were listed in Table 2. The group of equations was solved when the contact angles were substituted to Equation (6) to estimate the surface free energy and its components. The values of surface tension and components of the samples were collected in the Table 3.

The surface free energy of silica is 57.40 mJ/m^2, which agrees with the literature [45]. The γ_s^- of silica is 58.10 mJ/m^2, while that of asphaltene surface is 1.42 mJ/m^2. The γ_s^- is due to H-bonds, and the greater the γ_s^- is, the sample surface is more hydrophilic [42, 43]. It consists with the higher hydrophilicity of silica than asphaltene. After adsorption of HMPAM, the γ_s of asphaltene increased to 58.44 mJ/m^2, while its γ_s^- remarkably increased to 33.03 mJ/m^2. In contrast, the γ_s of silica increased slightly to 63.32 mJ/m^2. Its γ_s^{AB} increased from 12.57 mJ/m^2 to 19.64 mJ/m^2, while the γ_s^{LW} decreased from 44.83 mJ/m^2 to 43.68 mJ/m^2. To be noted, the surface free energy of both silica and asphaltene increased.

The free energy ΔG(mJ/m^2) of interactions between two solid surfaces can be calculated according to the following Equations [46].

Table 1: Surface tension and components (mN/m) of three model liquids [44].

Liquid	γ^{LW}	γ^+	γ^-	γ^{AB}	γ
Diiodomethane	50.80	0.00	0.00	0.00	50.80
Glycerol	34.00	3.92	57.40	30.00	64.00
Water	21.80	25.50	25.50	51.00	72.80

$$\Delta G_{adh} = \Delta G^{LW} + \Delta G^{AB}, \tag{7}$$

$$\Delta G^{LW} = 2 \times \left[\left(\gamma_w^{LW}\right)^{1/2} - \left(\gamma_2^{LW}\right)^{1/2}\right] \times \left[\left(\gamma_1^{LW}\right)^{1/2} - \left(\gamma_w^{LW}\right)^{1/2}\right], \tag{8}$$

$$\begin{aligned}\Delta G^{AB} =& 2 \times \left\{\left(\gamma_w^+\right)^{1/2} \times \left[\left(\gamma_1^-\right)^{1/2} + \left(\gamma_2^-\right)^{1/2} - \left(\gamma_w^-\right)^{1/2}\right]\right. \\ &\left. + \left(\gamma_w^-\right) \times \left[\left(\gamma_1^+\right)^{1/2} + \left(\gamma_2^+\right)^{1/2} - \left(\gamma_w^+\right)^{1/2}\right] - \left(\gamma_1^+\gamma_2^-\right)^{1/2} - \left(\gamma_1^-\gamma_2^+\right)\right\},\end{aligned} \tag{9}$$

Table 2: Contact angles of three model liquids on different surfaces.

Different surfaces	Contact angle (°)		
	Diiodomethane	Glycerol	Water
Asphaltene	55.6	57.9	82
Silica	28.5	27.5	36.7
HMPAM on asphaltene	34.5	29.2	34.3
HMPAM on silica	31.3	15.7	22

Table 3: Surface free energy (mJ/m^2) of different surfaces.

Different surfaces	γ_s^{LW}	γ_s^+	γ_s^-	γ_s^{AB}	γ_s
Asphaltene	31.12	3.48	1.42	4.45	35.57
Silica	44.83	0.68	58.10	12.57	57.40
HMPAM on asphaltene	42.27	1.98	33.03	16.17	58.44
HMPAM on silica	43.68	2.51	38.4	19.64	63.32

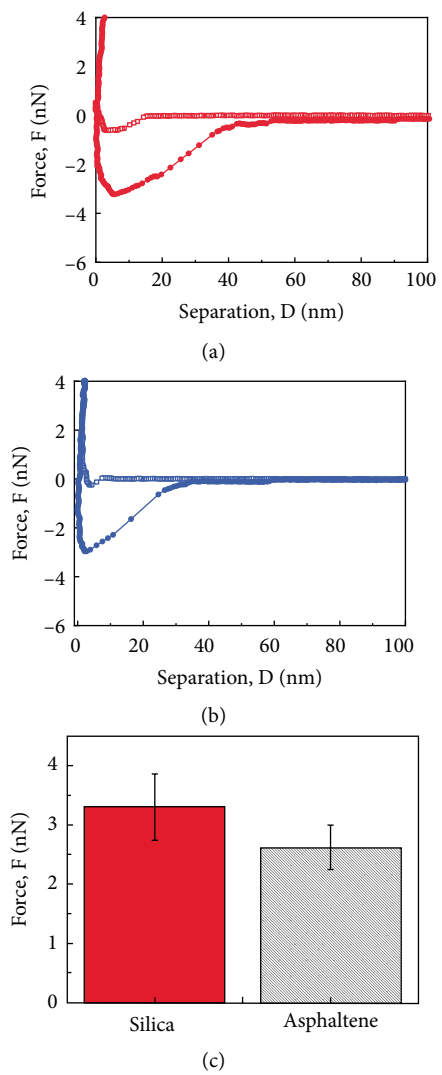


Figure 3: Typical force-distance profiles of the HMPAM-coated silica probe on surfaces in 100 mM KCl solution: (a) silica, (b) asphaltene, and (c) summary of the adhesion.

