



Original Paper

Impact of temperature and salinity on fines detachment: AFM measurements and XDLVO theory

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ABSTRACT

Fine particle detachment and subsequent migration can lead to severe pore plugging and consequent permeability decline. Therefore, it is crucial to quantify the critical condition when fine particle detachment occurs. The frequently observed deviations or even contradictions between experimental results and theoretical predictions of fines detachment arise from an insufficient understanding of adhesion force that can be highly influenced by salinity and temperature. To clarify the intrinsic influence of salinity and temperature on fines detachment, adhesion forces between carboxyl microspheres and hydrophilic silica substrates in an aqueous medium were measured at various salinities and temperatures using atomic force microscopy (AFM). The AFM-measured adhesion force decreases with increasing salinity or temperature. Trends of mean measured adhesion forces with temperature and salinity were compared with the DLVO and XDLVO theories. DLVO theory captured the trend with temperature via the impact of temperature on electric double layer interactions, whereas XDLVO theory captured the observed trend with salinity via the impact of salinity on the repulsive hydration force. Our results highlight the significance of hydration force in accurately predicting the fate of fines in porous media.

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1. Introduction

The detachment and consequent migration of fine particles with injected fluids (the so called “fines migration”) are widely observed during hydrocarbon and geothermal resource exploitation (AL-Saedi and Flori, 2018), which can lead to severe pore blocking and subsequent permeability decline (Rosenbrand et al., 2015; Yang et al., 2020). However, those plugged fines redirect the flow of the injected fluid towards previously unswept areas, thereby increasing the sweep efficiency of waterflooding in heterogeneous reservoirs (Chavan et al., 2019; Dang et al., 2016; Katende and Sagala, 2019). Besides, once these neutrally wetted or oil-wet clay particles detach, the exposure of more water-wet rock surfaces gives rise to wettability shifting towards a mixed wettability state, beneficial for

enhancing oil recovery (Ahmetgareev et al., 2015; Ding and Rahman, 2017; Park et al., 2018). Therefore, to effectively avoid the adverse effects of fine detachment on reservoir development while utilizing its beneficial effects, accurate prediction and control of fine detachment are crucial prerequisites.

Following contact with the surface, rolling has been recognized as a process preceding fines attachment and detachment in laminar flow (Sharma et al., 1992; Yang et al., 2022). The balance of mobilizing hydrodynamic and arresting adhesive torques is demonstrated as the critical criterion for fines immobilization and release (Bedrikovetsky et al., 2011; VanNess et al., 2019). Accordingly, interaction energies between fines and grain surfaces play a major role in determining the adhesive torque.

The adhesive electrostatic force acting on a fine particle is conventionally described using the DLVO (Derjaguin-Landau-Verwey-Overbeek) theory (Kalantari et al., 2014). Core floods and microscopic visualization tests have typically been designed to quantify the extent of fines migration in porous media under a particular fluid environment, considered an indirect representation of the magnitude of adhesion force (Bradford et al., 2009; Bedrikovetsky et al., 2012). While DLVO theory has the advantage of

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simplicity and provides a qualitative match to observed trends in detachment in many experiments, it fails in providing a universal solution to the interactions between fines and solid surfaces. For example, an easier detachment of attached fines has been repeatedly observed in a high salinity environment, inferring a weaker adhesion between fines and surfaces when the double layer is expected to collapse (Shen et al., 2018; Li et al., 2020). However, the DLVO theory indicates the adhesion force should increase with increased ionic strength due to increased primary minimum depth (Assemi et al., 2006; Zhao et al., 2020). These findings have prompted researchers to acknowledge the significance of non-DLVO forces in explaining adhesion interactions within fluids. An extended DLVO theory (XDLVO), which includes the hydration force, has been developed (Mahmood et al., 2001; Pazmino et al., 2014). Hydration force represents a short-range repulsion between hydrophilic surfaces, which diminishes exponentially with increasing separation distance (Israelachvili and Pashley, 1983; Parsegian and Zemb, 2011). Incorporating hydration force reduces the primary minimum depth, thereby decreasing adhesion, which explains the easier particle detachment in a high salinity environment.

In addition to salinity, temperature also plays an essential role in determining the adhesion force, yet its impact remains controversial. Core flooding experiments indicated that increasing temperature results in decreased core permeability. For instance, Musharova et al. (2012) found that sandstone permeability decreased by 84.3% when the temperature increased from 23 to 150 °C, consistent with the trend observed by Schembre and Kovscek (2005). Similarly, Rosenbrand et al. (2015) observed a decrease in core permeability when the temperature increased from 20 to 80 °C, with permeability largely restored upon cooling. They attributed this to particle detachment at higher temperatures and reattachment to pore walls upon cooling. These studies demonstrate that elevated temperatures facilitate particle detachment and subsequent migration, causing pore blockage and reduced core permeability. However, experiments have also demonstrated that higher temperatures facilitate particle adsorption rather than detachment. Sasidharan et al. (2017) found that increased temperatures led to greater retention of microspheres in porous media, consistent with Yan et al. (2015) and Kim and Walker (2009). In summary, the impact of temperature on particle detachment remains unclear. Moreover, a comprehensive analysis of temperature influence on all DLVO parameters is unavailable in the literature (You et al., 2015, 2019).

