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Determination of Minimum Miscibility Pressure in supercritical extractor using oil saturated sample

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The main parameter for determination of the possibilities to enhance oil recovery by e.g. CO_2 injection into a specific oil field is the measurement of Minimum Miscibility Pressure (MMP). This pressure is the lowest pressure for which a gas can obtain miscibility through a multi contact process with a given oil reservoir at the reservoir temperature. The oil formation to which the process is applied must be operated at or above the MMP. Before field trial this parameter is to be determined at the laboratory which traditionally is done by help of a slim tube or a raising bubble experiments. However, in order to investigate the MMP, we suggest another method by using a supercritical extractor.

Spe-ed SFE equipment with oil saturated natural rock samples were used for the purpose. The clean chalk samples were saturated with oil from the Dan field under vacuum. The CO_2 gas was injected into the extractor vessel containing the sample at different increasing pressure levels. The oil displaced in such a way was collected and measured. The volume of extracted oil was plotted against the increasing pressure. The form of the graph is similar to that typically obtained from a slim tube experiment. Following the breakover point criterion the MMP read from the plot was found equal to 20 MPa.

1. Introduction and methodology

After application of the primary and the secondary oil recovery methods which include natural drive mechanisms and water injection, 2/3 of oil reserves in average in the world remain in the subsoil. For Denmark in the North Sea this value is 75%. To extract additional oil many tertiary methods exist. One of them is an injection of carbon dioxide in the oil formation. This method plays also an important role in the reduction of green house effect representing how utilization of carbon dioxide can be combined with the rational usage of natural resources and the saving of energy sources.

The traditional ways of MMP determination in oil industry are experiments on slim-tube and raising bubble apparatus intended especially for this purpose. The necessary components for such kind of experiments are only oil and additionally a carrier like glass beads in a slim tube experiment. In raising bubble experiment the process of miscibility is observed visually in a glass chamber when carbon dioxide begins to stay in the oil without creating bubbles by injection. It was noted that the performance of this

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experiment requires participation of an experienced specialist (Stalkup, 1992). In slim tube experiment normally glass beads or sand packed in a steel tube are saturated with the oil from the field under investigation. The carbon dioxide is injected at increasing pressure. The oil recovered in such way is collected and measured. On the plot the Oil recovery versus Pressure, the MMP is determined following breakover point criterion (Orr, 1982). Both these equipments are not widely available. Only one slim-tube is recorded in Denmark. The raising bubble apparatus is absent at all. For studying the miscibility phenomena we applied the high pressure extractor called ***Spe-ed SFE*** which sometimes can be found in the universities laboratories, where they are used for investigation the supercritical effects on extractions of chemicals from different matrices and catalytic conversions. Within the range of pressures up to 69 MPa and temperatures up to 240^{o}C the analyst can accomplish a wide range of extractions.

1.1 Preparation of the sample

The hydrocarbons of Denmark are produced mainly from the chalk rocks. That is why the capacity of samples of clean chalk to contain oil was determined. . For the experiment a cylindrical sample was prepared. Its geometrical parameters were measured, and the bulk volume of the sample was calculated by the equation $V_b=\pi/4D^2L$. The sample was first placed in an oven for 24 hour in order to remove all moisture from it. The dry and clean sample was weighed and placed in the saturation apparatus as shown in Figure 1. Laboratory set up was made in order to perform the saturation process. Oil was introduced from one side of the flask, and the vacuum was created by mean of vacuum pump. The sample remained 24 hours in the same air tight flask to achieve proper saturation. After the saturation the excess liquid was blot off and removed, the sample was weighed. The weight of the saturation oil was calculated.

1.2 Equipment operation

The extractor vessels filled with the saturated chalk plug was inserted into the oven module through the oven mountings brackets and set to 80^{o}C to simulate reservoir temperature (Fig.1). CO_2 at the desired system pressure was delivered through external cylinder into the extractor vessel for extraction process. The gas was injected at the planned pressure during 30 minutes. Then the inlet valves were closed, the vent and outlet valves opened to let the extracted oil to be collected through the Control and Collection module (C&C module on the figure) which is used for an easy collection of