Table 4: Free energy of interaction (mJ/m^2) between surfaces.

Surfaces	ΔG^{LW}	ΔG^{AB}	ΔG_{adh}
Two asphaltene surfaces	−1.65	−49.14	−50.80
Two silica surfaces	−8.21	43.48	35.26
Asphaltene and silica	−3.69	−16.22	−19.90
HMPAM adsorbed silica and asphaltene	−3.53	−12.63	−16.16
Two HMPAM adsorbed silica	−7.53	−30.68	−38.21
HMPAM adsorbed silica and silica	−7.86	−41.13	−48.99
Two HMPAM adsorbed asphaltene surfaces	−6.72	−33.67	−40.39
HMPAM adsorbed asphaltene and HMPAM adsorbed silica	−7.11	−33.39	−40.50

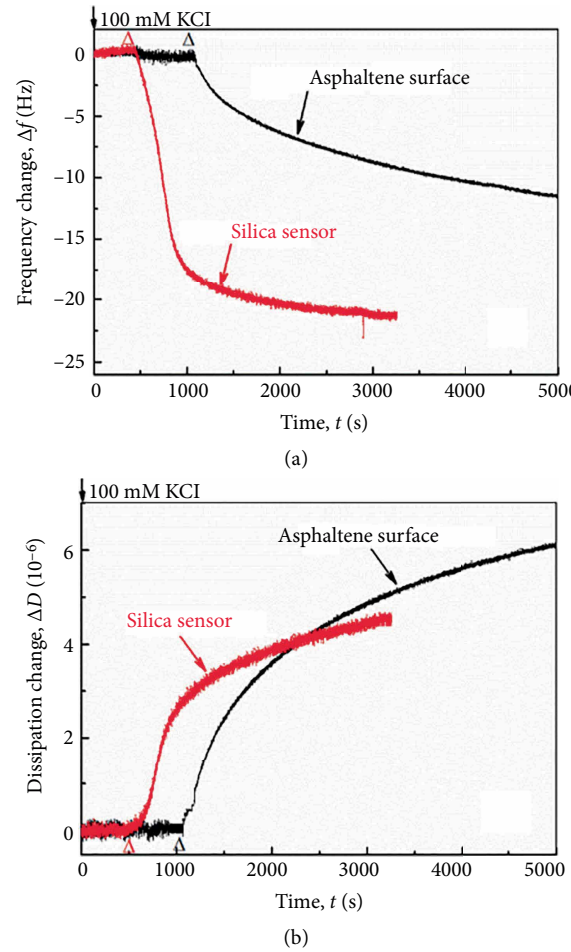


Figure 4: Changes of (a) frequency Δf and (b) dissipation ΔD obtained by QCM-D associated with adding 20 mg/L HMPAM in 100 mM KCl solution on silica and asphaltene sensors. (Δ-start injection).

where γ_1 and γ_2 are the surface energy parameters of two solid surfaces, and γ_w is the surface tension of water. When the value of ΔG is negative, the adhesion process will occur spontaneously. The more negative the value of ΔG is, the more likely the adhesion process happens. In Table 4, the ΔG of the two

silica surfaces is positive, and that of the two asphaltene surface is negative, implying that the adhesion of silica would not happen, and adhesion between two asphaltene surfaces could occur spontaneously. The ΔG_{adh} between silica and asphaltene is $-19.90\,mJ/m^2$ indicating that the coating of asphaltene on silica could occur.

The values of ΔG_{adh} between HMPAM adsorbed silica and asphaltene, silica or HMPAM adsorbed silica, are all negative, suggesting that all the above pairs of surfaces would adhere spontaneously. The absolute value of ΔG_{adh} between HMPAM adsorbed silica and silica is the largest. The values of ΔG_{adh} between HMPAM adsorbed asphaltene and HMPAM adsorbed asphaltene or HMPAM adsorbed silica are both negative. Both the thermodynamic calculations and AFM force measurements demonstrate that the adhesions of HMPAM on silica and asphaltene surfaces are spontaneous processes.

The adsorption of HMPAM on silica and asphaltene surfaces in 100 mM KCl were determined by QCM-D to verify the interaction mechanism. Figure 4(a) shows, the changes of frequency (Δf) of asphaltene and silica sensors decreased from the baseline to −14.6 Hz, and −21.5 Hz, respectively, indicating the adsorption amount of HMPAM on the silica surface was larger than that on the asphaltene surface. The adsorption time of HMPAM on asphaltene was longer which means the adsorption rate of HMPAM on asphaltene was slower than that on silica sensor. Meanwhile in Figure 4(b), the changes of dissipation (ΔD) of asphaltene surface (7.1×10^{-6} at adsorption equilibrium) was larger than that of silica (5.0×10^{-6} at adsorption equilibrium), indicating that the adsorption layer was less dissipative on silica surface. This result agrees with stronger interaction between HMPAM and silica surface by the AFM force measurement. Attractive hydrophobic force between HMPAM polymers led to continuous adsorption and formed multiple layers of HMPAM, which induces higher adsorption amount compared with the commercial PAM in previous study [36].

