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Original article

The effect of methylene blue and organic acids on the wettability of sandstone formation: Implications for enhanced oil recovery

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Abstract:
Fossil fuels are the primary global energy source, and their improved production will ensure a balance between the increasing energy demand and supply. Chemical-enhanced oil recovery has been well thought of as a promising method for increasing hydrocarbon production. However, the effectiveness of this method depends on wettability of rock-oil-brine systems' Previous studies have shown that oil-wet rock demonstrated a water-wet state when treated with surface active chemicals like surfactants, nanofluids. Moreover, increasing attention has become focused on the application of hazardous pollutants such as methyl orange and methylene blue to enhance the CO_2/H_2 containment security of the host rock by altering its wettability. Nevertheless, the capacity of methylene blue to modify the rock wettability for the production of trapped hydrocarbons in sandstone reservoirs is yet to be explored. Thus, in the present study, methylene blue is used as a wettability modifier to enhance the oil production from quartz rocks that have been aged with stearic acid solution (10-2 mol/L). First, the organic-aged quartz is treated with various concentrations of methylene blue (10-100 mg/L) for one week at 60 °C. Then, contact angle measurements are performed at different temperatures (25 and 50 °C) under various pressures (10-20 MPa) and brine salinities (0-0.3 M). Thus, the quartz is found to turn hydrophobic when aged in organic acid/n-decane solution at 20 MPa and 50 °C. However, when the rock is treated with various concentrations of methylene blue, the hydrophobicity is found to decrease, thus suggesting that oil recovery will be promoted by methylene blue treatment. Overall, the results demonstrate that the most favourable condition for reducing the hydrophobicity of the sandstone rock is via treatment with 100 mg/L methylene blue. Hence, the injection of methylene blue into deep underground sandstone reservoirs has the potential to produce more residual hydrocarbons.

1. Introduction

Due to rapid population growth and the increasing advancement of global technology, more energy resources will be needed to ensure an equilibrium between global energy demand and supply (Keshavarz et al., 2018; Mahesar et al., 2020; Aslannezhad et al., 2023). In this respect, fossil fuel is considered as the most efficient energy source worldwide (Schiffer et al., 2018; Pu et al., 2020). However, the amounts of oil recovered from primary and secondary sources are not sufficient to tackle the energy needs of the world (Muggeridge et al., 2014). Therefore, enhanced oil recovery (EOR) methods such as chemical EOR, thermal EOR, and gas injection have

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been adopted to augment oil recovery (Mansour et al., 2019; Haghighi et al., 2020; Mohanty et al., 2021; Nazarahari et al., 2021). The main EOR mechanism involves alteration of oil-wet wettability towards water-wet conditions, thus, wettability is specified as a fundamental property for optimising oil recovery (Ding and Gao, 2021).

There are different techniques of tertiary EOR, such as thermal, gas, and chemical injection (Pwaga et al., 2010; Kim and Kovscek, 2017; Gbadamosi et al., 2019; Askarova et al., 2020). Chemical EOR involves injection of various chemicals such as surfactants, alkalis, polymers, nanofluids, and combination of these chemicals to improve the efficiency of mature oilfields (Adil et al., 2018; Pal et al., 2022, 2023a, 2023b; Pothula et al., 2023). Findings from previous research have demonstrated significant alteration of rock wettability towards hydrophilic state in presence of these chemicals (Okunade et al., 2021; Alhammad et al., 2022, 2023a; AlZaabi et al., 2023; Kashefi et al., 2023; P et al., 2023; Zhang et al., 2023).

Methylene blue (MB) is a cationic dye that is commonly applied in textiles, food, cosmetics, wool, rubber, and pharmaceuticals industries (Kwok and Howes, 2006; Shah et al., 2006; Fito et al., 2020; Kayabasi et al., 2020). A large amount of this chemical is disposed into the environment, becoming a significant pollutant (Khan et al., 2022). Several methods have been used previously to remove methylene blue (MB) from water, such as precipitation, coagulation, adsorption, photocatalytic degradation, and ion exchange (Hosseinzadeh et al., 2017; Fadillah et al., 2019; Atchudan et al., 2020; Sivakumar and Lee, 2022). Although a considerable portion of previous research has been concentrated on the removal of MB contaminants from wastewater (Kuan et al., 2011; Gong et al., 2013; Pathania et al., 2017; Tang et al., 2017; Hidayat et al., 2022), however, no previous research has been carried out to assess the impacts of methylene blue on the wettability modification of quartz surfaces and its attendant effect on oil recovery from sandstone formations.