Those above-mentioned observations motivate us to measure localized adhesion forces under well-characterized solution conditions. The advancement of atomic force microscopy (AFM) and colloidal probe techniques have provided a quantitative method for characterizing the adhesion forces between particles and surfaces in aqueous environments (Butt et al., 2005; Ducker et al., 1991; Filby et al., 2012). However, the effects of temperature and salinity on adhesion forces remain controversial. Freitas and Sharma (2001) found that the measured adhesion force required to pull off a glass particle from a glass surface grows with increasing electrolyte concentration. On the contrary, the measurements reported by Assemi et al. (2006) and Zhao et al. (2020) demonstrated that the adhesion between carboxyl polystyrene microspheres and silica substrates decreases with increasing salinity. Temperature also plays an essential role in affecting adhesion strength. Lai et al. (2015) reported that the adhesion force between a silica wafer and the flat tip of AFM shows a non-monotonous variation with an increasing temperature in the air while decreases as temperature increases in dry nitrogen. Awada et al. (2011) reported that the adhesion force between an AFM tip and the cross-linked polydimethylsiloxanes decreases at elevated temperatures within a

range where the polymer is in the rubbery state. Shavezipur et al. (2012) observed that in the absence of electrostatic and capillary forces, the adhesion force between polycrystalline silicon surfaces is notably reduced by increasing temperature. In comparison, Cappella and Stark (2006) showed an increase in the adhesion force between the AFM tip and a polymer film with increasing temperature. The influence of temperature on adhesion force is yet to be conclusive. Moreover, we note that the available measurements are all carried out in a gas phase. In addition, the mechanism of the effect of temperature and salinity has not been discussed and analyzed in depth in the above literature.

In the current work, we systematically investigate the influence of salinity and temperature on the adhesion force between carboxyl polystyrene microspheres (representing typical fines) and hydrophilic silica substrates (representing common sandstone rocks) immersed in water. Colloidal probes of the carboxyl polystyrene microspheres are fabricated, and the distributed adhesion forces are directly measured at various electrolyte concentrations and temperatures. The averages of the measured adhesion forces are compared with those calculated from the DLVO theory and the extended DLVO theory accounting for hydration force. Our study highlights the significance of hydration force in affecting particle-surface interactions at high salinity and addresses deviations between experimentally observed fines adhesion and those predicted by theories. In addition, we highlight the mechanism by which environmental conditions (temperature and salinity) affect adhesion force. This enables accurate prediction of fines detachment, which is beneficial for predicting and controlling its impact on reservoir development.

2. Materials and methods

2.1. Colloidal probe

A carboxyl polystyrene microsphere with a diameter of 10 μm (Shanghai Aladdin Co., Inc) was glued to the tipless AFM probe (TL-FM-50, Nanosensors Inc.) under the imaging system of atomic force microscopy (Scan-Icon AFM, Bruker Co., Inc). The latex particles are hydrophilic due to the presence of carboxyl groups (Reed et al., 2012). The hydrophilicity was further confirmed by directly observing the contact angle (12.8°) of a water droplet sitting on a paraffin surface covered with a layer of latex particles. The spring constant of the probe is 2.79 N/m, as determined by the thermal tune method in AFM. We were unable to quantify the temperature effect on the spring constant in water, probably due to its sensitivity to external interferences in an aqueous phase. However, we tested the variation of spring constant at 25, 40, and 50 °C in air. Our measurements showed that the spring constants and corresponding adhesion forces at 40 and 50 °C decreased by approximately 5% and 13%, respectively, relative to that at 25 °C. The deviations fall within the error bands of adhesion force measurements. Accordingly, we neglect the variation of spring constant with temperature, albeit the adhesion force might be slightly overestimated under higher temperatures.

The detailed preparation method is as follows. Firstly, the carboxyl polystyrene microspheres were dispersed in the deionized water, followed by 15-min ultrasonic cleaning. An infinitesimal colloidal solution was dropped on a mica sheet and stands until the liquid was fully vaporized, after which the mica sheet was placed on the sample stage. A target microsphere was chosen via the imaging system, and its position coordinate was recorded. The slow curing epoxy resin (Kite Studio Co., Inc.) was used as the glue. A glue droplet with proper size (5 μm in diameter approximately) was adsorbed onto the probe, avoiding the contamination of excess glue to the particle surface. The probe was then moved to the target

colloid. The colloid was glued to the probe and stands for 24 h, ensuring a firm bonding. The colloid size was measured by the scanning electron microscope (GeminiSEM 300, ZEISS, Germany), as shown in Fig. 1.

The roughness of colloids was characterized by the AFM under tapping mode within a scanning size of $100\text{ nm} \times 100\text{ nm}$. A scanasyst-air probe (Bruker Inc.) with a spring constant 0.4 N/m was used for the roughness measurement. The root mean square (RMS) roughness, defined as the root-mean-square average of the height deviation taken from the mean image data plane, was determined using NanoScope Analysis 1.5.

2.2. Electrolyte solution

NaCl solutions of 0.001, 0.01, and 0.1 mol/L were prepared using NaCl (analytically pure) and deionized water to investigate the effect of salinity on adhesion. The pH value of the NaCl solutions was measured to be 6.1 using a pH meter (Mettler Toledo Five Easy Plus).

2.3. Silica substrates

The hydrophilic SiO_2 substrates were fabricated by the thermal oxidation method after polishing the silicon wafer. SiO_2 substrates were cleaned prior to every experiment. The nominal thickness of the silicon dioxide film was about 300 nm. The size of the purchased silica substrates was $2\text{ cm} \times 2\text{ cm}$. The roughness of the surface was also acquired by tapping mode using AFM.

