Dehydration efficiency of AC electrical fields on water-in-model-oil emulsions

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A B S T R A C T

The effect of several factors has been studied in order to determine their influence on the electrostatic dehydration process of water-in-model-oil emulsions. Not only application time, waveform, strength and frequency of the applied electric field, but also temperature were all found to play a non-negligible role in the process. The results obtained have been correlated to observed phase separation measured with a Turbiscan Lab instrument.

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1. Introduction

The components present in crude oils such as asphaltenes, inorganic particles or natural surfactants stabilize the water-in-oil emulsions [1]. Separation of these emulsions is crucial for the petroleum industry since they are troublesome both from a process and product quality viewpoint. Several techniques [2,3] are used in order to achieve this process such as chemical demulsification [4,5], pH adjustment [3], gravity or centrifugal settling [6,7], filtration [3,7], heating treatment [3], but one of the most effective and utilized method in the industry today is electrostatic demulsification [8–10]. The combination of high energy-efficiency, since it permits a reduction of the use of heat, and also the fact that it avoids the use of chemical demulsifiers makes this technique environmentally friendly.

The utilization of electrical methods for dehydrating crude oil emulsions is not new and has been reviewed elsewhere [11–13]. Basically electrostatic separation, or electrocoalescence, consists of the application of an electric field which assists the merging of small water droplets into larger ones that will settle more quickly in a separation tank [14,15]. When a water-in-oil emulsion is exposed to an electric field, two main phenomena are observed: flocculation and coalescence of the droplets [16]. Among other studies, Less et al. [9,10] have studied these two mechanisms by means of bulk rheology. The formation of water droplet chains (flocculation) increases the resistance of the emulsion to flow and therefore its viscosity, while an increase of droplet size (coalescence) leads to a viscosity decrease.

In the absence of an electric field, water droplets are randomly distributed according to the law of Brownian motion [17] and Stokes’ law of sedimentation. As the field is applied and increased, the droplets align themselves with the field as an effect of polarization, leading to linear flocculation with water droplets attaining a linear chain-like configuration between the electrodes [2]. In low electric fields, the aqueous droplets return to a random distribution when the electric field is switched off. The critical electric field Ecrit is the value which causes irreversible breakdown of the film and subsequent coalescence of droplets [2,9,18].

Herein, we have measured the destabilization of emulsions under the influence of an AC electric field. Electrorheology, therefore viscosity measurements, and observed phase separation by using the Turbiscan Lab instrument [19], were employed and several parameters, such as frequency, duration of application or temperature were modified independently to determine their direct influence on the dehydration of the emulsions process.
2. Experimental

2.1. Preparation of emulsions

Water-in-oil emulsions were prepared using, for the oil phase, 70 vol.% of a transformer synthetic model oil, Nytro 10-XN (kindly provided by Sintef Energy Research), and for the water phase, 30 vol.% of synthetic brine consisting of 3.5 wt.% NaCl in Milli-Q water (>18.2 MΩ cm). In order to stabilize the emulsions, 0.05 wt.% of Span 80 (provided by Fluka) was added to the mixtures. Prior to the emulsification process, the samples were heated at 60 °C for 15 min. Emulsification was carried out with an Ultra Turrax (IKA, T18 with 18 and 12.7 mm of stator and rotor diameters, respectively) at 16,000 and 21,000 rpm for 60 and 90 s, respectively. The emulsions were then immediately poured into the rheometer cup for further testing.

The droplet size distribution of the water-in-model-oil emulsions was determined using a Malvern Zetasizer Nano ZS and was measured to be centered at 200 ± 20 nm.