Consequently, the main idea of this study is to assess the possibility of disposing methylene blue into the underground reservoir to increase the oil recovery of such rocks by modifying the rock-wetting characteristics. Herein, we used methylene blue (at different concentrations from 10-100 mg/L) as a chemical modifier to change the hydrophobic quartz surface (aged in stearic acid (SA)) to a hydrophilic state. The wettability was evaluated from advancing (θ_a) and receding (θ_r) contact angle (CA) values measured at downhole pressures (10-20 MPa), temperatures (25 and 50 °C), and salinity (0 to 0.3 M). The study highlights the conditions for achieving a significant reduction in rock hydrophobicity through the injection of MB into the underground reservoirs for recovering trapped hydrocarbons.

2. Materials and methods

2.1 Materials

Pure quartz substrates were bought from Ward's Natural Science (Home | Ward's Science (wardsci.com)). Stearic acid and n-decane ($\geq$ 99 mol%, pure) were purchased from Sigma-Aldrich, Australia. Nitrogen (BOC, gas code-234, purity $\geq$ 99.999 mol%) and deionized (DI) water was utilized to clean the organic-aged and non-aged quartz substrates. Various brine solutions (0.1 to 0.3 M) were prepared by mixing NaCl (Sigma-Aldrich, purity $\geq$ 99.5 mol%) and DI water. Methylene blue (from Sigma-Aldrich; Fig. 1) was prepared at diverse concentrations (10-100 mg/L) to modify the stearic-acid-aged quartz substrate's wettability.

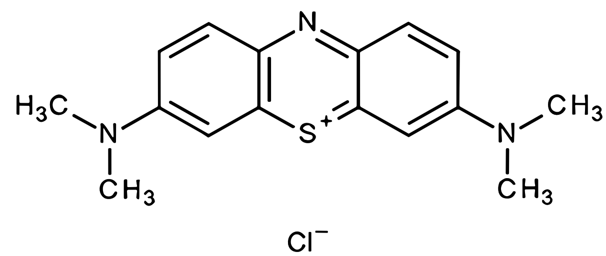


Fig. 1. Chemical structure of methylene blue (Farch et al., 2023).

2.2 Cleaning and aging procedure of quartz substrate

It is essential to clean the quartz substrates to ensure the contact angle measurements will produce a reliable result (Bikkina, 2011; Iglauer et al., 2014). Firstly, the quartz rocks were cleansed with DI water and dried with ultra-pure N_2. After that, the substrates were left in the oven for 1 hour at 60 °C to remove any residual organic impurities. Once the quartz substrates were dried enough, it was placed for 30 mins in a 2 wt% NaCl brine and drops of HCl acid were added to make the pH = 4. This process is necessary to ensure that the stearic acid is effectively adsorbed on the quartz samples (Ali et al., 2019). After that, the NaCl/HCl-treated quartz samples were dried with ultra-pure N_2 to remove the left-over brine from the surface. Then we placed the substrates in a 10-2 mol/L of stearic acid/n-decane solution for a week; sample containers were tightly sealed and kept in an oven to alter the quartz surface wettability into hydrophobic condition. Subsequently, the organic-acid contaminated quartz substrates were aged with different concentrations of methylene blue (10-100 mg/L) and were placed in an oven for a week.

2.3 Contact angle measurement procedure

The CA measurement setup is presented in Fig. 2. The θ_a and θ_r were determined using the tilted plate goniometric approach at tilted angle of 17°. The process was explained in previous studies (Saraji et al., 2013; Li et al., 2018b). The substrate was positioned in an optical cell, and two high-precision ISCO syringe pumps, a Teledyne ISCO D-260 product with a pressure accuracy of 0.01%, were used for injecting decane or brine at different pressure and temperature conditions. N-decane was the surrounding fluid whereas a needle was used for dispensing brine droplet onto the quartz surface placed on a tilted plate. A professional video camera (Basler ace acA640-90 um monochrome USE 3.0 camera fitted with a Ricoh TV lens 50 mm 1:1.4; frame rate 30

