

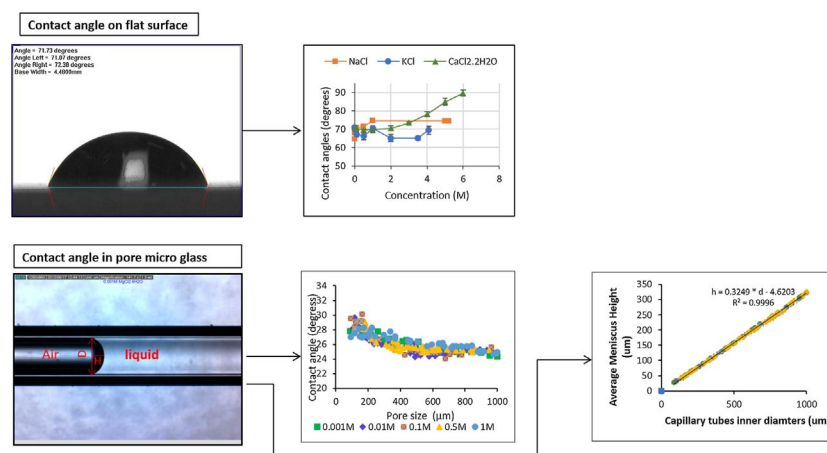
Effect of aqueous electrolyte concentration and valency on contact angle on flat glass surfaces and inside capillary glass tubes



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GRAPHICAL ABSTRACT



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ABSTRACT

Multiphase flow in underground formations is controlled to a large extent by capillary forces that depend on the interfacial tension, contact angle and pore diameter. The interfacial tension and contact angle depend mainly on the type of brine and its concentration. In this study, the static contact angles on flat glass surfaces have been explored as a function of brine concentrations, while the static contact angles inside glass micro-tubes have been studied as a function of pore diameter and brine concentrations for monovalent (NaCl, KCl) and divalent (MgCl₂, CaCl₂) brine solutions. The concentrations ranged from 0.001 to 6 M under ambient conditions, depending on the investigated salt, while the inner diameters of the glass micro-tubes ranged from 100 to 1000 μm. A linear correlation ($h = 0.3249 \times d - 4.6203$) between the glass micro-tube inner diameter and the meniscus height was obtained for the above solutions for concentration of less than or equal to 1 m. This correlation can be used with the Cheong equation to calculate the contact angle of the investigated solutions when only the micro-tube pore diameter is known. The results also showed that salt concentration and valency have more influence on contact angles on flat glass substrates than inside micro-sized glass tubes. In general, the contact angles on a flat glass surface increased with increasing concentration. On the other hand, the contact angles inside glass micro-tubes

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showed no variation with increasing concentration but increased slightly as the capillary inner diameters decreased. The difference might be associated with the curvature of the three-phase contact line. The contact angles on flat glass surfaces ($\approx 63\text{--}90^\circ$) were much higher than those inside the glass capillaries ($\approx 24\text{--}33^\circ$).

1. Introduction

The wetting property of surfaces is of high importance for a wide range of applications that include detergency, self-cleaning, liquid surface coating, flotation, chemical reactions at solid-liquid surface, mass transfer in packed columns and fuel cells, multiphase flow in reservoirs, and enhanced oil recovery [1,2,3,4]. The characteristics of multiphase flow through porous media is influenced to a large extent by capillary forces, which depend on liquid-liquid and gas-liquid interfacial tensions, contact angle, and pore diameter. Hence, an advance in the understanding of the wettability of solid surfaces is of high practical importance in many fields.

Wettability refers to the tendency of a fluid in a multiphase system to adhere or spread on solid surfaces [5–7]. Many qualitative and quantitative methods have been proposed in the literature [8,1] to determine the wettability of a fluid-liquid-solid system. Among these quantitative techniques are the Amott test, the USBM, and the contact angle measurement [8,9]. The Amott test and the USBM method are used to evaluate the average wettability of a reservoir rock while the contact angle is used to determine the wettability of a specific solid substrate [8]. The contact angle refers to the angle formed between the liquid drop/bubble surface and the solid/liquid interface which is measured through the dense phase. Due to its simplicity and being less time consuming, the contact angle measurement is still the most used method, despite some intrinsic problems, e.g. its high sensitivity to contamination [5,10]. According to Anderson, when the contact angle is $0^\circ\text{--}75^\circ$, $75^\circ\text{--}115^\circ$, or $115^\circ\text{--}180^\circ$, the solid surface is considered to be water-wet, intermediate-wet, or oil-wet, respectively [11].

The wettability of a liquid on a solid surface is generally dependent on the physical and chemical properties of the solid surface, and the surface tension of the liquid [1]. The system wettability can be affected by the adsorption and desorption of polar compounds and/or organic materials [11], brine composition, temperature and duration of aging in oil, initial water saturation, and surface roughness and heterogeneity [9,12].