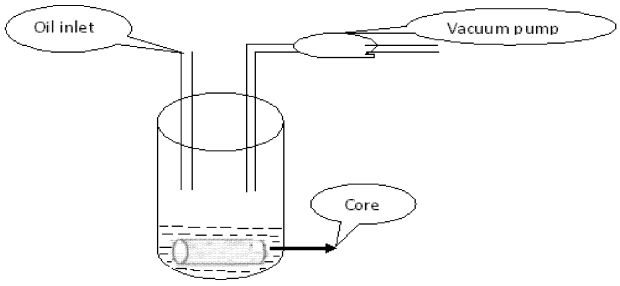

Fig.1 Setup for saturation process

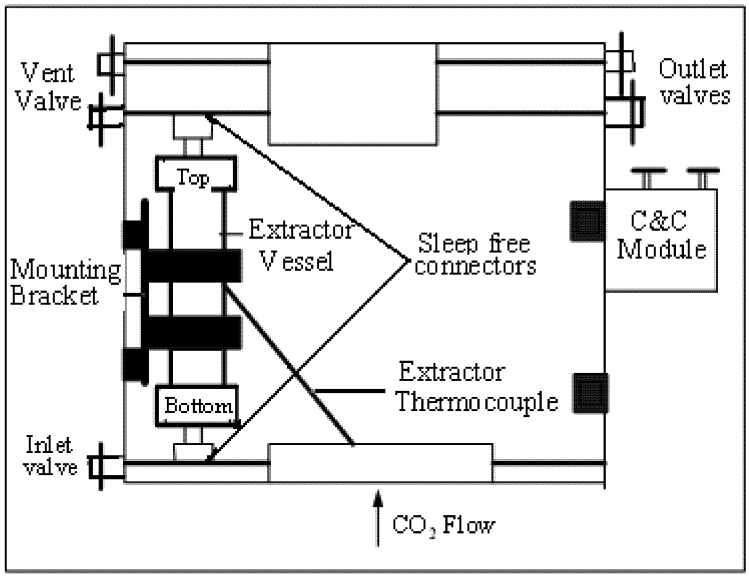

Fig. 2 Schematic diagram ***Spe-ed SFE.***

residues into collection tubes via outlet valves. The oil extracted was collected during 5 minutes. Such way the extraction was repeated from the pressure of 10 MPa till 37.5 MPa with the interval of pressure in 20 MPa. The collection tubes were weighed before and after collection to determine the weight of extracted oil. The schematic diagram below shows the layout of ***Spe-ed SFE.***

2. Results and Calculations

2.1 Determination of MMP

The graph obtained is shown on Fig.3. The oil recovery volumes grow gradually first to the values of 12-14 MPa where they form a kind of step and secondly to the breakover point which is according to a definition made by Orr (1982) is a criterion for MMP determination. Above the breakover point, the volume of extracted oil doesn´t change significantly with the increasing pressure. Such type of correlation is typical for the slim tube experiment. Based on such similarity, the MMP at 80^{o}C for the oil in this investigation was 20 MPa.

2.2 Porosity calculation

The porosity (the volume of the void space inside the rock) of the sample was calculated through the volumes of the oil adsorbed by the chalk in order to find out if the saturation time was sufficient to fill in all the porous space and to check if the closed pores existed where the oil couldn´t have penetrated. The density of oil measured in the laboratory was equal to 0.8025g/cm^3. The initial weight of core before saturation W_I =16.81 gm. The final weight of core after saturation with oil was W_F = 21.65 gm.
The total volume of the oil which the chalk adsorbed was 6.03 cm^3.
For the porosity calculation the following data are needed to be calculated:

The volume of the porous space

$$V_p = \frac{W_F - W_I}{\rho_g} = 6.03\text{cm}^3 \quad (1)$$

The bulk volume

$$V_b = \pi r^2 L = 15.1\ \text{cm}^3 \quad (2)$$

where the radius r = 1.2 cm and the length L=3.4 cm of the cylinder.

Porosity calculated as $\Phi = \frac{V_p}{V_b} * 100 = 40\%$ (3)

The value of porosity of 40% is equal to the porosity value calculated when the sample was saturated with pure water. It means that all the porous space was saturated with oil for the time of saturation 24 hours. No closed pores existed, and the oil penetrated into all the pores that before was saturated with water.

2.3 Calculation of oil recovery

The total oil displaced from the sample was 1.46 gm. By using the value of density of the oil the volume of oil extracted after treating was calculated to be 1.8 cm^3. Therefore, the percentage of oil recovery during the period of experiment was 29 %.

3. Discussion

The shape of our experimental plot of the amount of oil extracted from the oil saturated chalk sample in the process of carbon dioxide injection as a function of pressure is similar to the plots for miscibility of oil in slim tubes (Yellig and Metcalfe, 1980), pressure/composition diagrams (Stalkup, 1992; Turek, 1980) and solubility of some chemicals like naphthalene (McHugh and Paulaitis, 1980) and others in supercritical carbon dioxide. Generally, the graphs from all the above mentioned methods of analysis similarly show the amount of the chemical substance soluble or extracted as a function of pressure. These metods of analysis also demonstrate the gradual growth till the specific pressure above which it doesn't grow substantially. In general this allows implementing such graphs for the determination of the most efficient pressure for the extraction of the chemicals by carbon dioxide. In our case it is a Minimum Miscibility Pressure of CO_2 injection for the oil field operations. Based on this similarity, in the situations when the equipment especially designed for the MMP determination is absent, any high pressure extractor like ***Spe-ed SFE*** in our experiment can be used. Instead of only oil or artificially simulated porous media like sand or glass beads, we used natural chalk samples similar to the real reservoir rocks from the Danish oil fields.

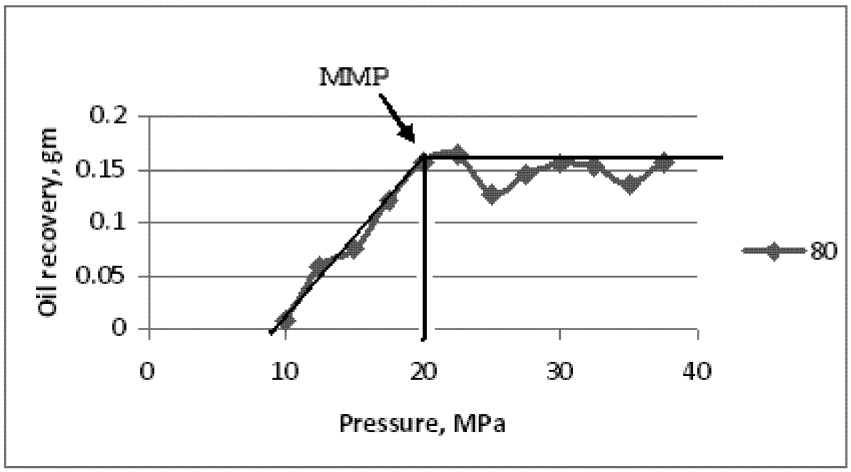

Fig.3. The graph for the determination of Minimum Miscibility Pressure (MMP).

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