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Effect of Temperature and PH on Wettability Alteration and Adsorption of Amphiphilic Polymer-Coated SiO_2 Nanoparticle on Oil-Wet Porous Media

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Abstract: Wettability alteration of the oil-wet reservoir rocks to the water-wet state is an important factor for enhanced oil recovery (EOR). One of the main challenges of applying nanoparticles for wettability alteration is related to the colloidal stability and poor adsorption of the nanofluids in the harsh conditions of the reservoirs. In the present work, comparative studies were followed between polymer-coated silica nanoparticles by polyethylene glycol (Mn ~ 2000) and amphiphilic polymer-coated silica nanoparticles by polyethylene glycol (Mn ~ 2000 or 5000) and propyl chains to determine wettability alteration and adsorption of these modified nanoparticles on porous media. Water contact angle and UV–Vis adsorption measurements showed which the efficiency of amphiphilic polymer-coated nanoparticles depends on the wettability state of glass and it was improved significantly by oil-wet glass substrates due to the existence of the hydrophobic polymer on the surface of the nanoparticles. Moreover, effective parameters including temperature, and pH were studied. The better performance was obtained for the modified silica nanoparticles by polyethylene glycol (Mn ~ 5000) and propyl chains at 1000 ppm concentration in pH of 6 and the temperature range of 25-55°C. Our study demonstrated amphiphilic polymer-coated silica nanoparticles can be considered as a promising agent which has the potential for EOR purposes.

Keywords: Wettability, Adsorption, Silica Nanoparticle, Enhanced Oil Recovery

1. Introduction

Hydrocarbon is the main energy source which is expected to remain as the primary source of energy in the coming decades [1-3]. On average, about one-third of the oil of the reservoir rocks can be extracted by the secondary recovery method (conventional water injection into oil reservoirs) [1]. Because of the non-renewability of this energy and the limitation to hydrocarbon production, it is essential and inevitable to study and develop methods to recover the residual oil from the reservoir rock [2]. Various methods for improving the recovery of crude oil have been performed which is called enhanced oil recovery (EOR). EOR studies have been focused on interfacial tension reduction between water and oil [3], viscosity control [4], and wettability alteration of the reservoir [5]. Recently, using nanoparticles has attracted significant interest for EOR purposes [6-10]. These nanoparticles can penetrate small pores of the reservoir rocks to alter the wetting of rock from an oil-wet state to a strongly water-wet one [11]. Wettability alteration of the rock to a water-wet state can substitute the stored oil in the matrix pore scale by the waterflood and consequently increases the oil production [12].

The stability of the nanoparticles has a direct impact on adsorption as well as the wettability alteration of the reservoir rocks. In the harsh conditions of the oil reservoirs like acidic pH, electrostatic repulsive forces between nanoparticles reduce, which consequently unstable nanoparticle suspension causes agglomeration and precipitation and this process decreases the efficiency of nanoparticles to wettability alteration of the rocks [9, 10]. To overcome the challenge which it was mentioned, adding a surfactant to the nanofluid has already been studied [13-16]. The major disadvantages of using surfactants, especially in high concentrations, forming micelles can happen by the hydrophobic tails of surfactants

which reduces the stability of the nanofluid [14]. On the other hand, surfactants would be adsorbed and plug small pores of the rocks which reduces the efficiency of the nanoparticles to penetrate the pore space of the rocks [17]. ShamsiJazeyi and et al. [18] suggested modified nanoparticles by polymers can improve the performance of the nanoparticles in the harsh conditions of the oil reservoirs. Using polymers as a modifier can stabilize the nanoparticle suspension by the steric repulsion mechanism and based on the polymer types, it can improve the interfacial tension reduction, viscosity control, and wettability alteration.