4. Summary and Conclusions

In this work, the adsorption and adhesion of a hydrophobically modified polymer HMPAM on silica and asphaltene was investigated using surface force measurements, thermodynamic analysis and QCM-D measurement. The surface force measurements and thermodynamic analysis of interaction energy have clearly shown the HMPAM have strong attractive interaction with both silica and asphaltene. The results show that the HMPAM polymer has strong attractive interaction with both silica and asphaltene. The adhesion force of HMPAM on silica surface (3.30 ± 0.56 nN) is stronger than that on asphaltene surface (2.96 ± 0.41 nN). QCM-D measurement suggests the adsorption amount of HMPAM on the silica surface (Δf of −21.5 Hz) was larger than that on the asphaltene surface (Δf of −14.6 Hz). And the adsorption layer was less dissipative on silica surface (ΔD of 5.0×10^{-6}), agreeing with the stronger adhesion force. On silica surface, the attraction and adhesion can be mainly induced by (1) hydrogen bonding between the C=O, NH_2 groups in HMPAM and the –OH group of silica surface; (2) electrostatic interaction between HMPAM and silica surface. On asphaltene surface, the attraction and adhesion may be attributed to hydrophobic interactions between the long hydrocarbon chains of HMPAM and asphaltene. Furthermore, it was revealed that HMPAM performed continuous adsorption and formed multiple layers on both silica and asphaltene surfaces, which could be attributed to the hydrophobic chains of HMPAM polymers. This work has illustrated the interaction mechanism of HMPAM on hydrophilic silica and hydrophobic asphaltene surfaces, which provide insight into the development of polymers for applications in oil reservoir exploitation and oil sand tailings treatment.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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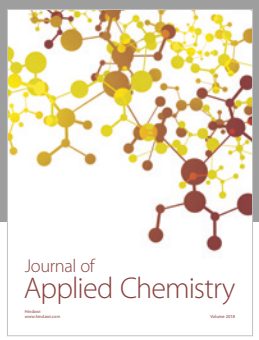

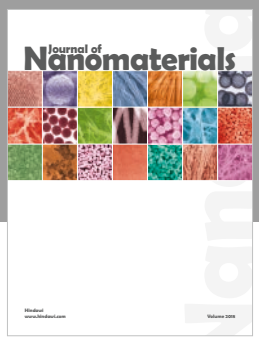

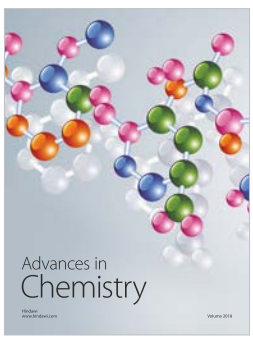

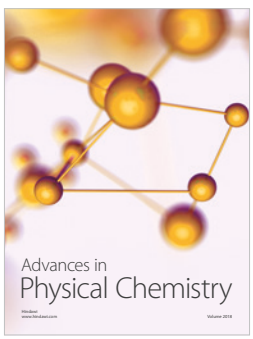

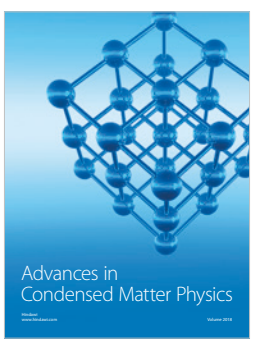

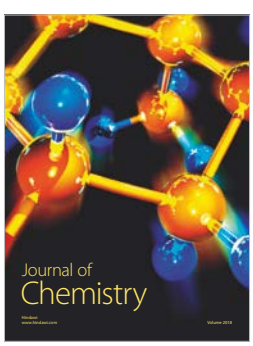

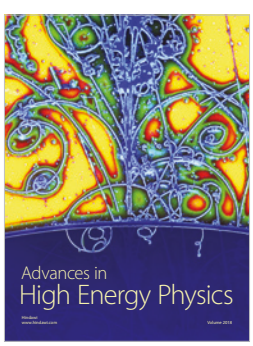

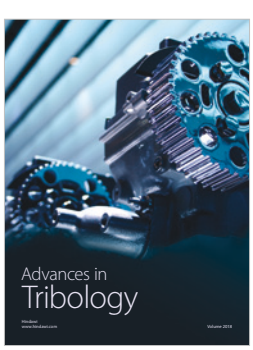

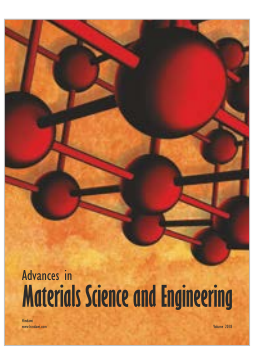

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