2.2. Instrumentation

The rheological properties of the emulsions were measured on a Physica MCR-301 rotational rheometer in controlled shear mode, using the RheoPlus software. This commercial rheometer, equipped with an electrorheological temperature device (ERD, PTD200/E), was modified for allowing the connection with an AC source. The electrical part of the equipment consisted of a high voltage amplifier (Trek, 609E-6), a function generator (Wavetek, 187–4 MHz) and a digital oscilloscope (Yokogawa, DL1540–150 MHz). The radii of the measuring bob and cup are 13.32 and 14.46 mm, respectively and the electrode displacement is shown in Fig. 2. The cup is grounded. The rotating bob is also the high potential electrode, since a spring directly connected to the generator slides on it. Thus, at low shear rates, the quality of the measurements may be affected by interference effects even if the mean values of the rheological variables are clearly identifiable. At higher shear rates this problem does not appear. The original measuring bob had sharp edges which were proven to lead to electric field enhancements in their proximity. The effects in these areas interfered with the effects in the long parallel gap where the electric field was more uniform. To avoid this inconvenience the lower and upper edges were rounded. By doing so, the absolute viscosity values proved to be slightly lower than the actual ones. Nevertheless the viscosity variations are correct and the electric field lines more evenly distributed, so that the results can be considered more reliable [20]. In order to allow for an effective electric field application, the rheometer AC cup was insulated with PVC tape (3 M, Super 33+) 0.178 mm thick and designed to perform up to 105 °C.

Herein, the viscosities of all emulsions were first monitored at the targeted temperature under the influence of the shear only, for detecting any instability. Afterwards a voltage was applied for 5–20 s. The experiments were performed at different temperatures (20, 40 and 60 °C), electric field strength (2, 3.5, 5 and 10 kV/cm) electric field waveform (sinusoidal, triangular and square) frequencies (50, 100, 500, 1000, 5000 and 50,000 Hz) and time of application of the electrical field (5, 10 and 20 s).

The observed phase separation was monitored with a Turbiscan Lab [19]. The Turbiscan measures two different parameters: backscattering and transmission which depend both on size and concentration of droplets in the samples. In this study, we will only talk about the backscattering since all the transmission values recorded were null. By measuring the backscattering profile as a function of the height of the sample, at different times, we can detect particle size change (coalescence, flocculation) and phase separation (sedimentation, creaming) [19].

The measurement consists of a reading head moving along a cylindrical cell, while scanning the entire sample height. The reading head itself consists of a pulsed near infrared light source and 2 synchronous detectors. The instrument allows for simultaneous detection of transmitted and backscattered light (135°) from the sample. The reading head acquires backscattering data every 40 µm. All the experiments were performed at 20 °C. The acquisition is then repeated with a programmable frequency to obtain a superimposed profile characterising the relative stability of the sample.

3. Results and discussion

3.1. Electrorheology

In order to ascertain that the shear rate does not influence the destabilization process, time-dependent viscosity measurements...
were performed on the emulsions at various temperatures (20, 40 and 60 °C) in the absence of an applied electric field. For all the temperatures studied here, no variation of viscosity was observed (results not shown), for time frames exceeding the usual experimental time (3 min), confirming the noneffect of the chosen shear rate on the emulsion stability.

3.1.1. Influence of the duration of the applied electric field

The influence of the duration of the applied electric field on the destabilization of the emulsions has been studied and the results are shown in Fig. 3. Viscosity of the emulsions as a function of time at 20, 40 and 60 °C with sinusoidal, triangular and square electric waveforms were measured. The electric field was applied for 5, 10 and 20 s. It is obvious from observation of these figures that the degree of destabilization is proportional to the duration of the applied electric field. For instance, for a sinusoidal waveform, at 20 °C, the viscosity reductions observed, when the electric field is applied for 5, 10 and 20 s, are 1.57, 3.27 and 5.25 cP, respectively, corresponding to viscosity losses of 12.8, 17.2 and 22.4%. This trend is consistent for all the systems studied here irrespective of the waveform or the temperature. All the numerical results are summarized in Table 1.

3.1.2. Influence of the waveform signal

The influence of the waveform signal on the destabilization of the emulsions is shown in Fig. 4. Viscosity of the emulsions as a function of time at 20, 40 and 60 °C with electric fields applied for 51, 101 and 20 s was measured for sinusoidal, triangular and square waveform signals. Whatever the duration of the electric field applied or the temperature chosen, it is obvious from Fig. 4 that the choice of the electric waveform signal is clearly an important factor regarding the destabilization process using AC electric fields. The efficiency of the waveforms in destabilizing the emulsions can be ranked as (in decreasing order of efficiency) square–sinusoidal–triangular. A possible explanation would be that in term of pure geometry (if one takes into consideration the area of the form), a square voltage is 1.27 and 2 times more efficient than a sinusoidal or triangular one, respectively. Even if these ratios are not strictly observed in terms of viscosity reduction, this trend is nevertheless always observed. For instance, at 60 °C, when an electric field is applied for 10 s, the viscosity reductions observed, when the waveform of the applied electric field is square, sinusoidal and triangular, are 4.32, 3.88 and 3.06 cP, respectively, corresponding to viscosity losses of 19.9, 17.2 and 14.7%. All the numerical results are summarized in Table 1.