In this work, we systematically studied the wettability alteration and adsorption of polymer-coated and amphiphilic polymer-coated nanoparticles based on effective parameters, including temperature and pH. The main purposes of this study were to focus on the effects of molecular weights and wettability properties of the polymers for modified nanoparticles, specifically, the effect of the hydrophobic polymer (propyl chains) on adsorption and wettability alteration of the substrates. To better simulate the reservoir rocks, the fatty acid (palmitic acid) was used for the treatment of the glass to an oil-wet state [19, 20].

2. Experimental Details

2.1. Materials

A solid-glass bead (borosilicate, diam. 3 mm, Sigma-Aldrich) was applied as simulated porous media. Non-porous silica nanoparticles (AEROSIL ® 200) were used with a specific area and average primary diameter of 200±25 m^2/g and 12 nm, respectively. Polyethylene glycol methyl ether (PEG1, averages Mn ~ 2000, Sigma-Aldrich) and polyethylene glycol methyl ether (PEG2, averages Mn ~ 5000, Sigma-Aldrich) were used as a hydrophilic agent and for linking covalently to the silica nanoparticle surface, it was functionalized by 3-glycidoxypropyltrimethoxysilane (3-GTO, Sigma-Aldrich, 98%). As a hydrophobic agent, trimethoxy (propyl) silane (C3S, Sigma-Aldrich, 97%) was used. Other chemicals were used acetic acid (Merck, glacial, 100.0%), n-hexane (Merck, 99.9%), acetonitrile (Ameretat Shimi, 99.9%), sulfuric acid (Merck, 98%), hydrogen peroxide (Merck, 30%), acetone (Merck, 99.9%), ethanol (Merck, 99.9%), and palmitic acid (CARLO EBRA, 99%).

2.2. Samples Preparation

Functionalizing silica nanoparticles with polymer was followed by our previous work [28, 33]. Next Modified nanoparticles were dispersed by magnetically stirred for 1 h and then were homogenized by using the ultrasonic bath for 30 min. The glass beads were washed ultrasonically for 30 min in acetone, ethanol, and distilled water, respectively. For rendering the surface of the glass to the oil-wet state, the glasses were refluxed in the piranha solution at 250°C for 24 h (3: 1 concentrated (98%) sulfuric acid and (30%) hydrogen peroxide). This treatment is vital to remove the organic matter and adding the hydroxyl group to the surface of the glass and this process alters the surface of the glass to a strongly water-wet state [22-24]. Finally, the oil-wet glasses were washed with ethanol and distilled water to remove any traces of physisorbed fatty acid on the surface of the glasses.

The prepared substrates (glasses or oil-wet glasses) were immersed in the modified nanofluids at room conditions in a static test. For avoiding to adsorption of the modified nanoparticles by gravity, the modified nanofluids were stirred smoothly (60 rpm) during the treatment. Graton and Fraser [25] have studied the porosity of variable packing arrangements of uniform spheres and as they indicated, irregular compact packing of uniform spheres is the rhombohedra or close-packed, where the porosity is 26.0% and due to the sphere of equal size, the porosity is independent of the radius of spheres. In our tests, we prepared irregular compact packing of glass beads which due to the equal size of glass beads, the porosity is 26.0%. Eventually, the treated substrates (glasses or oil-wet glasses) were washed by the distilled water and let be dried at ambient conditions in an oven, prepared for contact angle measurement. Figure 1 shows the schematic of the test in this study. The pH and temperature ranges were 2-10 and 25-55°C, respectively.

2.3. Contact Angle and Adsorption Isotherm Measurements

To study the wettability of treated glass and oil-wet glass substrates by the modified nanoparticles, the sessile drop technique was used with 0.1-0.3 μL distilled water droplets on at least five glass beads, and the average of these contact angles was measured by Image J software. A high-resolution video camera (CCD) was used to record all of these processes. For investigating the adsorption of the modified nanoparticle on substrates, the calibration curve at 400 nm was applied by using Hash DR ultraviolet-visible (UV–Vis) of the spectrophotometer. The adsorption isotherms were obtained by using the following equation [26]:

$$q(t) = (C_i - C_x)V \times M^{-1}$$

C_i and C_x are initial and final concentrations of the modified nanofluids (mg/L). V and M are the volumes of solution and mass of substrates that were fixed at 20 mL and 20 g, respectively. Finally, q (t) is the content of modified nanoparticles that is adsorbed on the substrates (mg/g_{glass}).