2.4. Adhesion force measurements

The adhesion force between a microsphere and a silica substrate was measured using AFM. The detailed procedure was described in Filby et al. (2012). The retracting speed, loading force, and loading time were set as constant in this study, as we focus on investigating the influence of salinity and temperature on adhesion force. To be specific, following Xu et al. (2013, 2014), the retracting speed was $1.37\text{ }\mu\text{m/s}$, the loading force was 100 nN, and the loading time was 0 s. The contact-in-liquid mode was selected.

The prepared electrolyte solution was added between the probe and substrate, ensuring that the probe is submerged. The NaCl solutions of 0.001, 0.01, and 0.1 mol/L were sequentially used to measure the adhesion force under various temperatures (25, 40, and $50\text{ }^\circ\text{C}$). Solutions that have been preheated to a designated temperature were replenished continually during measurements to slow down the liquid evaporation caused by heating. Fifty

locations were randomly chosen for testing under a specific experimental condition.

2.5. Zeta potential measurements

The carboxyl polystyrene microspheres were dispersed in the prepared NaCl solutions of 0.001, 0.01, and 0.1 mol/L. Each solution contains 0.01 wt% of microspheres. Zetasizer NanoZS (ZS-3600, Malvern Instruments Ltd., Worcestershire, U.K.) was used to measure the zeta potential of particles under different salinity. The obtained zeta potentials could be employed to calculate the double layer force between the carboxyl polystyrene microsphere and silica surface.

3. Theory

3.1. DLVO

DLVO theory has been widely used to quantitatively evaluate the adhesion force required for particle detachment. According to the DLVO theory, the total interaction potential energy V_{DLVO} between a microsphere and silica substrate includes the attractive van der Waals potential energy V_{LW} , the electrostatic double-layer potential energy V_{EDL} , and the repulsive Born potential energy V_{B} :

$$V_{\text{DLVO}} = V_{\text{LW}} + V_{\text{EDL}} + V_{\text{B}} \quad (1)$$

The adhesion force (interaction force) between a microsphere and silica is derived from the interaction potential energy with respect to distance:

$$F = -\frac{\partial V}{\partial h} \quad (2)$$

Detailed calculation formulas (Eqs. (S1)–(S5)) and parameters (Tables S2–S4) are provided in the supporting information.

3.2. XDLVO

Non-DLVO forces have also been acknowledged as significant in the interactions between fines and surfaces (Li et al., 2020; Mahmood et al., 2001; Pazmino et al., 2014). Hydrophobic surfaces generally demonstrate attractive hydration force, leading to an increased adhesion forces (Israelachvili, 2011). However, a short-range repulsive hydration force arises between hydrophilic surfaces when the salinity of the solution reaches a certain level (Molina-Bolivar and Ortega-Vinuesa, 1999). The hydration force, arising from the hydrated cation adsorbed on the negatively-charged surface, is introduced in the extended DLVO theory besides the typically considered van der Waals force, double-layer force, and Born repulsion force in the conventional DLVO theory formulated in Section 3.1. According to the XDLVO theory, the total interaction potential is given by the following formula:

$$V_{\text{XDLVO}} = V_{\text{LW}} + V_{\text{EDL}} + V_{\text{B}} + V_{\text{H}} \quad (3)$$

Detailed calculation formulas (Eqs. (S6)–(S8)) and parameters for calculating the hydration interaction are provided in the supporting information.

4. Results

Our experimental data (Fig. 2) show that the adhesion force decreased monotonically with increased salinity and temperature. It is worth noting that the AFM-measured adhesion force exhibits a distinguished decline at 0.1 mol/L. For instance, at $25\text{ }^\circ\text{C}$, the adhesion force decreases from 36.5 to 25.4 nN when the salinity

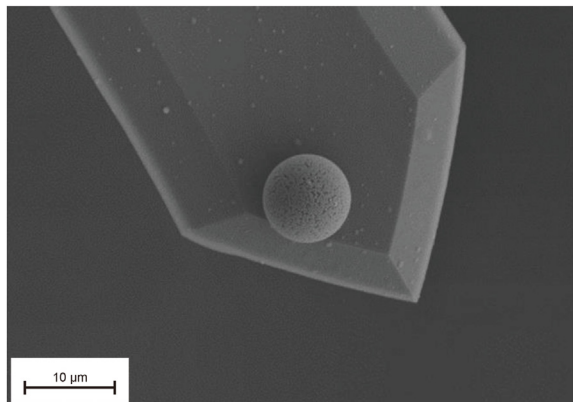


Fig. 1. SEM image of preparation of colloidal probe.