A summary of the effects of duration and waveform is shown in Fig. 5. For all the temperatures studied here, 20 °C (A), 40 °C (B) or 60 °C (C), the effect on the destabilization of the emulsions of the duration of the applied electric field is clearly shown; the longer the duration, the better the separation. From this figure, the influence of the waveform signal chosen when the voltage is applied can be seen. As previously stated, a ranking of the waveforms could be imagined where the square form would be the most efficient followed by the sinusoidal form and then by the triangular.

3.1.3. Influence of the temperature

The crucial influence of temperature on the destabilization process is depicted in Table 1. For all the systems studied, the viscosity reduction, expressed in percent, always increases with increasing temperature. For instance, when an electric field is applied for 5 s, using a triangular waveform, the viscosity reductions observed at 20, 40 and 60 °C are 1.32, 2.54 and 2.46 cP respectively, corresponding to a viscosity reduction of 2.3, 7.6 and 11.0%. These results were expected since an increase of the temperature generally favors film destabilization [21].
3.1.4. Influence of the strength of the applied electric field

All the previous experiments were performed by applying a constant electric field strength of 3.5 kV/cm. The effect of the strength of the applied electric field was also studied, and the results are shown in Fig. 6 and Table 2. Here, all the experiments were run at 40 °C with an electric field applied for 20 s. The strengths of the electric fields applied were 2, 3.5, 5 and 10 kV/cm. From the results, it is obvious that the strength of the applied electric field is also crucial in the efficiency of the demulsification process. Indeed, for all the waveforms studied (the square waveform results are shown in Fig. 6, for the sinusoidal and triangular waveforms, the results are not shown1), only reversible flocculation was observed with an applied electric field of 2 kV/cm. The applied field is not strong enough to enhance coalescence of the water droplets. For higher electric fields (3.5, 5 or 10 kV/cm) flocculation, immediately followed by coalescence, was observed. Once again, the importance of the strength of the electric field is proven since the magnitude of the viscosity reduction is proportional to the field strength. These results are summarized in Fig. 7. At a given temperature and for a given duration of the applied electric field, the effect of the strength of the applied electric field, on the destabilization of the emulsions, is clearly shown. The separation is better if a strong field is applied. From this figure, the influence of the waveform signal chosen when the voltage is applied is easily observed. Again, a ranking of the waveforms could be imagined where the square form would be the most efficient followed by the sinusoidal form and the triangular.

3.1.5. Influence of the frequency

All the previous experiments were performed by applying a constant frequency of 50 Hz. The frequency of the applied electric field was also studied, and the results are shown in Fig. 8 and presented in Table 3. Here, all the experiments were run at 40 °C.
Influence of the frequency. Viscosity as a function of time at 40 °C for emulsions subjected to sinusoidal electric waveforms. Influence of the frequency.

All the curves obtained show an increase of the viscosity corresponding to coalescence [16]. The dehydration efficiency of the emulsions increases with the frequency. For instance, the viscosity reductions observed vary between 5.19 and 15.88 cP, with an electric field applied for 20 s. The frequencies of the electric fields applied were 100, 500, 1000, and 5000 Hz, respectively. For higher frequencies, the viscosity losses of 14.8–51.1% for an applied frequency varying between 50 and 5000 Hz, corresponded to flocculation, immediately followed by a decrease of the electric field for a given temperature (40 °C) and a given waveform signal (sinusoidal).