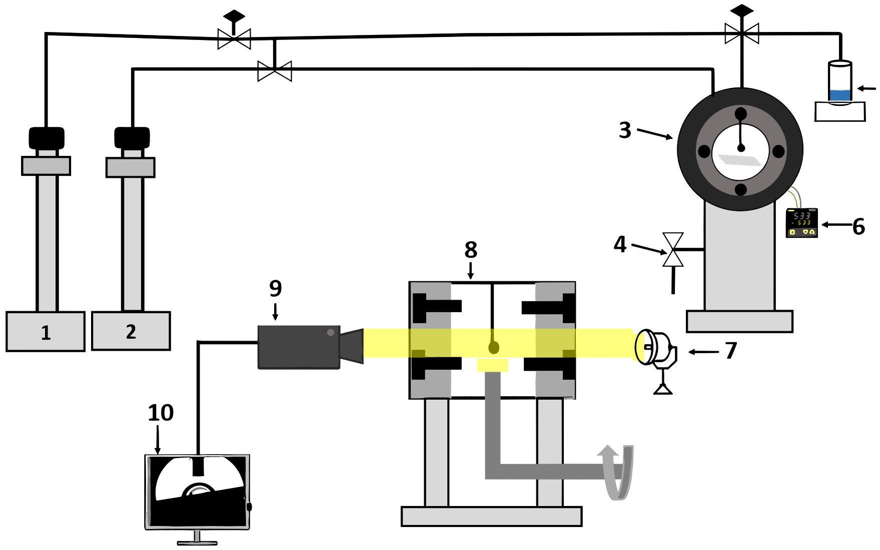
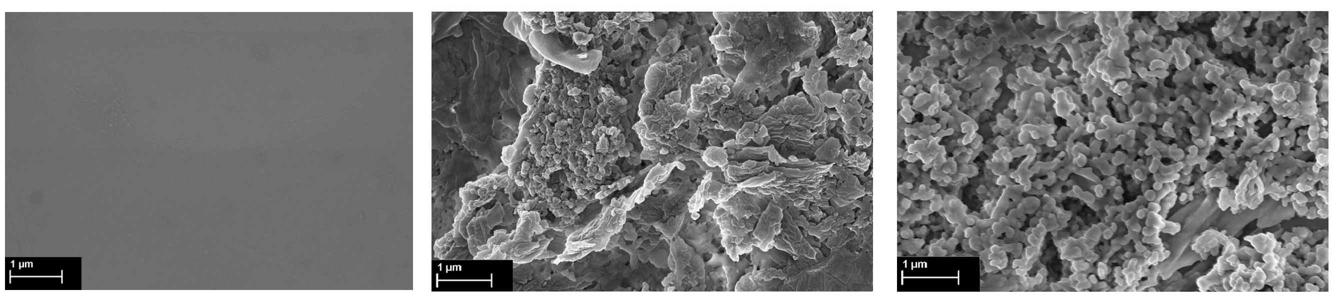


Fig. 2. Setup of the CA measurement (1) syringe pump controlling the flow of brine, (2) syringe pump controlling the flow of n-decane, (3) front view of high-pressure high-temperature (HPHT) cell containing a titled plate, (4) pressure relief valve, (5) refill/drain system, (6) temperature controller connected to an electrical heater, (7) light source, (8) side view of HPHT cell with titled plate, (9) high-speed camera and (10) Computer for contact angle analysis (Alhammad et al., 2022).



(a) (b) (c)

Fig. 3. FESEM images showing the morphology of (a) clean quartz, (b) stearic-acid-aged quartz and (c) MB (100 mg/L) modified quartz at a magnification of 1 μm.

fps; resolution 656×480 pixels) was utilized to capture the entire process. Finally, θ_a and θ_r were extracted with ImageJ software from the recorded video.

