



Surface characterization of reservoir rocks by inverse gas chromatography: Effect of a surfactant



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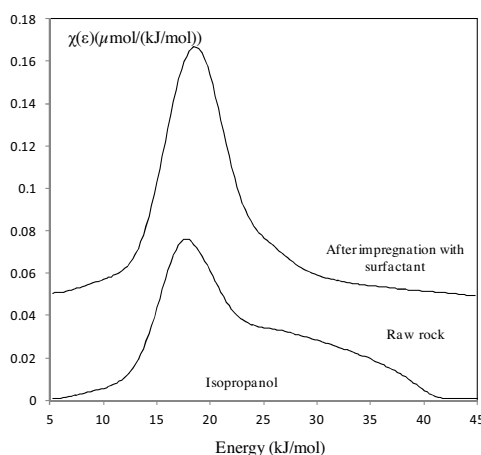
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HIGHLIGHTS

- Four reservoir rocks were characterized by inverse gas chromatography.
- The studied rocks are quartz with a high purity.
- The surface heterogeneity was determined by inverse gas chromatography at finite concentration.
- The heterogeneity indexes, reflecting the high energy sites, are higher than 30%.
- The progressive impregnation with a surfactant hides chemical function.

GRAPHICAL ABSTRACT



The asymmetric distribution function obtained by Inverse gas chromatography at finite concentration on a reservoir rock becomes symmetric after the surface covering by the surfactant. This latter hid chemical functions likely to interact with the injected isopropanol probe.

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ABSTRACT

The description of the surface chemistry of reservoir rock is essential in understanding the physico-chemical interaction between the rock surface and its environment. In this study, inverse gas chromatography (IGC) is used to characterize the surface chemistry of rocks extracted from four petroleum reservoirs in Algeria. By means of IGC at infinite dilution (IGC-ID), the dispersive component of the surface energy (γ_{sd}), the nanomorphological index LM (χ_t) and the specific component of the surface energy were determined at 100 °C. IGC at finite concentration (IGC-FC) was implemented using *n*-octane and isopropanol probes, allowing the irreversibility indexes and the adsorption isotherms to be obtained. From the latter, the BET specific surface area and BET constant of the injected probes were determined. Energy distribution functions and indexes of heterogeneity were also computed for the *n*-octane and isopropanol probes. The results showed that the polar probe, isopropanol, interacts more strongly with the rock surface than the apolar probe. In the second part of this study, the influence of a surfactant used in drilling mud on the heterogeneity of the surface energy was

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investigated. Different surfactant impregnation ratios were examined by means of IGC-FC. The results indicated that the progressive covering of the solid surface by the surfactant hid chemical functions likely to interact with the injected probes and made the surface smooth by covering the roughness of the surface, which led to the decrease in all parameters obtained by IGC-CF.

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1. Introduction

Oil recovery is strongly dependent on the oil wettability of an oil-field reservoir. Wettability describes the tendency of fluid to adhere to a solid surface in the presence of another immiscible fluid [1]. For reservoir rock-fluid systems, it is the measure of the affinity that the rock has for either oil or water. Cuiec [2] proved the existence of water-wet, mixed-wet or oil-wet rocks. The wettability of the reservoir rock may change due to the interactions of drilling mud with reservoir rocks. A change in wettability from water-wet to oil-wet conditions is accompanied by a decrease in the relative permeability to oil and a corresponding increase in the water/oil production ratio [3–5].

The wettability properties of reservoir rocks are related to their surface energies and surface heterogeneities. Thus, an accurate description of the surface chemistry of the reservoir rock is essential to improve understanding of the factors affecting reservoir wettability, and thus accurately predict reservoir performance.