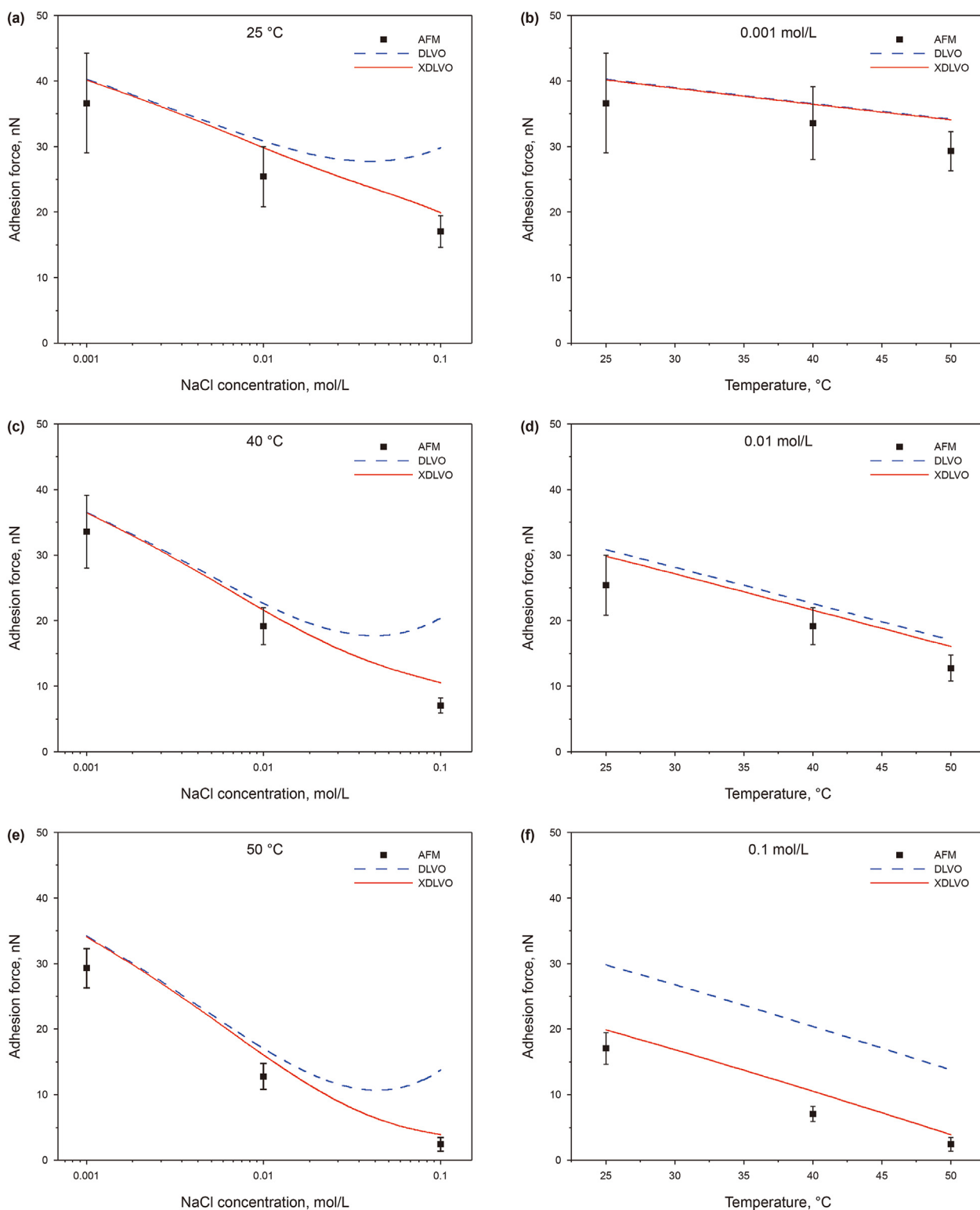


Fig. 2. Adhesion force between latex microspheres and silica substrate as a function of salinity (NaCl concentration) and temperature, with measurements (symbols) and calculated (lines) grouped according to variations in NaCl concentration (a, c, e) and temperature (b, d, f). Error bars reflect the standard deviation from average among 50 measurements.

increases from 0.001 to 0.01 mol/L and further declines to 17.1 nN when the salinity rises from 0.01 to 0.1 mol/L. Those weak attraction forces are in accordance with the measured data between two hydrophilic surfaces, reported by Freitas and Sharma (2001).

In contrast, DLVO theory predicted a non-monotonic

relationship between adhesion force and salinity, with a minimum adhesion force at approximately 0.04 mol/L salinity (Fig. 2(a), (c), and (e)). Given the decreased repulsion associated with increased zeta potentials, the overall decreased adhesion force with increased salinity reflects the predominance of hydration force at high

salinity. The possibility that extended DLVO (XDLVO) interactions explain the experimental (monotonic) trend will be explored below. The agreement between experimental and DLVO-predicted trends with temperature (Fig. 2(b), (d), and (f)) arises from the impact of temperature on electric double layer (EDL) as well as London-van-der-Waals (LVAW) interactions, as described below.

4.1. Salinity effect

At a certain temperature, the adhesion force decreases as the salinity increases, showing a similar trend as reported in Assemi et al. (2006) and Zhao et al. (2020). However, this tendency deviates from DLVO prediction, which suggests comparable values at 0.01 and 0.001 mol/L. Commonly, the electrical double layer force is much more sensitive to salinity relative to the van der Waals force and Born repulsion force. This is because zeta potential, as one of the essential parameters in the expression of double-layer force, is highly responsive to changes in salinity. The zeta potentials of both latex particles (measured) and silica (adopted from Tufenkji and Elimelech, 2004) in solutions with different NaCl concentrations are shown in Fig. 3. As the salinity increases, the absolute value of zeta potential descends, indicating a weaker repulsive double-layer force. Therefore, in light of the traditional DLVO theory, one expects that increasing salinity results in a higher adhesion force and retained particle concentration, and on the contrary, decreasing salinity yields a lower adhesion force and retained particle concentration. Yet, this is not always true (Zhao et al., 2020).