2.4. Characterizations Tests

The chemical bonding between the surface of silica and polymer was studied by Fourier transform infrared (FT-IR) spectroscopy (Tensor 27, Equinox 55, Bruker). NT–MDT atomic force microscopy (AFM) was used in a non-contact mode to obtain the roughness of prepared substrates. Zeiss scanning electron microscope (SEM) and Oxford energy dispersive spectroscopy (EDS) were applied to study the morphology and composition of substrates before and after treatment by the modified nanoparticles.

3. Results and Discussion

Nanofluids can alter the oil-wet reservoir rock to the water-wet state which enhances oil production [27]. One of the major challenges of this process is the stability of the nanofluids suspension in the harsh conditions of the oil reservoirs which can limit adsorption of the nanoparticles on the reservoir rocks and consequently wettability alteration [10]. Here we prepared the polymer-coated silica nanoparticles and studied systematically the performances of these modified nanoparticles in the harsh condition of the oil reservoirs. In this study, the PEG1, PEG1/C3S, and PEG2/C3S are referred to as the modified silica nanoparticle by polyethylene glycol methyl ether (Mn ~ 2000), the modified silica nanoparticle by polyethylene glycol methyl ether (Mn ~ 2000) and trimethoxy (propyl) silane, and the modified silica nanoparticle by polyethylene glycol methyl ether (Mn ~ 5000) and trimethoxy (propyl) silane.

3.1. Analysis of Non-treated Substrates

In Figure 1, it is seen SEM, EDS, and AFM of the samples. Figures 1 A, B, and C are the glass, the treated glass by piranha solution, and the oil-wet glass (modified by palmitic acid), respectively. Treatment by piranha solution has increased RMS roughness of the glasses from 8 to 20 nm and has decreased the θ of the glasses from 62° to 7°. After modification by the fatty acid, the RMS roughness and θ have been increased to 24 nm and 114°, respectively. By considering Table 1 and Figure 1C, the presence of carbon indicates which fatty acid was adsorbed vastly on the surface of the glass and has a homogeneous adsorption distribution.

