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Microwave treatment of crude oil emulsions: Effects of water content



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ABSTRACT

The main objective of this work was to investigate the effect of water content in crude oil emulsions upon the microwave demulsification process. The microwave heating properties were examined for samples of neat water, neat oil and W/O emulsions with volume fractions of water ϕ ranging from 0.04 to 0.77. The observed changes in initial heating rates correlated with structural transformations in the disperse phase of as-prepared emulsions. In course of heating emulsions with $\phi < 0.40$, a characteristic succession of endothermic and exothermic processes was observed, the latter process triggering the phase separation of free water. The optimal conditions for microwave irradiation were determined by calculating the amount of microwave energy required for the onset of phase separation. In the studied emulsions, minimum energy consumption was observed at volume fractions of water $\phi \approx 0.18$ and $\phi \approx 0.56$.

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1. Introduction

A problem in mature oil fields is the sizeable amount of water accompanying the produced crude oil. Water is also injected into the crude in course of steam treatment of producing wells or during de-salting operations. Petroleum emulsions readily form from water/oil mixtures in turbulent flows or due to pressure gradients in reservoir pores, in the chokes at the wellheads and in various valves in piping used for oil production. These emulsions can increase pumping and transportation costs, facilitate the corrosion of producing and processing equipment, and the poisoning of refinery catalysts (Sjöblom, 1996, 2001).

The native petroleum emulsions typically are water-in-oil (W/O) ones (Berridge et al., 1968) as oils contain “indigenous surfactants” such as asphaltenes, resins, naphthenic acids and oil-wet fine solids (Sjöblom et al., 2003) while, according to Bancroft’s rule (Bancroft, 1913), the liquid in which the surfactant is soluble becomes the continuous phase. These surfactants migrate to the oil–water interface and can stabilize water droplets by steric and electrostatic interactions, or by rigid-film forming (Sjöblom, 1996, 2001).

The traditional methods of eliminating petroleum emulsions (demulsification) include intensive utilization of heat and chemical compounds. Usually these methods are expensive, chemical additives may pollute the wastewater streams, or follow the hydrocarbon into the refining process. An alternative and cost-effective technology is microwave demulsification, first patented ca. three decades ago (Wolf, 1986). Microwave energy activates the aqueous, indigenous surfactant

and interfacial films components selectively, providing differential energy input. Microwave demulsification has no secondary pollution problems, because no chemical additives are required. Also, microwaves have a high penetration power; therefore faster separation rate and less energy consumption than in conventional heating are possible (Fang et al., 1988; Chan and Chen, 2002; Hong et al., 2004; Xia et al., 2004).

A good knowledge of native W/O emulsions is necessary for controlling and improving demulsification processes at all stages. Many studies have been carried out in the last decades; however, there are still many unsolved questions related to the peculiar complex behavior of these emulsions (Kilpatrick, 2012). The complexity comes mostly from the continuous oil composition, in particular from the “indigenous surfactants” contained in the crude (Sjöblom et al., 2003; Kilpatrick, 2012). Another complicating factor is an existence of multiple structural states of the disperse water phase (Evdokimov et al., 2003, 2005, 2008; Evdokimov and Losev, 2011).

This paper gives new information with respect to the characteristics of water-in-native crude oil emulsions being subjected to microwave treatment and can be used to obtain insight into underlying mechanisms of de-emulsification. Some preliminary results of the below described experiments have been reported in a Russian journal (Evdokimov and Novikov, 2007).

2. Experimental

2.1. Materials and sample preparation

The virgin crude oil was collected from the well #624 at Korobkovskoye reservoir (Volgograd, Russia), and had a density

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of 832 g/L, a specific heat of 1.9 J/g K, a pour point below -18°C , and contained ca. 1 wt% asphaltenes, 8 wt% resins, and 2 wt% waxes. Oil samples were stored in the dark, in air. Water was a double distillate stored in air, with $\text{pH} \approx 5.5$.

All preparation procedures were conducted in a thermally controlled closet at 20°C . Water and oil were mixed at various water cuts (volume fractions of water) ϕ ranging from 0 to 1, homogenized using an axial flat blade impeller (~ 2000 rpm) for 8–10 min and allowed to equilibrate for 2–3 min. A type of emulsion was inferred by a “drop test” (Ashby and Binks, 2000). All our emulsions, with water contents up to 80% w/w, appeared to be oil-continuous (W/O) as they dispersed in oil and remained as drops in water. Finally, a sample of emulsion (70 ± 0.5 g) was transferred to a 100 mL glass beaker, employed in microwave experiments.

2.2. Experimental equipment and procedure

In this study, a domestic microwave oven Elenberg MS-1700M was used. At an operation frequency of 2.45 GHz its rated microwave power outlet is 700 W (in a continuous, 100% mode). Treatment of samples at full power resulted in a temperature increase with a rate too high for reliable identification of the pre-demulsification behavior of emulsions. Hence, in all further experiments the oven was operated at minimal power (in a pulsed, 17% mode).

A plastic sample holder was fixed at the center of the oven's cavity. In each experiment, a beaker with emulsion (at $20 \pm 0.1^{\circ}\text{C}$) was placed into the holder and subjected to microwave irradiation for a required time interval, determined with an accuracy of ± 0.5 s. For temperature measurement after microwave treatment (within 2–3 s), a calibrated thermocouple thermometer ($\pm 0.1^{\circ}\text{C}$) was inserted at the axis of a sample, 15 mm below the emulsion's surface.