Theoretically, the potential energy of a particle is locally minimized. That is, fine particles attached to a surface are trapped at a local minimum of the energy profile. An energy barrier might exist at low salinity, separating the primary minimum and the secondary minimum at the energy–distance profile, while at high salinity, the energy barrier is absent. Indeed, when measuring the adhesion force using AFM, the external force imposed by the AFM cantilever can provide enough energy to force particles through the energy barrier and reach the neighborhood of the primary minimum. Accordingly, the AFM-measured force represents the adhesion force for a particle trapped around the primary minimum to be detached.

Fig. 4 demonstrates the calculated examples of the DLVO interaction profile under different salinity. At a distance greater than the primary minimum, the calculated double-layer force decreases as the salinity increases, agreeing with our knowledge.

Nevertheless, the double-layer force curves intersect at around the primary minimum, exhibiting a non-monotonic variation, i.e., increases first and then decreases with increasing salinity. Given that the Hamaker constant is negligibly changed with salinity (Gregory, 1981), we conclude that the van der Waals force and Born repulsion force are less dependent on salinity. Therefore, the absolute value of the total DLVO force at the primary minimum is anticipated to decrease first and then increase with an increasing salinity, opposite to the variation trend of double-layer force at the same location.

The deviation between DLVO theory and experimental data was caused by the non-monotonic trend of the primary minimum force. This deviation between the classical DLVO theory and the experimental data might be ascribed to the ignorance of the hydration force that is proportional to salinity and repulsive between hydrophilic surfaces at short separations. Fig. 4(a) (dashed lines) presents the calculated force profiles under different salinity using XDLVO theory that accounts for the hydration force. It is shown that the primary minimum force obtained from the XDLVO theory exhibits a pronounced decreasing tendency with increasing salinity and shows a remarkable decline when the salinity increases from 0.01 to 0.1 mol/L, in line with the variation of the AFM-measured adhesion force. Furthermore, as demonstrated in Fig. 2, the forces obtained by DLVO and XDLVO theory are comparable at 0.001 and 0.01 mol/L, while showing a great deviation at 0.1 mol/L, suggesting that the hydration force becomes dominant at a salinity close to 0.1 mol/L and beyond. Accordingly, we conclude that the primary minimum force obtained from the XDLVO theory can better explain the experimental results of the adhesion force, especially at high salinity.

4.2. Temperature effect

It is demonstrated in Fig. 2 that the adhesion force decreases with increasing temperature at a given salinity. Pashley (1981) reported that the hydration force in an electrolyte solution containing K^+ or Na^+ maintains changeless when the temperature increases from 21 to 65 °C. As a result, DLVO interactions dominate as the temperature varies, and the temperature influence on adhesion force is anticipated to be well described by the DLVO theory. However, a comprehensive summary of the temperature influence on major parameters of interactions is rarely reported. A circumstantial analysis on the temperature dependencies of all DLVO parameters for the latex-water-silica system is given in Table S1 in the

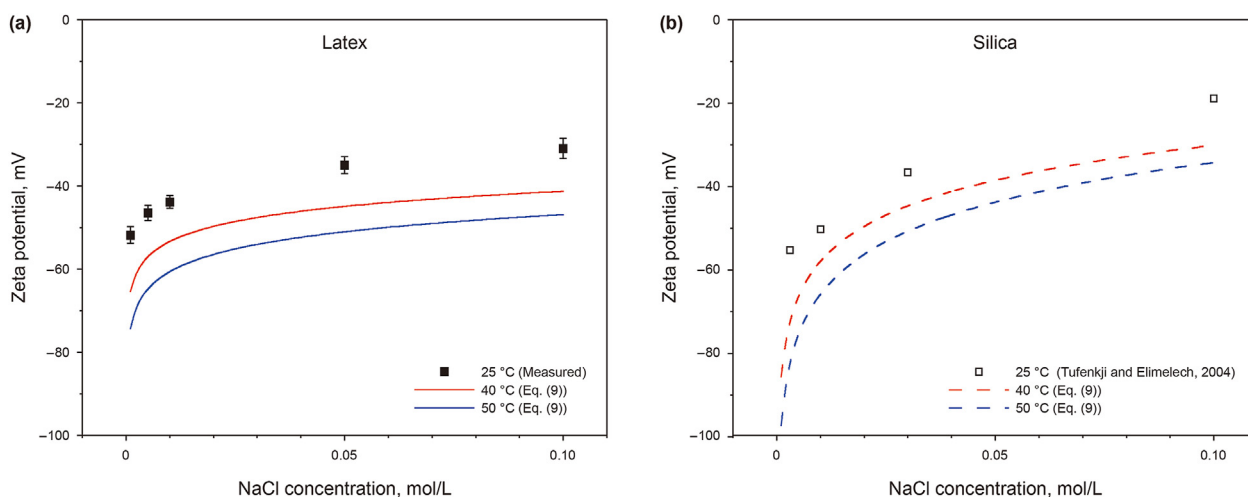


Fig. 3. Zeta potentials as a function of NaCl concentration: (a) Latex; (b) Silica. The squares are experimental data measured at 25 °C. The solid and dashed lines are calculated using Eq. (9) derived by Schembre and Kovscek (2005) at 40 and 50 °C.