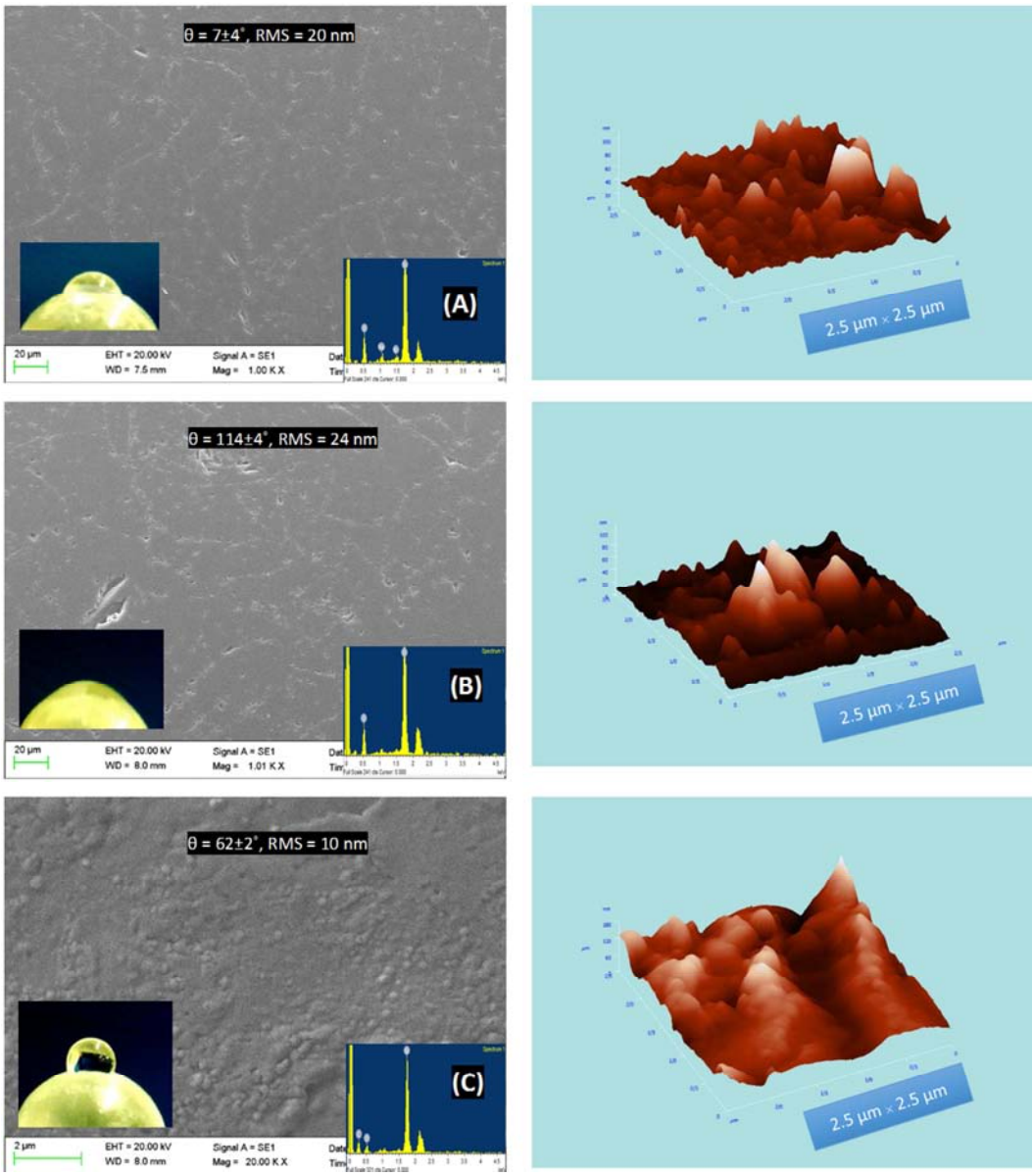

Figure 1. *SEM, EDS, AFM, and contact angle images of substrates: A, B, and C are the surface of the glass, treated glass by piranha solution, and oil-wet glass, respectively.*

Table 1. *Surface composition of glass, treated glass by piranha solution, and oil-wet glass.*

Element (wt%)	Glass	Treated glass by piranha	Oil-wet glass
C	-	-	52.36
O	41.21	45.1	18.66
Si	52.09	54.9	28.98
Br	3.66	-	-
Na	3.04	-	-