Table 3
<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>Viscosity reduction (cP—%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>5.2–14.8</td>
</tr>
<tr>
<td>100</td>
<td>6.1–19.7</td>
</tr>
<tr>
<td>500</td>
<td>8.1–26.3</td>
</tr>
<tr>
<td>1,000</td>
<td>9.0–29.5</td>
</tr>
<tr>
<td>5,000</td>
<td>15.9–51.1</td>
</tr>
<tr>
<td>50,000</td>
<td>0</td>
</tr>
</tbody>
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(50,000 Hz), only flocculation is observed (curve not shown); the droplets do not coalesce, suggesting that there is an upper limit for the applied frequency in terms of emulsion destabilization.

3.2. Observed phase separation: utilization of the Turbiscan

The separation process can be observed using the Turbiscan. The profile obtained is characteristic of the sample homogeneity and droplet concentration. It is represented by a curve showing the percentage of backscattering light as a function of the sample height (in mm). The acquisition along the sample is then repeated with a programmable frequency (multi-scans) to obtain a superimposition of product fingerprints characterising the stability or instability of the sample, represented by the time evolution of profiles.

Fig. 9A–C shows three typical Turbiscan spectra of backscattering (one scan every 7 min for 24 h). The data are presented as a function of time (0:00–24 h) and sample height (0–50 mm). In these experiments, only the backscattering profiles were used to study the separation process since even after 24 h, the solutions were still opaque. No free water or free oil could be observed.

Three systems, considered by the authors as representative, were studied in order to elucidate the effect of the waveform and the strength of the electric field influences on the separation process. First, the backscattering profile of an emulsion that was not submitted to any electric field was measured (Fig. 9A). From this figure, one can observe that there is no much difference between the first and the last scan, indicating a stable emulsion. Then, the backscattering profiles, first, of an emulsion which was submitted to an electric field of 3.5 kV/cm with a sinusoidal waveform (Fig. 9B) and then of an emulsion which was submitted to an electric field of 10 kV/cm with a square waveform (Fig. 9C), which was proved to be the most efficient system by electrorheology, were measured. The comparison of the three obtained profiles shows that there is a significant difference between the systems. At around 40 mm, the sedimentation front changes faster in the case of the emulsion that was submitted to the higher electric field strength, revealing a faster sedimentation, thus the presence of bigger droplets. Moreover, lower in the sample (<40 mm), the delta backscattering increases revealing either an increase of the droplet size or of the droplets concentration, probably both. Once more it is possible to confirm the ranking of the separation process efficiency determined by electrorheology, where an electric field of 10 kV/cm with a square waveform is more efficient than an electric field of 3.5 kV/cm with a sinusoidal waveform, itself more efficient than no electric field at all.

Fig. 10 shows backscattering as a function of time for the three emulsions described above. For all samples, the kinetic profiles were calculated, from the total height of the sample normalized with respect to the reference scan, i.e., the difference in backscattered light between the reference and the sample at a given time. Fig. 10 shows that more light was backscattered for the sample that has been subjected to the application of an electric field, revealing the presence of bigger droplets in the sample. The emulsion that has not been subjected to any electric field is the most stable sample.

It is also possible to compare the samples at one given time, via the calculation of the Turbiscan Stability Index (TSI).

![Image](https://via.placeholder.com/150)

Fig. 7. Viscosity reduction at 40 °C as a function of the strength and of the waveform signal of the electric field applied.

![Image](https://via.placeholder.com/150)

Fig. 8. Viscosity as a function of time at 40 °C for emulsions subjected to sinusoidal electric waveforms. Influence of the frequency.
4. Conclusions

The dehydration efficiency of AC electric fields on water-in-model-oil emulsions has been studied using electrorheology and the Turbiscan technology [19]. Destabilization was investigated with respect to the effect of the temperature and characteristics of the applied electric field: duration, field strength, frequency and waveform. Increasing either the temperature, frequency (until an upper limit), duration of the applied electric field or the field strength resulted in significantly increased/improved separation performance. Moreover, it was found that the waveform also greatly affected the destabilization efficiency. The waveforms can be ranked in decreasing order of destabilization efficiency as square, sinusoidal and triangular. In the light of these results, it is then possible to optimize an electrocoalescer in terms of efficiency by just changing one or several of these parameters in order to obtain a satisfying emulsion separation at a lower cost.

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Appendix A. Supplementary data


References