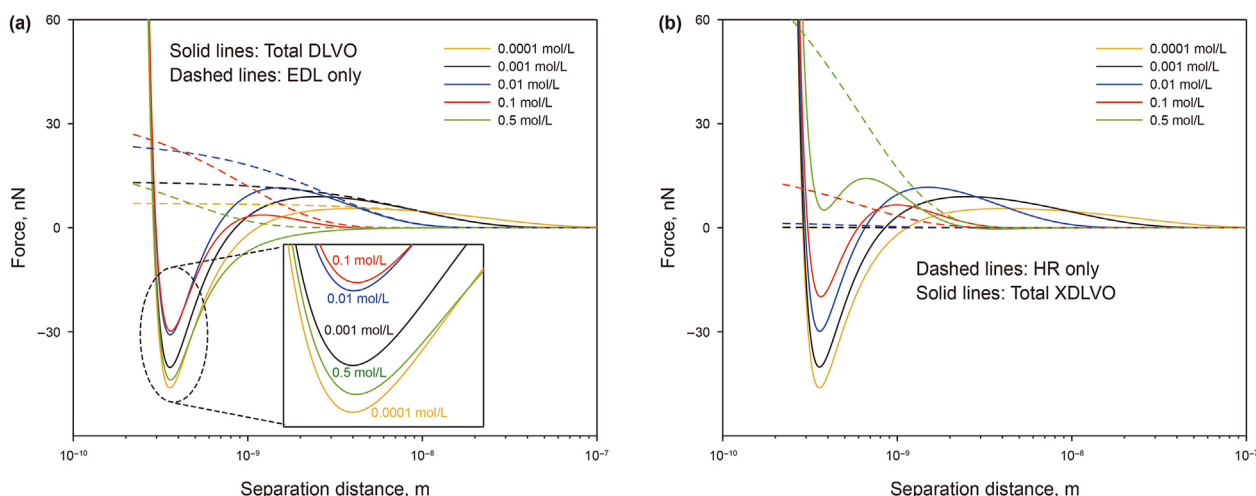


Fig. 4. Effects of salinity on DLVO (a) and XDLVO (b) forces between latex microspheres and silica substrates at 25 °C.

supporting information.

Fig. 5 presents the DLVO-calculated force at elevated temperatures. The shallower primary minimum with increased temperature is mainly attributed to increased double-layer repulsion with increased temperature (Fig. 5(a)), as the van der Waals force and the Born repulsion force vary negligibly with temperature (Fig. 5(b)). The stronger double-layer repulsion with increased temperature arises from a larger magnitude of the negative zeta potential for both latex and silica (Fig. 3). It is noteworthy that temperatures higher than 50 °C may have a more significant influence on the Hamaker constant and thus the van der Waals force and the Born repulsion force.

As expected, both the DLVO- and XDLVO-based and the AFM-measured adhesion forces decline with an increasing temperature. The adhesion force shows a slight decrease with the temperature at a lower salinity. In comparison, a more evident downtrend is observed at higher salinity, which might be attributed to the dominance of the temperature influence on the double layer force within the range between 25 and 50 °C. In addition to its effect on the surface charge, an increasing temperature enables expanding the buffer layer of ions (Alizadeh and Wang, 2020), yielding a lower zeta potential. The lower is the salinity, the broader is the buffer

layer, and the less sensitive is the buffer-layer thickness to temperature. At a high salinity, the buffer layer is easier to be expanded as the temperature increases, resulting in a more significant reduction in adhesion force.

In conclusion, DLVO theory captured the trend with temperature via the impact of temperature on electric double layer interactions. The XDLVO theory (DLVO + hydration interaction) captured not only the effect of temperature through DLVO, but also captured the observed trend with salinity via the impact of salinity on the repulsive hydration force. The XDLVO theory provided a good explanation of the impact of temperature and salinity on the adhesion between latex and hydrophilic silica, as shown in Fig. 2.

5. Discussion

5.1. Impact of surface roughness

We noticed theoretical predictions, grounded in either DLVO or XDLVO theory, are slightly higher when compared to the AFM-measured adhesion force. As suggested by Assemi et al. (2006), surface roughness could be one of the potential reasons contributing to this deviation. In contact mechanics studies, the presence

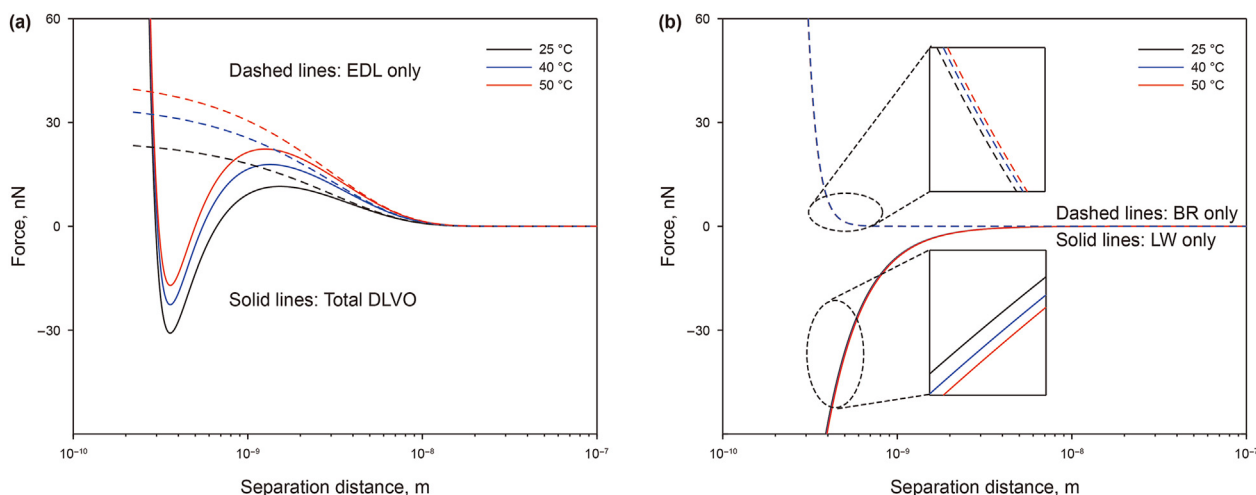


Fig. 5. Temperature effects on DLVO and double layer force (a) and on van der Waals force (LW) and Born repulsion (BR) (b) between latex microspheres and silica substrates at 0.1 mol/L.