3.2. Effect of PH

In our previous investigation [28, 33], the most efficient nanofluid concentrations for the θ reduction of the substrates were 3000 ppm for PEG1 and PEG1/C3S and also 1000 ppm for PEG2/C3S in 2 h exposure time. Therefore, these concentrations were selected to study. In Figures 2 A and B, there were studied wettability alteration and adsorption of the treated glass and the oil-wet glass by the modified nanoparticles as a function of the pH. For all the samples, it is observed an optimal pH for the θ reduction and adsorption. For PEG1 treatment, the optimal pH is around 4 and for the range of pH 6 - 10, θ and adsorption of the glass and oil-wet glass are almost constant. This trend was reported for the adsorption of modified silica nanoparticles by polyethylene glycol onto silicate clays [26]. For PEG1/C3S and PEG2/C3S treatments, this optimal pH is around 6 and the efficiency of these modified nanoparticles for the θ reduction and the adsorption is decreased in the range of pH 6-10. Eventually, in high pH, adsorption and θ reduction of substrates have been decreased due to increasing electrostatic repulsive forces between the nanoparticles and substrates [29-31]. Besides, by changing substrates from water-wet state to oil-wet state, the amount of adsorption remained almost constant for PEG1 while increased for PEG1/C3S and PEG2/C3S. Figures 3A1, B1, and C1 are treated glass by PEG1, PEG1/C3S, and PEG2/C3S in pH 6, respectively. In the treated glass by PEG1 and PEG1/C3S, Figures 3A1 and B1, adsorbed modified nanoparticles have heterogeneous distribution due to agglomeration of the modified nanoparticles in the adsorption process. On another hand, in the treated glass by PEG2/C3S (Figure 3C1), the adsorbed modified nanoparticle has homogeneous distribution due to the highest stability of the modified nanofluid. Figures 3A2, B2, and C2 are the treated oil-wet glass by PEG1, PEG1/C3S, and PEG2/C3S, respectively.

Similar to the treated glass substrates, homogeneous adsorption distribution is observed only in the treated oil-wet glass by PEG2/C3S. By comparing the EDS measurements of the treated oil-wet glasses by modified nanofluids, there is a remarkable increase in the weight percent of silicon and a decrease in the weight percent of carbon for the treated oil-wet glass by PEG2/C3S (Table 2).

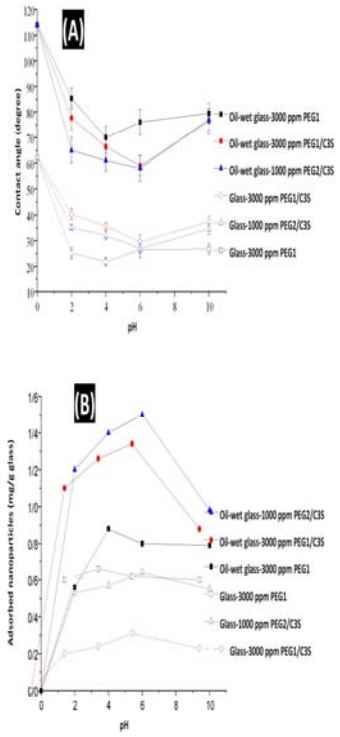

Figure 2. *Effect of pH on the A) water contact angle and B) adsorption of glass and oil-wet glass substrates (2 h exposure time, ~ 25°C, 3000 ppm PEG1 and PEG1/C3S, 1000 ppm PEG2/C3S).*

