



Quantifying mineral surface energy by scanning force microscopy



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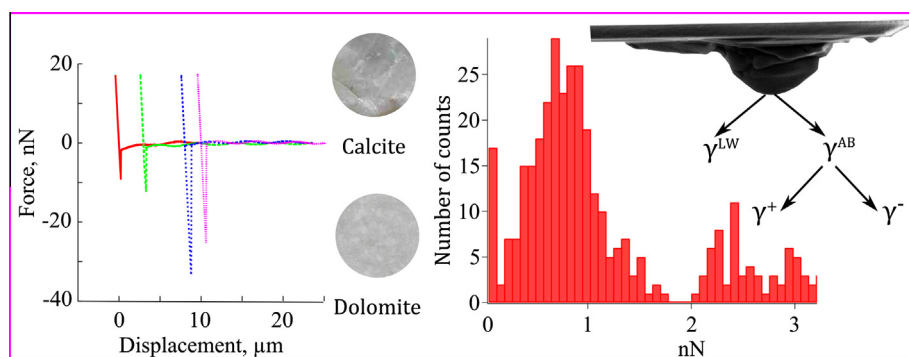
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GRAPHICAL ABSTRACT



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ABSTRACT

Fundamental understanding of the wettability of carbonate formations can potentially be applied to the development of oil recovery strategies in a complex carbonate reservoir. In the present study, surface energies of representative carbonate samples were evaluated by direct quantitative force measurements, using scanning force microscopy (SFM) at sub-micron scale, to develop a reliable method to predict reservoir wettability. Local adhesion force measurements were conducted on appropriate calcite and dolomite samples and performed in air as well as in the presence of polar and nonpolar fluids. This study demonstrated that, by comparing measurements of adhesion forces between samples of the same mineral in different fluids, it is feasible to determine the surface energy of a given mineral as well as its polar and nonpolar components. The derived values are in agreement with literature. A proof-of-principle protocol has been established to quantify surface energy using SFM-based adhesion measurements. This novel methodology complements the conventional contact angle measurement technique, where surface energy can only be examined at large length scale. The reported methodology has great potential for further optimization into a new standard method for fast and accurate surface energy determination, and hence provides a new tool for reservoir rock wettability characterization.

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1. Introduction

Understanding characteristics of reservoir rocks, reservoir fluids and the interaction between them is the basis of reservoir characterization. For example, at the primary production design stage, it is crucial to understand the relative permeability and capillary pressure to determine the fluid flow patterns that will be implemented to control oil production. When planning a secondary or tertiary production strategy, it is critical to re-evaluate the same parameters. As such, fluids interfacial tension and reservoir rock wettability are two critical parameters for any reservoir characterization to be used for production design which includes estimating reserves and recovery factor.

Wettability of reservoir rocks has a strong influence on the amount of recoverable oil because a water-wet mineral surface is beneficial when oil is recovered by water flooding [1]. In this case, water imbibition into pores is supported by the positive capillary pressure, which improves the oil recovery. It is widely accepted that carbonate rock formations are moderately to strongly oil-wet [2,3]. This is attributed to the adsorption of polar compounds contained in crude oil such as carboxylic acids and asphaltenes, onto the carbonate surface, which results in the rock surface changing from hydrophilic to hydrophobic [2,4]. Consequently, oil relative permeability is lower and displacement efficiency is poor. To achieve better oil recovery rates, it is highly desirable to: (i) acquire a fundamental understanding of the actual carbonate reservoir wettability, (ii) evaluate the role of reservoir fluids on surface wettability, and (iii) predict reservoir wettability using surface energy measurements.

Several techniques are available for reservoir wettability measurements; these include imbibition and centrifuge capillary pressure, which are done on reservoir core plugs [5]. Industry standard techniques, e.g. the Amott-Harvey imbibition test and the U.S. Bureau of Mines method, suffer from sample alteration and fluid loss during the coring operation and transport to the laboratory and therefore do not necessarily provide an accurate reservoir wettability. Another technique to determine wettability is the contact angle measurement, which is based on monitoring the contact angle between (at least) three different liquids (with known surface tension components) and the sample surface. This approach requires a smooth, flat and nonporous rock surface on which a droplet of test liquid is placed. However, all reservoir rocks are porous, so contact angle measurements can only provide qualitative results. Therefore, currently there is no suitable and reliable technique for measuring a quantitatively accurate wettability.