of surface roughness reduces the adhesion force via a reduced contact area or radius (Assemi et al., 2006; Ziskind et al., 1997). Additionally, surface roughness is also an important parameter that can alter van der Waals and electric double layer interactions (Elimelech and O'Melia, 1990; Suresh and Walz, 1996). Chequer et al. (2021) considered the total electrostatic force of particles on a rough surface to be the derivative of the sum of the particle–solid surface (V_p-S) and particle–asperity interaction energies (V_p-a).

The silica substrate under study is of an average asperity height of 0.27 nm with an RMS roughness of 0.34 nm over the $1.5 \mu\text{m} \times 1.5 \mu\text{m}$ scan range (Fig. 6), inferring that the silica surface is rather smooth, given that an RMS roughness varying from 0.1 to 0.3 nm is considered as molecularly smooth (Kokkoli and Zukoski, 1998; Fuller and Tabor, 1975). Accordingly, the roughness of the silica can be considered negligible. In contrast, the average asperity height of the latex microsphere is 1.20 nm, with an RMS roughness of 1.51 nm over a $100 \text{ nm} \times 100 \text{ nm}$ scan size (Fig. 6(b)), which is significant enough to reduce adhesion force (Rasmuson et al., 2019; Das et al., 1994). However, it is important to note that roughness is unaffected by temperature and salinity and does not alter the trends of mean measured adhesion under varying conditions. Therefore, the effect of roughness is not considered further in this paper.

5.2. Determination of adhesion force and implication on maximum retention function

Particle detachment has been hypothesized to occur when the drag torque surpasses the adhesive torque (Bedrikovetsky et al., 2011, 2012). The adhesive force acting on a fine particle is conventionally described using the DLVO theory (Yang et al., 2022). The DLVO theory is closely related to conditions such as salinity and temperature, resulting in the establishment of a model of particle detachment—maximum attached particle concentration as a function of salinity and temperature. This model is applicable not only to detrital fines detached against electrostatic forces (Hashemi et al., 2023), but also to authigenic fines detachment (Borazjani et al., 2024). The current approach to determining the adhesive force relies on the location where particles are trapped. If particles were initially immersed in a high salinity environment and trapped at the primary minimum, then this minimum should be used to quantify particle adhesion to surface when the salinity decreases, provided the trapped particles cannot overcome the energy barrier from the left side. Conversely, if particles were initially immersed in a low-salinity fluid, they are expected to be trapped at the secondary minimum unless they can transition to the primary minimum from the right side of the energy barrier. In this scenario, the secondary minimum should be utilized (Yang et al., 2022).

However, the weak secondary force indicates no immobile attachment occurs under unfavorable conditions due to fluid shear and particle diffusion. This contradicts visualization tests showing that attached particles are incompletely removed in low salinity (Ryan and Gschwend, 1994; Lenhart; Sarier, 2003; Torkzaban et al., 2010). This suggests that partial particles may also be captured at the primary minimum. This discrepancy may arise from charge heterogeneity and the extent of overlap between the zone of influence (ZOI) and heterodomains (Ron et al., 2019; Rasmuson et al., 2019). Nonetheless, the DLVO theory assumes no physical or chemical heterogeneity. Moreover, we also found that the force corresponding to the primary minimum of the DLVO profile exhibits a counterintuitive, non-monotonous variation with decreasing salinity at the low-salinity range that can result in a non-monotonous maximum retention concentration (expected to be monotonous).

Another observation that contradicts DLVO predictions is the decreased adhesion with increased salinity at high electrolyte concentrations. Specifically, at a salinity of 0.1 mol/L, the DLVO theory predicted adhesion force (primary minimum) significantly overestimates the experimental measurements, and the force corresponding to the primary minimum of the DLVO profile exhibits a non-monotonic change, decreasing and then increasing with decreasing salinity, which could result in a non-monotonic maximum retention concentration (expected to be monotonic). Furthermore, easier detachment of attached fines has been repeatedly observed in high salinity environments, inferring a weaker adhesion between fines and surfaces when the double layer is expected to collapse (Shen et al., 2018; Li et al., 2020). However, the DLVO theory suggests that the adhesion force should increase with increased ionic strength due to the increased depth of the primary minimum (Assemi et al., 2006; Zhao et al., 2020). These findings have prompted researchers to acknowledge the significance of non-DLVO forces in explaining adhesion interactions within fluids. Our measurements inferred the significance of the repulsive hydration forces between hydrophilic surfaces in environments with elevated salinity. Nonetheless, the observed decrease in adhesion force with increasing salinity may result in a reduced maximum retention function, contrasting with previously published experimental results (Bedrikovetsky et al., 2011, 2012; Chequer et al., 2021; Hashemi et al., 2023; Borazjani et al., 2024).