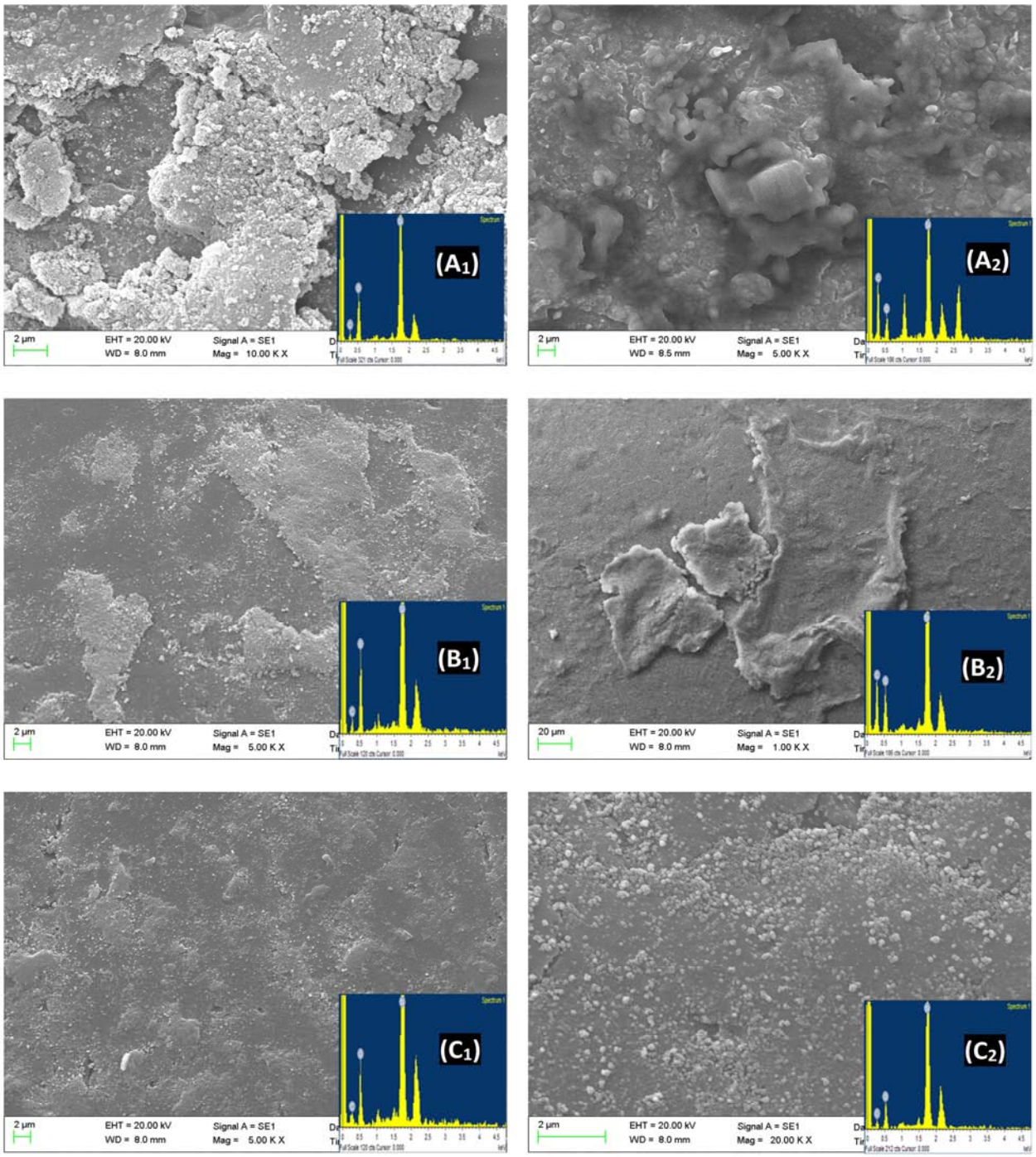

Figure 3. *SEM and EDS images of treated glass and oil-wet glass by modified nanoparticles in pH of 7 (2 h exposure time, ~ 25°C, 3000 ppm PEG1 and PEG1/C3S, 1000 ppm PEG2/C3S).*

Table 2. *EDS measurement of surface composition, glass, and treated oil-wet glass by modified nanoparticles (2 h exposure time, ~ 25°C, 3000 ppm PEG1, and PEG1/C3S, 1000 ppm PEG2/C3S).*

Element (wt%)	Treated glass by			Treated oil-wet glass by		
	PEG1	PEG1/C3S	PEG2/C3S	PEG1	PEG1/C3S	PEG2/C3S
C	24.81	22.68	19.92	58.86	48.85	29.35
O	44.89	42.38	37.55	24.26	30.81	31.98
Si	30.29	34.93	42.52	16.88	20.34	38.67