Previous studies have mostly been conducted by means of two commonly-used methods to determine the surface energy of a solid: wetting (contact angle) measurements by means of liquid probes, and vapor adsorption measurements (IGC) using vapor probes. Contact angle measurement is one of the most widely-used methods for determining the surface energy of solids. However, the results obtained with this method provide only a macroscopic average of the surface energy of the studied solid and the measurements are difficult to implement on powders due to their surface roughness, porosity and irregular shapes. The limitations of the various methods for measuring contact angles for powders are discussed in a review by Buckton [6].

Vapor adsorption measurements using IGC-ID are not affected by these surface roughness problems and they provide microscopic variations of the surface properties. This method makes it possible to study the interactions of a probe molecule with the surface adsorption sites. Recently, Arsalan and Palayangoda [7,8], successfully quantified the fundamental interactions of the reservoir rock-fluid system by characterizing the surface energy of sandstone and carbonate rocks using IGC-ID.

Despite its huge potential, IGC-ID has its drawbacks when used on heterogeneous surfaces, where only high-energy sites are considered under these conditions. In other words, IGC-ID delivers absolute thermodynamic parameters only on a perfectly homogeneous surface, i.e. a surface for which the interaction potential remains constant from one adsorption site to another. No real solid can be considered as truly homogeneous and the presence of sites having different interaction potentials will influence the behavior of the injected probe, and thus the properties measured by IGC-ID [9].

It is therefore of great interest to have a method to estimate the degree of heterogeneity of the solid surface. For this reason, IGC-FC was performed in this work after a standard study of the rocks by IGC-ID.

When the concentration of the injected probes is increased, a growing number of less active sites become involved in the interaction with the probe molecules. In this case IGC-FC allows the determination of desorption isotherms and is a suitable

method for evaluating the heterogeneity of the surface energy of powders.

To the best of our knowledge, no practical experimental study has been carried out to characterize the surface heterogeneity of natural reservoir rocks at the molecular level. The present paper is an attempt to contribute to this goal.

2. Theory of inverse gas chromatography

IGC is a well-suited method for exploring the chemical surface properties of non-volatile materials at the molecular scale. It is receiving more attention as it offers several advantages, such as ease and rapidity of measurement, the possibility of using a variety of probes and a large range of temperatures.

The outcome of an IGC measurement is the retention time of the injected probe t_r . This value reflects the intensity of interactions between the solid surface and the probe.

The retention volume, namely the volume of the carrier gas required to sweep out an injected probe from the column is related to the retention time by Eq. (1):

$$V_n = (t_r - t_0) \times D_c \quad (1)$$

where t_r is the retention time of the probe, t_0 the retention time measured with a non-retained probe (methane) and D_c is the corrected flow rate, which is related to D_m , the flow rate measured with the flow meter, according to Eq. (2):

$$D_c = D_m \times j \times \left(\frac{T_c}{273, 15} \right) \quad (2)$$

where T_c is the oven temperature, and j the James Martin coefficient for taking into account the compressibility of the carrier gas under the effect of the pressure drop ΔP , as shown in Eq. (3):

$$j = \frac{3 \left(1 + \frac{\Delta P}{P_{atm}} \right)^2 - 1}{2 \left(1 + \frac{\Delta P}{P_{atm}} \right)^3 - 1} \quad (3)$$

Depending on the amount of probe molecules injected into the column, two IGC techniques may be distinguished, at infinite dilution (IGC-ID) and at finite concentration (IGC-FC).

2.1. Inverse gas chromatography at infinite dilution (IGC-ID)

At infinite dilution conditions, a very small quantity of probe molecules (vapor) are injected in the column at the limit of detector sensitivity, in such a way that the interaction between probe molecules will be negligible and only the solid surface-probe molecule interactions are favored. Under these conditions, Henry's law can be applied and the retention volume is directly related to the free energy of adsorption [10] according to Eq. (4):

$$-\Delta G_0^a = RTL \ln(V_n) + K \quad (4)$$

where R is the ideal gas constant, T the temperature, V_n the net retention volume and K a constant that depends on the choice of a reference state of the adsorbed probe and also on the total area of the solid accessible to the probe.

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