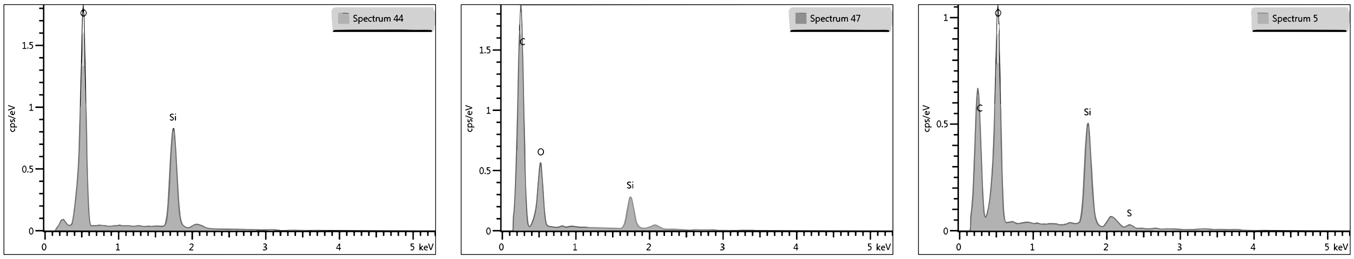
3. Results and discussion

The success of hydrocarbon recovery from conventional and unconventional reservoirs depends on the affinity of the reservoir rock for the oil/water system (Agbalaka et al., 2008; Murray and Narayanan, 2019; Ahmadi et al., 2020; Alhammad et al., 2022; Rego et al., 2022; Yekeen et al., 2023). The foremost reason for treating the quartz surface with MB is to alter the wettability from hydrophobic to hydrophilic conditions and improve hydrocarbon recovery from the matured reservoirs (Zhang et al., 2007; Kamal et al., 2017; Li et al., 2018a; Omran et al., 2020; Tackie-Otoo et al., 2022; Alhammad et al., 2023b). The change in and after placing the stearic acid-aged quartz in different MB concentrations was used for appraising the rock wetting state at various downhole conditions. The contact angle datasets are presented and discussed in this section.

3.1 Quartz sample characterization

The Field emission scanning electron microscopy (FE-SEM) images of the pure quartz and the stearic-acid aged quartz before and after modification with MB are presented in Fig. 3. Here, the surface of the pure quartz is seen to be smooth and flat (Fig. 3(a)), whereas the stearic-acid aged quartz exhibits a rough surface (Fig. 3(b)). This is consistent with previous result (Alhammad et al., 2022, 2023b). However, after modification with MB, a rod-shaped morphology is observed (Fig. 3(c)). This is because the positively charged MB is attracted to the negatively charged quartz surface to form a monolayer of adsorbed molecules.

In addition, the energy-dispersive X-ray spectroscopy (EDS) analyses of the various samples are presented in Fig. 4. Here, the main components of the pure quartz are seen to be silicon (Si) and oxygen (O) (Fig. 4(a)), whereas the stearic-acid aged quartz contains a significant amount of carbon but less silicon and oxygen (Fig. 4(b)), thus suggesting that the quartz surface has turned hydrophobic. By contrast, the amo-



(a) (b) (c)

Fig. 4. EDS analysis of (a) clean quartz, (b) stearic-acid-aged quartz, and (c) quartz aged with methylene blue at 100 mg/L. The Si represents silicon, O represents oxygen, C represents carbon and S represents sulphur.

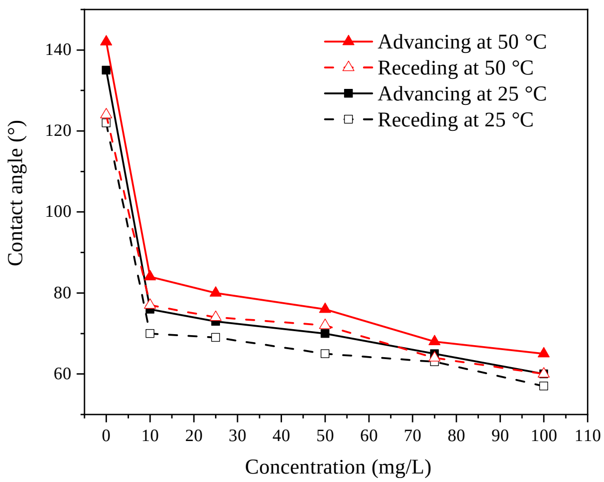


Fig. 5. The impacts of different MB concentrations on and at 0.3 M and 20 MPa.

unt of carbon is significantly reduced, and the presence of sulphur has increased, after modification with MB (Fig. 4(c)). This indicates that the surface of the stearic-acid aged quartz is changed to a hydrophilic state upon modification with MB. Moreover, this result is consistent with a previous study with Üner (2019) who used an EDS analysis to investigate the modification of activated carbon produced from Arundo donax before and after treatment with MB. In that case, the percentages of carbon and silicon decreased from 84.41% and 0.9%, respectively, before MB adsorption, to 78.96% and 0.43%, respectively, afterwards, while the sulphur content increased from 0.2% to 2.12%. This confirms that the organic content of the surface decreases, and the surface becomes more hydrophilic, when treated with MB solution.