Despite the extensive theoretical and experimental work performed in the past few decades, surface energy of solids is still a matter of ongoing debate. The approach proposed by van Oss and coworkers [6–8], known as the van-Oss-Chaudhury-Good theory (vOCG), has been used widely in the oil industry. According to this theory, surface interactions are split into two components: Lifshitz-van der Waals (LW) forces (that include London dispersion forces, Debye induction, and Keesom orientation forces where dispersion forces are the main component) and another group of forces that originate from electron donor–acceptor (Lewis acid–base, AB) interactions. By resolving the equations developed from the van Oss model, the surface energy of a solid surface can be calculated as well as its polar and nonpolar components. A great advantage of this approach is accessing information about the acid–base properties of a given surface by measuring its contact angle with various test liquids. Although this method being widely applied as documented in the literature, numerous drawbacks of this approach have been reported [9–12]: (i) there is a lack of a broad spectrum of test liquids that are well characterized; although there are adequate choices for basic liquids, there are only limited choices for predom-

inantly acidic liquids; (ii) inconsistent surface energy components are obtained when using different sets of liquid tests; (iii) surface energy measured by contact angle measurements depends strongly on surface and environmental conditions.

In a previous study [13] the vOCG approach was used to interpret the surface energy components of calcite and dolomite based on contact angle measurements. The results showed that the surface energy components are dependent on the selected polar probe liquid and the surface characteristics, e.g. roughness and cleanliness. The dispersive surface energy component can be calculated with a high confidence level whereas the polar component calculation was less consistent. Using a probe fluid triplet of 1-bromonaphthalene/water/formamide (B/W/F), the total surface energy of calcite and dolomite were estimated as 58.2 ± 1.5 mN/m and 58.1 ± 0.8 mN/m respectively. At the same time, according to measurements performed using a 1-bromonaphthalene/water/ethylene glycol (B/W/E) fluid triplet, dolomite has slightly higher total surface energy, 59.4 ± 1.4 mN/m, compared to the previous system, whereas that of calcite decreased to 44.2 ± 0.2 mN/m. These results, summarized in Table 1, are within the range of published values [14–16].

One of the major benefits in measuring solid surface energy directly is that it provides the true surface tension in the presence of liquid and facilitates accurate prediction of rock wettability. However, since inconsistent values of surface energy components were reported in the literature, it is crucial to examine whether the roots of inconsistency are related to the fluids used, the surface conditions or the technique being used.

When exposed to organic solvents, surface dissolution is no longer an issue for calcite; however, the volatility and viscosity of the solvents pose additional technical challenges to contact angle experiments. An alternative approach is using scanning force microscopy (SFM), a direct quantitative measurement, to determine surface energy. By quantifying the force needed to detach two surfaces in contact, the surface energy can be calculated based on the contact geometry.

The major objective of this work is to develop a new methodology, based on SFM, to quantify the total surface energy and the respective contributing polar and nonpolar components for typical reservoir minerals. The adhesion forces measured by SFM can be used to calculate surface energy components. The advantages of SFM over the conventional methods, are that it not only provides surface topography with a molecular scale resolution, but also measures adhesion forces acting at the tip–sample interface. Various operational principles are described and reviewed extensively in the literature [17–20]. SFM adhesion force measurements in the oil industry are limited due to the challenges posed by variations caused by heterogeneity of the sample and characteristics of the tip [21]. It was also reported that surface roughness could result in uncertainties in determining surface adhesion force [22,23].

Table 1

Calculated surface energy by components based on contact angle measurements for calcite and dolomite using the van Oss method [9,10] and compared to reported data in the literature. All values are in mN/m.

Mineral	γ_s^+	γ_s^-	γ_s^{AB}	γ_s^{LW}	γ_s^{TOT}	Ref.
Calcite	3.2	39.3	22.3	35.8	58.2	B/W/F [13]
	0.6	27.9	8.3	35.8	44.2	B/W/E [13]
	0.8	58.8	14.1	42.2	56.3	[14]
	0.1	29.0	2.8	43.8	46.7	[15]
	1.3	54.4	16.8	40.2	57.0	[16]
Dolomite	1.3	57.9	17.2	40.9	58.1	B/W/F [13]
	1.4	59.0	18.4	40.9	59.4	B/W/E [13]
	1.9	33.3	15.9	43.5	59.4	[14]
	0.2	30.5	4.9	37.6	42.5	[16]

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