Contact angle measurement of fluid with salinity variation on sandstone rock surface

C W Winardhi and U Fauzi
Department of Physics, Institut Teknologi Bandung, Jalan Ganesha 10, Bandung 40132, Indonesia
E-mail: chandra.winardhi@gmail.com

Abstract. Interaction between fluids and solid surfaces plays important roles in many fields, e.g.: enhanced oil recovery (EOR), environmental engineering, agriculture, materials, etc. Research on this topic is intensively carried out by many researchers. In this research, a simple experimental setup for fluid contact angle measurement is constructed using an optical microscope and video camera with 29 frames per second. Measurement of contact angle of fluid with variation of salinity, i.e.: 1M, 2M, 3M, 4M, and 5M is carried out by recording a single droplet placed on a rock surface. Each frame of the recorded video are then analysed using image processing technique to detect the edge and calculate the contact angle. A single 8th order polynomial was applied to the edge of the wetting type droplet, but two types of polynomial fitting curves are applied for edge of non-wetting type droplet. It is obtained that on preparatory glass, the higher the molarity, the longer the droplet will evaporated. On the sandstone surface, fluid contact angle on sandstone surface is about 80° and decreases with time with different trend of decrement. This may be due to complex interaction between fluid and pores at sandstone surface spot.

1. Introduction
Interaction of fluids and solid interaction is important [1] and required in many fields such as hydrocarbon exploitation, environmental engineering, agriculture, materials, etc. For example, application in environmental engineering is non-aqueous phase liquid / NAPL, in geophysics engineering (carbon dioxide geo-sequestration) [2] and also in petroleum engineering for enhanced oil recovery (EOR). Nowadays, many oil industries was using EOR especially for unconventional oil production. There were many methods used in EOR to alter the fluid and solid interaction for example surfactant polymer injection [3], low salinity brine injection [4], thermal injection [5], or carbon dioxide injection [6]. These interaction was controlled by two macroscopic parameters which are surface tension and contact angle [7]. It is therefore, better understanding of these interaction between fluids is important.

Srinivasan et al. [8] conducted research on electro-wetting effect on wettability, by measuring the changes of contact angle. Shedid et al. [9] investigated oil interaction on rock surface with temperature and salinity variations. Al-Rossies et al. [10] varied pH value against contact angle and surface tension. Study of carbonated water effect on rock wettability was done by Seyyedi et al. [11]. This

1 To whom any correspondence should be addressed.
topic is intensively discussed and becomes hot issues. In this study measurement contact angle of fluid was carried out to understand fluid behaviour on rock surface with salinity variation.

2. Samples and Experimental Technique

2.1 Samples
The sample tested in this study was sandstone reservoir rock which is a typical hydrocarbon reservoir rock (see figure 1). The fluid used in this experiment, was made by dissolving NaCl salt in distilled water. Five concentration, i.e.: 1M, 2M, 3M, 4M, and 5M are produced, the amount of NaCl for each solution is listed in table 1.

![Sandstone sample](image1)

**Figure 1.** Sandstone sample (left) and sandstone microscopy image of sandstone sample surface (right). (Red circle shows some noticeable cavity on the rock surface.)

| Distilled water volume (mL) | NaCl Mass (mg) | Molarity (M) |
|---------------------------|---------------|--------------|
| 20                        | 1.169         | 1            |
| 20                        | 2.338         | 2            |
| 20                        | 3.506         | 3            |
| 20                        | 4.675         | 4            |
| 20                        | 5.844         | 5            |

2.2 Experimental Technique
Room condition of the laboratory was maintained before conducting experiment data. The room temperature is set around 18~20°C. The fluid was stored in an open container filled with water to adjust the temperature. Droplet image was observed using Nikon YS100 microscope with a slight modification (see figure 2). A recording camera was installed into the setup and then were placed inside an acrylic chamber. The volume of the droplet was dropped using 5µL hand micropipette. After the droplet placed, observation was conducted until droplet disappeared and then each frame is processed by means of image processing.
2.3 Image Processing

Before contact angle was calculated from the video, several steps required to enhance the quality of each image for better edge detection. The first step, spatial filter Gaussian and Laplace were applied to reduce noise and enhanced the edge of the droplet. The color image (RGB) was converted to grayscale image and then Otsu algorithm [12] was used to get the binary image (black and white image). Afterwards, Sobel algorithm [13] was applied to detect the edge of the droplet for contact angle calculation as shown in figure 3.

![Figure 2](image1.png)

**Figure 2.** Experiment setup using microscope Nikon YS100 inside an acrylic chamber (c). Micropipette placed on top of the chamber (a), the rock sample (d) was placed below. A temperature and humidity sensor are also placed inside the chamber (e) and (f) is video camera to acquire the data.

![Figure 3](image2.png)

**Figure 3.** Real image has been filter using Laplace and Gaussian (top left), image then converted into grayscale (top right) and then binary (bottom left). Finally into edge images (bottom right).

Droplets were divided into two types, i.e.: wetting type droplet and non-wetting type droplet respectively. To differ these two types, polynomial of 8th order is fitted to the edges. A non-wetting
type droplet will have a low r-square and the edge were not well fitted with a single polynomial. Therefore, non-wetting type was divided into left and right part. Then both parts are fitted with different polynomial function (see figure 4 left).