3.2 Effect of different concentrations of methylene blue on stearic acid-aged quartz

The effects of varying MB concentrations on the stearic acid-aged quartz at temperatures of 25 and 50 °C with a fixed salinity of 0.3M and a revealed by the contact angle measurements in Fig. 5. Thus, after aging in stearic acid (but before MB modification) the surface of the quartz is oil-wet due to the adsorption of organic molecules on the rock surface, thereby resulting in higher θ_a and θ_r values. Hence, with 0 mg/L MB at 25 °C, the θ_a and θ_r are 135° and 122° respectively. However, when the temperature is increased to 50 °C, these CA values are increased to 142° and 124°, respectively, due to the strong molecular attraction between the organic molecules and the rock surface. After modification with various concentrations of MB, however, the contact angles are consistently decreased, with the optimum effect being observed at an MB concentration of 100 mg/L MB. Thus, at 25 °C, the θ_a decreases from 76° at 10 mg/L MB to 60° at 100 mg/L MB, while the θ_r value is decreased from 70° to 57°. Similarly, at 50 °C, the θ_a decreases from 84° to 65°, and the θ_r decreases from 77° to 60°, as the MB concentration is increased over the same range.

It is not possible to compare the present contact angle results with relevant literature values because there are presently no published studies on the effects of MB on the contact angles of the oil-brine-quartz system, thus highlighting the novelty of the present study. However, a previous study by the present authors examined the effects of various concentrations of methyl orange (MO) on the wettability of stearic acid-aged quartz to find that the θ_a and θ_r values were significantly reduced to 31° and 29°, respectively, in the presence of 100 mg/L MO at 25 °C and 0.3 M salinity under a pressure of 20 MPa (Alhammad et al., 2022, 2023b; Alhamad et al., 2023). Generally, the θ_a and θ_r values were less after treatment with MO than those observed after treatment with MB. This may be because there are fewer carbon atoms in the MO molecule ($C_{14}H_{14}N_3NaO_3S$) than in the MB molecule ($C_{16}H_{18}ClN_3S$). Nevertheless, the present results indicate that the surface energy of the stearic acid-aged quartz is decreased in the presence of MB, thus signifying weaker attractive forces between the organic acid molecules and the quartz surface.

3.3 Influence of temperature and pressure on stearic acid and MB-aged substrates

The temperature and pressure effects on contact angle datasets are illustrated in Fig. 6. Overall, the CA increased with temperature and pressure in consistency with previously reported experiments (Abdulelah et al., 2021; Aftab et al., 2023). Duffy et al. (2021) used the surface tension component method to model contact angles of a quartz/decane/water for temperatures from 25 to 200 °C. They found that the CA increased from 16° to 57.4° with temperature from 25 to 200 °C. Wang and Gupta (1995) demonstrated through the pendant drop