3.3. Effect of Temperature

Temperature can impact the wettability and adsorption of reservoir rocks [16]. Increasing temperature can decrease the stability of the nanofluid. However, studies have shown which increasing temperature enhances the θ reduction of the various substrates due to decreasing the surface tension and viscosity of the liquid molecules and increasing the Brownian motion of the nanoparticles [32]. The effects of temperature are complicated to study due to involving several variables and cannot be generalized on the oil recovery as this parameter affects both the nanofluids and the reservoir system [32]. Because of these complexities, we studied just altering temperature on the θ reduction and adsorption of the modified nanoparticles in the range of 25-55°C. Figures 4A and B show the effect of the temperature on the wettability alteration and the adsorption of glass and oil-wet glass substrates, respectively. It is notable which the θ reduction and adsorption for PEG1/C3S treatment are improved by increasing temperature. On another hand, the increasing temperature has little impact on the θ reduction and adsorption of PEG1 and PEG2/C3S treatments. For justification of this result, it seems the lowest stability of PEG1/C3S causes this modified nanoparticle to be more sensitive to increasing temperature. This behavior can be related to the negative charge of modified nanoparticles and glass substrates. As by increasing temperature, the stability decreases, and subsequently, this process can reduce the repulsive force between the negative charge of the modified nanofluid and the glass substrates which improves the PEG1/C3S adsorption and θ reduction. For PEG1 and PEG2/C3S treatments, it can be concluded that these modified nanoparticles are less sensitive to altering temperature and consequently, the behavior of adsorption and θ reduction of these modified nanoparticles by increasing temperature is not considerable.

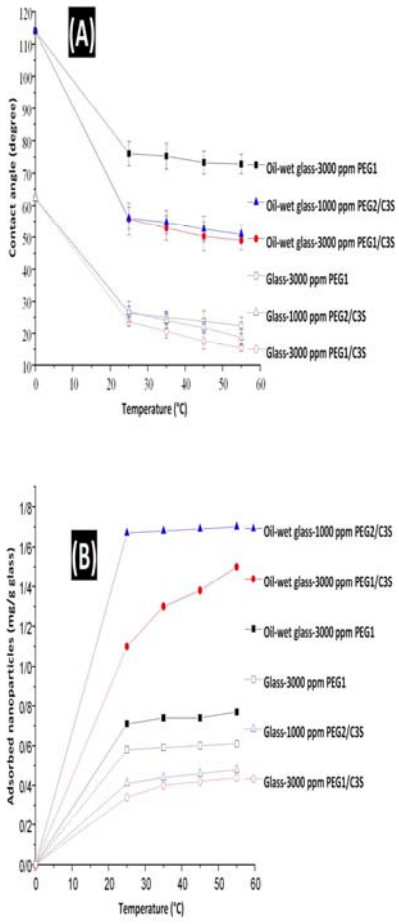

Figure 4. *Effect of temperature on A) the water contact angle and B) adsorption of glass and oil-wet glass substrates (2 h exposure time, 3000 ppm PEG1 and PEG1/C3S, 1000 ppm PEG2/C3S).*

4. Conclusions

This work presents a comprehensive study of the adsorption and wettability alteration of modified nanoparticles by polymers based on effective parameters, including pH and temperature. Our studies showed that the amphiphilic polymer-coated nanoparticles have a better performance in comparison with polymer-coated nanoparticles, especially in the oil-wet system. By comparing three types of polymer-coated nanoparticles, better performance was found for the modified silica nanoparticles by polyethylene glycol (Mn ~ 5000) and propyl chain treatment. The optimal concentration of this modified nanoparticle was 1000 ppm to the θ reduction of glass from 62° to 26.8° and oil-wet glass from 114° to 56.4° in 2 h exposure time. Besides, adsorbed modified nanoparticles were 1.64 mg/g $_{glass}$ for the treatment of the oil-wet glass substrates and 0.44 mg/g $_{glass}$ for the treatment of the glass substrates. The optimal pH was obtained at 6, and in the temperature range of 25-55°C, the sensitivity of this modified nanoparticle wasn't notable. Eventually, we conclude that amphiphilic polymer-coated nanoparticles are an effective agent for wettability alteration of the reservoir rocks.

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