



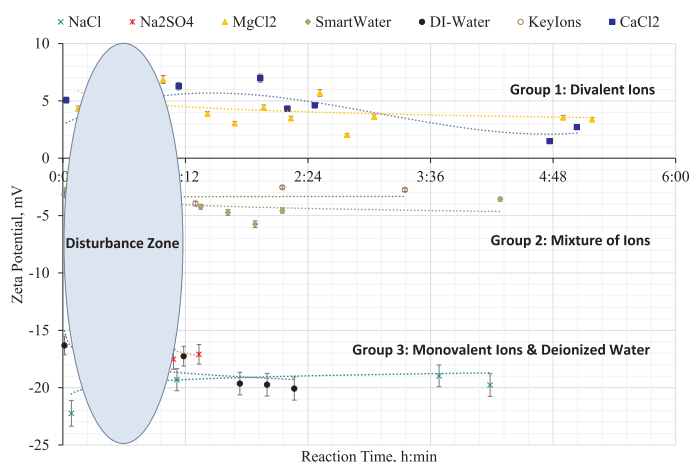
Dynamic interactions of inorganic species at carbonate/brine interfaces: An electrokinetic study



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GRAPHICAL ABSTRACT



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ABSTRACT

Adjusting brine chemistry during waterflooding of carbonate and clastic reservoirs shows favorable impact on oil recovery. In carbonates, inorganic ions in the injection water play a key role on altering rock-fluid interactions and subsequently influencing rock wettability. Emphasis in this study is given to the direct measurements of carbonate particles zeta potential at different ionic content and aging times. A disturbance at interfaces was observed for most brine solutions especially within the first hour of interactions. The zeta potential of particles became steady with time as a result of attaining chemical equilibrium at interfaces. Individual ions including cations, and anions altered carbonate zeta potential and interacted differently at interfaces, in spite of having an identical salinity. Monovalent ions, for example sodium, significantly reduced the absolute surface potential of limestone particles. The hardness ions, calcium and magnesium, reversed the zeta potential of both calcite and limestone particles towards the positive side. The electrical properties, in general, were found to be affected by the ionic content and cation to anion ratio in the injection water. The dissolved divalent cations play a role in the interactions at the Stern layer boundary, which eventually impact the zeta potential at interfaces. In view of these zeta potential results, specific brines – SmartWater (10-times diluted seawater), key ions (calcium, magnesium and sulfates) and salt solutions containing strictly sodium or sulfate – were able to create electrical repulsions between oil/water and carbonate-particles/water interfaces. As a result, such tailored brines will alter the rock wettability to water-wet and therefore can enhance the oil recovery in carbonate reservoirs. This further

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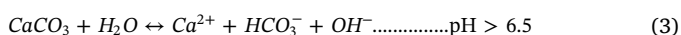
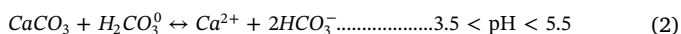
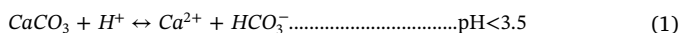
understanding of ions specific impact on surface potential, hence wettability, provide essential insights for the successful design of optimal SmartWater recipes.

Greek symbols

ζ Zeta potential, mV

1. Introduction

The impact of salinity and ionic content on oil recovery and wettability has been thoroughly studied by several research institutes, as well as oil and service companies worldwide [1–5]. Different potential mechanisms for the observed wettability alteration were proposed including: ion adsorption, calcite dissolution, ionic exchange, and improving fluid diffusion. The main interactions are expected to take place at rock/fluid and fluid/fluid interfaces. Certain ions (Potential Determining Ions: PDI) play a more significant role than others. SmartWater, for example, has been formulated in different ways by either dilution or increasing concentration of either calcium, magnesium or sulfates in seawater. The required adjustment in salinity could also vary from one reservoir to another and it highly depends on the specific interactions between the crude oil, brines and rock combination. Most of the previous laboratory studies used the conventional experimental techniques such as spontaneous imbibition [1], coreflooding tests [2], contact angles [6], and NMR [7] with very limited work on zeta potential [8]. In this paper, attention is given to carbonate reservoirs because the interaction mechanisms responsible for wettability alteration are not yet well understood. The reaction mechanisms in clastic reservoirs will not be covered since the mineralogy is completely different and since salinity effects in clastic reservoirs has been thoroughly studied in the literature. Reactions on calcite surfaces appear to be much more complex than those involving silicates. Hence, the electrical properties at the calcite-water interfaces are still controversial. The solubility and dissolution of calcite, which is governed by the surface electrical charge and chemical equilibrium, is one of the main reasons in complicating the calcite interaction with aqueous solutions [9]. The dissolution process in reality can go through three mechanisms according to the pH range [10]. At a pH less than 3.5, the rate of dissolution is directly proportional to the hydrogen ion activity and is unrelated to the P_{CO_2} . Moreover, the dissolution rate depends on both pH and P_{CO_2} at pH values between 3.5 and 5.5. Above a pH of 6.5, water is involved in the dissolution reaction. The three mechanisms are described by the following reactions:



Fluids and rock interactions are also controlled by the different forces which can impact the disjoining pressure between the hydrocarbons and carbonate solid surfaces. These forces include London-van der Waals attractive force, electrical double layer forces, and short-range repulsive forces. In this paper, the electrical double layer forces are thoroughly studied by measuring the zeta potential of calcite and carbonate particles. The short-term interactions between the rock surface and SmartWater ions can directly impact surface roughness and ions structures at the interfaces. As a result, the dynamic interactions will eventually affect the electric double layer characteristics, rock wettability and oil recovery accordingly. Streaming and zeta potential are the two conventional techniques available to determine the effect of salinity and pH on surface charges. The zeta potential is defined as the

average potential in the electrical double layer at the zone of shear between a mobile charged particle and the immobile liquid phase in which a particle is suspended [11]. On the other hand, the measured potential difference across the capillary or porous media is known as streaming potential and is mostly related to the pressure gradient and shear plane potential [12].

A comprehensive review of the previous studies on zeta potential of calcite is summarized in Table 1. Various factors can significantly influence the zeta potential of calcite including nature of the calcite sample, solid/liquid ratio, presence of atmospheric CO₂ and the electrolyte medium [14]. These studies demonstrate the strong dependence on the suspension properties. Table 2 also shows the behavior of zeta potential after using outcrop and field limestone samples at different conditions. As shown in Tables 1 and 2, the effects of rock mineralogy, ionic content and fluids pH on the electrokinetic potential of reservoir rocks can be significant. Basically, the crude oil charges at interfaces were determined previously to be negative regardless of the brines' chemistry except at low pH values [13]. The opposite charges at oil/brine and mineral/brine surfaces result in an electrostatic attraction between the two interfaces, which can collapse the brine film and bring the oil in direct contact with the mineral surface. In this case, the system is expected to be a non-water wet. If both interfaces are negatively charged, there will be an electrical repulsion between the two surfaces, which tends to stabilize a brine film between the surfaces. In turn, the disjoining pressure will be increased and tends to make a thick water film [15]. In waterflooding, the oil/water and rock/water interfaces should carry similar surface charges to increase the electrical repulsion between the two interfaces and ultimately enhance the oil recovery.

Accordingly, one of the most important factors that determines the wettability of crude-oil/brine/mineral system is the electrical or zeta potential of the oil/brine interfaces and mineral/brine interface [16]. The main objectives of this study are to determine the ionic content and aging time effects on the zeta potential of calcite and limestone particles. In addition to SmartWater (10-times diluted seawater) and deionized water, individual monovalent and divalent ions in seawater were evaluated at an identical total dissolved solids (TDS). This work also attempts to address some of the main shortcomings of previous studies, which can be summarized as the following:

- Role of individual ions at fixed salinities on zeta potentials of calcite has not been studied.
- Very limited studies investigated the time effects on the zeta potential of carbonates.
- Effects of impurities in real reservoir rocks on zeta potential has not been studied in comparison to pure calcite.

2. Materials

2.1. Solid particles

Two types of solid particles were used in this work: idealistic pure calcite and actual reservoir limestone. For the first, a pure calcium carbonate disk was obtained from Italy as a white, fine-grained rock. The purity of calcite sample was confirmed by X-Ray Diffraction and Scanning Electron microscopy. The X-Ray Diffraction result indicated that the calcite disk contained more than 99 wt.% calcium carbonate. The Scanning Electron microscopy and Energy Dispersive spectroscopy also revealed that calcium carbonate was the main mineral component. As for the actual formation limestone, whole cores were obtained from

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