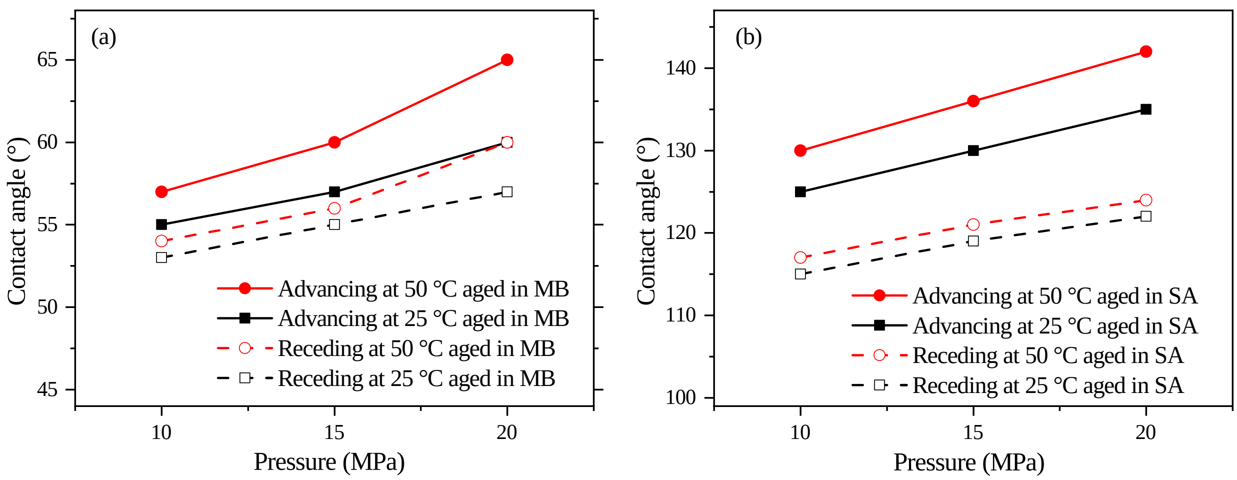


Fig. 6. Impact of different temperatures and pressures on quartz aged in (b) stearic acid and (a) methylene blue solution.

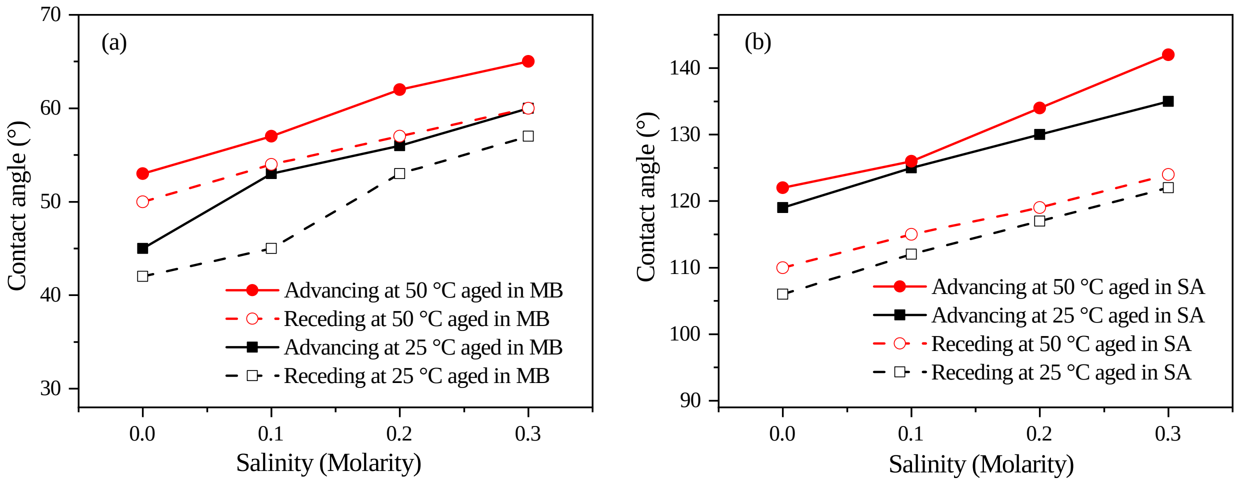


Fig. 7. Effects of different salinities on quartz aged with (b) stearic acid and (a) methylene blue.

technique that increased temperature increased the CA of the crude oil/quartz/brine system. Our previous assessment of the impacts of MO on quartz substrates wettability at reservoir conditions showed increasing and with pressure (10-20 MPa) and temperature (25-50 °C).

It is shown in Fig. 6 that the stearic acid-aged quartz surface became hydrophobic as the increased from 125° to 135°, and was elevated from 115° to 122°, respectively, when the substrates were aged in organic acid. Similarly, at 50 °C and increasing pressure from 10-20 MPa, the rosed from 130° to 142°, whereas the increased from 117° to 124°, respectively (more hydrophobic condition).

Likewise, Ali et al. (2021) demonstrated a drastic elevation in and of quartz when the quartz substrates were aged in humic acid at elevated pressures (0.1-25 MPa) and temperatures (303 and 333 K). The increased hydrophobicity of the rock surface is due to the adsorption of organic acid molecules onto the quartz surface, which has also been confirmed by previous studies (Alanazi et al., 2023; Hou et al., 2023). However, when the quart and values with pressure and temperature, the contact angles datasets were less for MB-treated quartz than stearic acid-treated quartz substrates. For instance, when pressure changed from 10 to 20 MPa at a temperature of 25 °C, the increased from 55° to 60°, and rose from 53° to 57°, respectively. Similarly, when pressure increased from 10 to 20 MPa and at 50 °C, the increased from 57° to 65° and the increased from 54° to 60°, respectively, suggesting that the effect of MB treatment on quartz surface resulted in a drastic reduction of rock hydrophobicity, thus, increasing hydrocarbon recovery.