3. Results and discussion

3.1. Temperature increase vs. irradiation time

In homogeneous samples of oil and water heating by microwaves occurs via conduction and dipolar relaxation losses (Fang et al., 1988; Feldman et al., 1997), described by the imaginary part ϵ'' of the complex dielectric permittivity:

$$\epsilon^* = \epsilon' - i\epsilon'' = \epsilon' - i(\epsilon''_{\text{cond}} + \epsilon''_{\text{dipol}})$$

Conduction losses quickly decrease with microwave frequency and in the GHz range usually become negligibly small compared to losses via dipolar processes (Whittaker and Mingos, 1994).

In the studied temperature range (20 – 45°C) external heat transfer from treated samples (both convective and radiative) may be regarded as negligible (Fang and Lai, 1995). Hence, at a constant specific heat power w generated by microwaves in a homogeneous liquid, a temperature increase ΔT during the time interval Δt :

$$\Delta T = \frac{w}{\rho C_p} \Delta t,$$

where ρ and C_p are the density and the specific heat capacity of a treated liquid, respectively, which may be temperature-dependent. However, for water heated from 20 to 45°C the product ρC_p remains virtually constant, changing merely by 0.8% (Lide, 1999), and the dependence $T(t)$ may be expected to be close to linear, as confirmed by experimental data in Fig. 1.

For clarity of presentation, only some of the experimental points are shown in the figure and characteristic data subsets

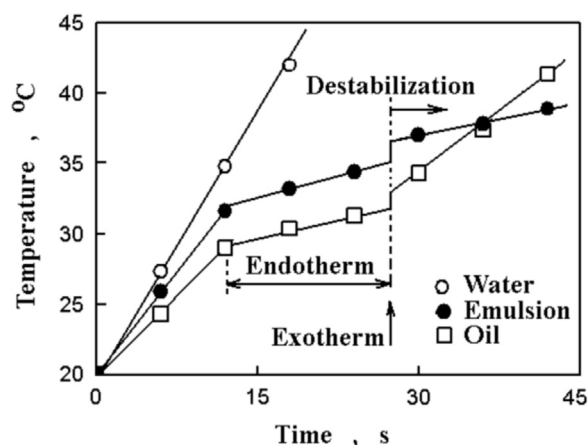


Fig. 1. Temperature increase in samples of water, oil and W/O emulsion vs. time of microwave irradiation.

are connected by straight lines. Open circles and squares, respectively, denote the data for 100% water and 100% crude oil samples. Filled symbols show a temperature increase in an emulsion with water cut $\phi = 0.263$. A linear approximation of the total $T(t)$ dependence for water provides the value of specific heat power generated by microwaves $w = 5.8 \text{ W/cm}^3$. In oil samples an initial linear $T(t)$ behavior (with $w = 1.2 \text{ W/cm}^3$) persisted only at temperatures below ca. 27°C (irradiation times < 12 s). The following slowing down of $T(t)$ dependence at 12 – 28 s (27 – 33°C) and an abrupt rise at ~ 27 – 28 s (33 – 34°C) are indicative of some endothermic and exothermic processes (structural modifications in the oil's molecular system), which are discussed in more detail below. Note that other microwave studies also revealed virtually linear $T(t)$ dependence in water, as well as endothermic/exothermic $T(t)$ features in native crude oils (Hong et al., 2004).

Finally, in the emulsion sample with $\phi = 0.263$ an initial linear $T(t)$ behavior again is followed by endothermic and exothermic features, characteristic for the original crude oil. A new feature in emulsion sample is a slower $T(t)$ increase after the exothermic rise which is due to the onset of emulsion destabilization, visually detected by an appearance of a layer of free water.

For the following analysis it is important that in all emulsion samples (with $0.042 \leq \phi \leq 0.769$) the initial $T(t)$ dependencies (at $t < 3$ – 5 s, $T < 23$ – 24°C) were linear with constant heating rates dT/dt , which may be regarded as representative of microwave interaction mechanisms with specific colloidal structures in non-disturbed, as-prepared emulsions. On the other hand, variations of dT/dt at higher temperatures evidently are indicative of structural modifications in emulsions, which terminate with the phase separation of free water.

3.2. Initial heating rate vs. water cut

Fig. 2 shows the effect of water cut ϕ on the initial ($t < 5$ – 6 s) heating rates dT/dt of emulsions subjected to microwave irradiation. The observed non-monotonic dependence on water content previously has never been reported for microwave-treated emulsions (cf. Wolf, 1986; Fang et al., 1988; Chan and Chen, 2002; Hong et al., 2004; Xia et al., 2004 and references therein). On the other hand, our recent rheological experiments have revealed non-monotonic effects of water cut in non-microwaved, as-prepared water emulsions in native crude oils (Evdokimov et al., 2008; Evdokimov and Losev, 2011). These effects were attributed to transformations of emulsions' morphology at some threshold values of ϕ , indicated in Fig. 2 by vertical dashed lines and denoted by numbers 1–4. Some specific structural states of emulsions are schematically illustrated in Fig. 3.

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