![Figure 4](image)

**Figure 4.** Overlay scatter plot of blue and red color circle around droplet edge and grayscale image with the two fitted curved, blue for left part and red for right part (left) and with one blue fitted curved (right).

The gradient of each tip was obtained from the tangent line of the polynomial function. After that contact angle was calculated from the tangent line (see figure 4). This method then tested on the sandstone surface with salinity variation.

3. Result and discussion

Both experiment and the image processing allow contact angle data to be measured for every frame easily. Contact angle measurement with salinity variation on preparatory glass shown on figure 5. Solution with 1M and 2M concentration have contact angle about 50°-60° at the first frame. Saline solution with 3M and 4M have contact angle around 60° and 5M concentration have contact angle around 50°. Every contact angle for each salinity will decrease with time due to evaporation of the droplet. Solution with 1M concentration was the fastest to evaporate, followed by 2M, 3M, 4M and 5M. It can also be seen on 4M and 5M concentration, the contact angle suddenly increases, which may occur due to crystallization of salt. Solution with higher concentration will evaporate longer.

![Figure 5](image)

**Figure 5.** Contact angle measurement data with salinity variation on preparatory glass.

Afterwards, droplet then dropped on sandstone surface. Figure 6 shows the result of contact angle measurement tested on sandstone surfaces. The contact angle for each salinity is about 80° at the first frame. Every contact angle for each salinity will also decrease as a function of time with different trend of decrement. Saline solution with 1M concentration have the longest decreasing time, followed by 2M, 5M and 3M concentration. Solution of 4M concentration has the quickest decreasing time. Contact angle of 1M and 2M saline solution decrease from around 80°, 4M concentration decrease
from 70° and 3M from 50°. All of the solution will decrease to below 20° except for 5M solution, it stops between 40° - 50° because crystallization of the salt. The crystallization happens as a result from evaporation of the droplet. The different trend of decrement may be due to evaporation and absorption of the sandstone pore and also complex interaction between the solution and the pore at sandstone surface spot.

**Figure 6.** Contact angle measurement data with salinity variation on sandstone rock surface.

### 4. Conclusion

Contact angle of fluid can easily obtain using this experimental technique. Contact angle measurement for wetting type can be fitted using 8th order polynomial directly, but non-wetting type need to be divided into two parts and fitted respectively. From the performed experiment, both experiment setup and image processing can measure the change of contact angle on a sandstone surface. Contact angle for every salinity concentration will gradually decrease both on preparatory glass and sandstone surface. Different trend of decrement on preparatory glass occur due to evaporation of the droplet. Higher concentration of salt, will make droplet evaporate longer. Contact angle measurement on sandstone also has a different trend of decrement due to evaporation and absorption of the sandstone surface.

### Acknowledgment

This research was funded by the research program from Ministry of Research, Technology, and Higher Education of The Republic of Indonesia (Kementrian Riset Teknologi dan Pendidikan Tinggi Republik Indonesia).

### References

[1] Santini M, Guilizzoni M, and Fest-Santini S 2013 X-ray computed microtomography for drop shape analysis and contact angle measurement *Journal of colloid and interface science* 409 pp 204-210

[2] Singh K, Bijeljic B, and Blunt M J 2016 Imaging of oil layers, curvature and contact angle in a mixed-wet and a water-wet carbonate rock *Water Resources Research* 52(3) pp 1716-1728

[3] Elraies K A and Tan I M 2012 The application of a new polymeric surfactant for chemical EOR *Introduction to Enhanced Oil Recovery (EOR) Processes and Bioremediation of Oil-Contaminated Sites* InTech

[4] Abdalla A, Sun R, Guiyun T, Huang A, dan Wang M 2017 Low Salinity as New Technique of Enhanced Oil Recovery *International Journal of Chemical Engineering and Applications* 8(2) p 117

[5] Pathak V, Babadagli T, and Edmunds N 2012 Mechanics of heavy-oil and bitumen recovery by hot solvent injection *SPE Reservoir Evaluation & Engineering* 15(02) pp 182-194
[6] Blunt M, Fayers F J and Orr Jr F M 1993 Carbon dioxide in enhanced oil recovery Energy Conversion and Management 34(9-11) pp 1197-1204
[7] Andrew M, Bijeljic B, and Blunt M J 2014 Pore-scale contact angle measurements at reservoir conditions using X-ray microtomography Advances in Water Resources 68 pp 24-31
[8] Srinivasan V, Pamula V K, Rao K D, Pollack M G, Izatt J A, and Fair R B 2003 3-D imaging of moving droplets for microfluidics using optical coherence tomography Proc. Micro Total Analysis Systems (mTAS) pp1303-1306
[9] Shedid S A and Ghannam M T 2004 Factors affecting contact-angle measurement of reservoir rocks Journal of Petroleum Science and Engineering 44(3-4) pp 193-203
[10] Al-Rossies A A S, Al-Anazi B D and Paiaman A M 2010 Effect of pH-values on the contact angle and interfacial tension Nafta 61(4) pp 181-186
[11] Seyyedi M, Sohrabi M, and Farzaneh A 2015 Investigation of rock wettability alteration by carbonated water through contact angle measurements Energy & Fuels 29(9) pp 5544-5553
[12] Otsu N 1979 A threshold selection method from gray-level histograms IEEE transactions on systems, man, and cybernetics 9(1) pp 62-66
[13] Sobel I 1990 An isotropic 3×3 image gradient operator Machine vision for three-dimensional scenes pp 376-379