3.4 Effect of salinity on stearic acid and MB aged quartz

It is vital to assess the influence of salinity on quartz/n-decane/brine for effective determination of wettability change that occurs at realistic reservoir conditions in the presence of resident formation brines. Thus, we conducted experiments at different salinity (0, 0.1, 0.2, and 0.3 M, NaCl) conditions (Fig. 7). We found that the changed from 119° to 135°, and rose from 106° to 122° with increasing salinity from 0 to 0.3 M at 25 °C, and 20 MPa, respectively, when quartz substrates were aged in stearic acid. Similarly, at 50 °C, and 20 MPa, the θ_a increased from 122° to 142°, and the θ_r increased from

110° to 124°, respectively, when quartz substrates were aged in stearic acid.

These results confirmed that at higher salinity, there is an enhanced electrostatic interaction between n-decane and quartz surface, leading to a wettability shift towards the hydrophobic condition (Awan et al., 2021). Also, we observed that the degree of change in contact angle was more significant in the absence of MB compared to MB-modified stearic acid-aged quartz substrates. For instance, at 25 °C and 20 MPa, with the increase in salinity from 0 to 0.3 M increased θ_a from 45° to 60° and θ_r from 42° to 57°, respectively, when quartz substrates were aged in MB. Similarly, at 50 °C and 20 MPa, with the increase in salinity from 0 to 0.3 M increased θ_a from 53° to 65° and θ_r from 50° to 60°, respectively, when quartz substrates were aged in MB.

In previous research, the effect of salinity on contact angles of oil-brine-rock systems was found to be salt concentration-dependent dependent (Shedid and Ghannam, 2004; Haagh et al., 2017, 2018; Zhao et al., 2020). Shedid and Ghannam (2004) found that contact angles decreased with a change in salinity over the range of 0-50,000 ppm, but when salinity increased from 50,000-100,000 ppm, the contact angle increased. The increasing contact angles within 50,000-100,000 ppm concentration were ascribed to the decreasing accumulation of the surface-active species on crude oil/aqueous phases interface in the presence of salt, thus increasing the contact angles and interfacial tension (IFT) (Standal et al., 1999). Similarly, in this study, the θ_a and θ_r increased with the increase in salinity from 0 to 0.3 M due to increased repulsive hydration forces and enhanced repulsive electrostatic double layer forces, thus preventing the MB from dissolving in the aqueous phases and spreading on the quartz surface. This process could prevent MB adsorption on quartz surface and promotes spreading of oil on the rock surfaces.

4. Conclusion

Wettability is a fundamental factor governing fluid flow in porous media, which has a direct impact on enhanced oil recovery and, thus, underground gas (H_2 or CO_2) storage (Kamal et al., 2017; Alhammad et al., 2023a; Aslannezhad et al., 2023). Changing the rock surface to a hydrophilic condition by injecting various chemicals such as surfactants and nanoparticles has been proposed as an effective technique for improving hydrocarbon recovery and increasing the gas storage (Al-Anssari et al., 2019, 2021; Al-Khdheeawi et al., 2020; Haghighi et al., 2020). However, there is a lack of data on rock wettability alteration in the presence of methylene blue. Consequently, we evaluated the feasibility of using MB, a chemical commonly disposed to the environment, to modify organic-aged quartz wettability for enhancing hydrocarbon production.

The θ_a and θ_r were measured for the rock modified with different MB concentrations (10, 25, 50, 75, and 100 mg/L) as a function of temperature (25 and 50 °C), pressure (10, 15, and 20 MPa), and salinity (0 to 0.3 M). The θ_a and θ_r increased with pressure, temperature, and salinity. However, when quartz was aged with different MB concentrations, the θ_a and θ_r values decreased at all investigated physio-thermal conditions, suggesting that treating organic acid-contaminated quartz with an optimum concentration of MB 100 mg/L restored the original hydrophilic wettability conditions. This study suggests injecting toxic chemicals such as MB into underground reservoirs can protect the environment from pollutants, thus optimizing hydrocarbon recovery.

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Conflict of interest

The authors declare no competing interest.

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