Incorporating hydration force reduces the primary minimum depth, thereby decreasing adhesion and explaining the monotonically decreasing adhesion force with increased salinity. Nevertheless, while XDLVO theory replicates the trend of varying mean adhesion forces with salinity, it is a mean-field theory that does not account for physical and chemical surface heterogeneity. Thus, it cannot explain variances around the mean. Future work could

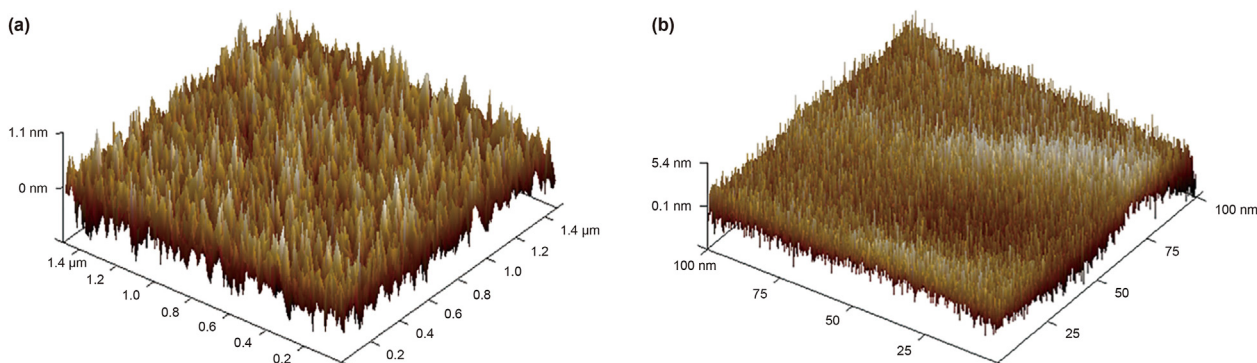


Fig. 6. AFM images of the scanned surface roughness: (a) Silica; (b) Latex.

explore the correlation between heterogeneity and the variance in measured adhesion forces. Additionally, when measuring adhesion forces using AFM, the external force exerted by the cantilever pushes particles through the energy barrier to the vicinity of the primary minimum. Thus, AFM-measured forces represent adhesion of particles trapped around the primary minimum rather than under unfavorable conditions. Accurately measuring the forces at which particles are captured at the secondary minimum remains challenging with current experimental equipment. Future work could lead to the determination of adhesion force at low salinity when an energy barrier prevents colloids from approaching the primary minimum.

The Johnson–Kendall–Roberts (JKR) theory has been applied to the calculation of the maximum adhesion force between elastic solids (Johnson et al., 1971; Prokopovich and Perni, 2011). According to the JKR model, interfacial parameters are required to calculate the thermodynamic work of adhesion. However, the surface tension of a solid is hard to be obtained (Starov, 2013), let alone its salinity and temperature dependence that have been proved to be crucial in determining the adhesion force. The contact angle is generally used to estimate the Lifshitz van der Waals component of the surface tension (Starov, 2013; Vrlinic et al., 2016). Yet, the difficulty in obtaining an equilibrium contact angle results in more uncertainty. Therefore, the comparison between experimental results and JKR theory is not performed in the present paper.

5.3. Representativeness of carboxyl polystyrene particles to fines migration

It is reasonable to question whether the results obtained from spherical polystyrene latex microspheres are applicable to the transport of natural colloids such as silica, kaolinite, illite, and chlorite. Firstly, due to the presence of carboxyl groups, the polystyrene microsphere surface is negatively charged and hydrophilic, consistent with the negatively charged fines. While these fine types differ fundamentally in shape and surface structure (Grasso et al., 1996; Harvey et al., 1995; Kim and Walker, 2009), they all exhibit breakthrough curves and retention profiles similar to those of polystyrene latex microspheres (Lenhart and Saiers, 2003; Wang et al., 2012; Won et al., 2021; You et al., 2019). Therefore, we claim that polystyrene latex microspheres are representative of studying fines migration underground.

6. Conclusions

The influences of salinity and temperature on the adhesion force between carboxyl polystyrene microspheres (representing typical fines) and hydrophilic silica substrates (representing common sandstone rocks) in water were systematically examined using AFM. The measurements were compared with the DLVO and XDLVO theories, which achieved accurate prediction of fines detachment. The main conclusions from our study are as follows.

The AFM-measured adhesion force decreased with increasing salinity or temperature. DLVO theory captured the trend with temperature via the impact of temperature on electric double layer interactions. However, DLVO theory predicted a non-monotonic relationship between adhesion force and salinity, with a minimum adhesion force at salinity of approximately 0.04 mol/L. This theoretical prediction deviated from the experimental trend.

In contrast, the decreasing tendency of the mean values could be matched by the XDLVO theory that incorporates the hydration force, accurately predicted the adhesion forces under varying salinity conditions. The shift from non-monotonic to monotonic trends between DLVO and XDLVO predictions resulted from decreased primary minimum due to increased hydration force with

increased salinity.

CRedit authorship contribution statement

Wei-Feng Yuan: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Yu-Long Yang:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Lu Yuan:** Visualization, Resources, Investigation, Data curation. **Ji-Rui Hou:** Writing – review & editing, Validation, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Yu-Long Yang is an editorial board member for Petroleum Science and was not involved in the editorial review or the decision to publish this article.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.petsci.2024.09.017>.

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