Brine composition (salinity, valency and pH) has a direct impact on the system's wettability, since it governs the charge of the solid surface, which, in turn, determines the kind of materials adsorbed. The silica surface charge in water is negative at high pH and positive at low pH but turns to negative when the pH level increases above 2–3.7. At neutral pH, the silica surface charge is normally negative and has a weakly acidic surface and, therefore, the adsorption of organic bases is favoured [13]. The presence of divalent ions such as Ca^{2+} , Mg^{2+} and SO_4^{2-} has an important role in changing the surface charge and wettability modifying process [14–16].

From the literature it is very clear that the impact of salinity on the surface tension, wettability alteration of core rocks, oil/water interactions, and oil recovery have been studied extensively by many investigators [17,18,13,10,19–21] but studies on the influence of electrolyte concentrations on the equilibrium contact angle [22,23] are rare. Only two studies were found. For instance, Sghaier et al. [22] measured equilibrium contact angles as a function of NaCl concentrations on both hydrophilic and hydrophobic glass surfaces. The hydrophilic surfaces showed a greater increase in contact angle with increasing salt concentration than the hydrophobic surfaces [22]. Leelamanie and Karube [23] examined the impact of aqueous electrolyte concentration on the contact angle of soil samples (Andisol and silica sand) with different hydrophobicities, using NaCl and CaCl_2 . They noticed that the increase in concentration leads to an increase in the contact angle, due to increasing surface tension, and that the relation

between concentration and contact angle is not linear. In addition, the contact angle showed almost negligible response to increasing concentration when the concentrations of NaCl and CaCl_2 exceeded 0.06 and 0.1 mol L^{-1} , respectively [23].

However, to the best of our knowledge, no study has been reported for the effect of electrolyte concentrations on the equilibrium static contact angle inside glass capillary tubes. The contact angle inside capillary porous media has been proposed in many simulations and model studies as an equivalent to the contact angle on flat surfaces [24]. However, a recent study by Li and co-workers found that the contact angles inside capillary tubes for deionised water, 1-propanol, n-decane and crude oil are different from their contact angles measured on flat surfaces [24]. One of the aims of this study is to extend these investigations to the salts that commonly exist in saline and formation waters. In this study, the static contact angles on flat glass surfaces have been explored as a function of brine concentrations, while the static contact angles inside glass micro-tubes have been studied as a function of pore diameter and brine concentrations for monovalent (NaCl, KCl) and divalent (MgCl_2 and CaCl_2) brine solutions. The concentrations ranged from 0.001 to 6 M under ambient conditions, depending on the investigated salt, while the capillary tubes' inner diameters ranged from 100–1000 μm .

2. Materials and methods

All the salts were analytical reagent grade and purchased from SIGMA-ALDRICH, except magnesium chloride which was supplied by Fisher Scientific (UK). Both the microscope glass slides (Thermo Fisher Scientific UK, 10143562CE) and glass micro-tubes were made of clean glass (Thermo Fisher Scientific UK, TWL-611-010M). Each glass micro-tube has a wall thickness of 1.5 mm and an outer diameter of 6.0 mm. The composition of both the glass tubes and plain glass microscope slides were white soda-lime glass (SiO_2 : 72.20%, Na_2O : 14.30%, CaO : 6.40%, K_2O : 1.20%, MgO : 4.30%, Al_2O_3 : 1.20%, Fe_2O_3 : 0.03% and SO_3 : 0.30%) [24]. The glass microscope slides and capillary tubes were cleaned with hot water for 2–3 min under tap pressure, then rinsed thoroughly with deionized water. To avoid potential surface contamination, disposable gloves were used during handling and conducting contact angle measurements. The glass samples were placed under intensified heat (550°C) until they became red in colour, to remove any trace of organic contamination [25]. To prepare the micro-glass tubes, the glass tubes were melted on a butane flame (Butane Battery, D2-BS 0167) and stretched to a long distance to obtain a uniform micron-sized glass pore. The diameters of the glass capillaries investigated ranged from 100 to 1000 μm . To avoid contamination, the micro-capillary tubes and microscopic slides were stored in a dust proof closure and used as soon as possible for contact angle measurements. Each time, the measurements were conducted on a new microscopic glass slide and a new micro-sized glass tube.

2.1. Contact angle measurements on a flat surface and inside micro-glass tubes

The static contact angle on a flat glass surface, Fig. 1, was measured with the sessile drop technique. A liquid drop of $0.5 \mu\text{l}$ volume was placed carefully on the glass substrate using a pipette to minimize the effect of the droplet size and vibration on the measurement. The $0.5 \mu\text{l}$ liquid drop has a radius of less than 2.7 mm. This means that the gravity effects on the drop shape can be neglected, according to Spyridon and Kranias' calculations [26]. An average of five measurements was taken

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