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Impedance spectroscopy dependent water content detection in dynamic oil-water emulsions

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The water content in static crude emulsions has been extensively studied. However, the dynamic emulsions under shear conditions have a higher stability of the water content, primarily affecting the characteristics of the oil–water two-phase flow in the gathering lines. In this study, a dynamic apparatus was fabricated to investigate the impedance characteristics of water content in oil–water emulsions under the influence of the shear force. The characteristic frequency was proposed to describe the water content in a cubic function. The equivalent circuit model of emulsion has provided an explanation for the variation of the characteristic frequency is mainly caused by the capacitance with the increasing water content. The dynamic conditions have an effect on the electrical properties, which is related to the measurement of water content of the emulsion system. The characteristic function of water content can contribute to a quantitative characterization of the water content of dynamic crude oil, and promote the efficiency and safety of crude oil exploitation. © 2018 Author(s).

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I. INTRODUCTION

In the current oilfield development process, secondary exploitation strategies that rely on increasing the reservoir pressure by injecting water into the reservoir lead to the generation of water content in crude oil. The oil–water mixtures tend to form a stable emulsion under the influence of shear force during transport. The water content of an emulsion is the dominant factor affecting the characteristics of an oil–water two-phase flow. Moreover, the shear conditions maintain the dynamic balance of the rupture and coalescence of the emulsion droplets under relative conditions, leading to more stable water content of the emulsions than in static emulsions. Therefore, the characterization of water content in dynamic emulsions is crucial for the petroleum industry.1–5

Certain measurement techniques have been developed to determine the water content of crude oil, which can be classified into two broad groups: indirect and direct measurements. Indirect measurements use sampling and a subsequent laboratory analysis, whereas direct measurements use an on-line emulsion analyzer. Indirect measurement techniques, such as distillation, titration, and electric dehydration, were more commonly used in the past. However, the accuracy of these techniques is in consistent and is highly dependent on the extent to which the collected sample is representative of the pipeline composition.6–8 Thus, indirect techniques are inapplicable to the present oilfield...
automation management. In recent years, the application of direct techniques has become more extensive owing to their real-time capability and convenience. Direct techniques such as density-based, capacitance, microwave-based, ray-based, ultrasonic, and spectroscopic techniques are used in different situations according to the specific requirements. Density-based techniques are feasible and accurate for measuring the composition of O/W or W/O emulsions. However, density-based techniques function only as long as the densities of oil and water differ. If the densities of oil and water are the same, the emulsion density becomes independent of the composition. The capacitance is widely used because of the low-cost and good maintainability. However, it is generally applied to W/O emulsions with low water content. Ray-based and ultrasonic techniques are based on the different absorption coefficients of the oil and water phases, and are preferred for the sake of precision.\(^6,9\) In addition, some new techniques have also been developed in recent years. For example, an optical non-contact method, called all-optical detection, has been proposed for measuring the water content within the full range of 0–100% in crude oil using a non-contact laser source and receiver.\(^14\) By using THz time-domain spectroscopy (THz-TDS), Song et al. investigated a series of crude oil types with a high water content of 50–100%, and pointed out that the amplitude and absorbance of a THz pulse can be used to characterize the water content in water–oil mixtures.\(^15\) However, most of these techniques cannot reflect the water content of the emulsions in a pipeline in real-time owing to the instability and phase transformation of emulsions under static conditions.

Impedance spectroscopy is a developing technique that enables the fast and nondestructive electrical characterization of petroleum fluids during different stages of exploration and processing. This technique is based on the frequency dispersion characteristics of the impedance parameters of the oil–water emulsions in an AC field.\(^16\) The existence of a natural active substance leads to an interfacial film with thickness and toughness between the oil and water phases.\(^17,18\) The charged particles tend to gather in interfacial oil–water film causing interfacial polarization. Thus, the emulsions exhibit various electrical characteristics at different frequencies, which is called frequency dispersion.\(^19\) Under other fixed conditions (water salinity and temperature), the frequency dispersion characteristics are mainly affected by the disperse phase (water) concentration. Therefore, the frequency dispersion characteristics of the electrical parameters have been gradually applied to the measurement of the water content of oil–water emulsions. In a previous study, Goualused used impedance spectroscopy to analyze the dielectric and conductivity characteristics of Boscan asphaltenes and maltenes within a low-frequency range of 40–10\(^6\) Hz, where the molecular size and aggregation of the asphaltenes can be determined.\(^20\) Tjomsland et al. compared the impedance spectra and infrared spectra of petroleum fractions, calculated the Cole-Cole parameters, and analyzed the multivariate components.\(^21\) N. Perini used electrochemical impedance spectroscopy (EIS) to analyze the crude oil and oil–water emulsions within the frequency range of between 10\(^4\) and 10\(^7\) Hz, and concluded that the impedance spectra of dehydrated crude oil and emulsion have a significant difference. In addition, the time constant of W/O emulsions increases with an increase in the water content, which can be applied to an analysis of the electrostatic demulsification of water-in-oil emulsions.\(^22\) Nevertheless, the application of impedance spectroscopy on dynamic oil–water emulsions remains limited, particularly the characterization of the water content. The testing apparatus developed in the present work allows the impedance characteristics of emulsions to be studied under the shear conditions. This phenomenon was also analytically investigated, and a function proposed for modeling the water content.

In this study, the impedance characteristics of oil–water emulsions with a water content of 10.00–70.00% were experimentally and analytically investigated, and the characteristic frequency was proposed to describe the water content in a cubic function. An equivalent circuit model of emulsions is provided to understand the variation in the impedance parameters caused by the water content. Basing on the model, the variation of the resistance and capacitance with the water content has been studied. The results indicate the increasing water content has greatly promoted the capacitance of the emulsion system. This investigation shows that the impedance spectroscopy can be regarded as an effective method to characterize the water content of two-phase oil–water flows.
II. EXPERIMENTAL METHODS

A. Fabrication of apparatus

A laboratory testing apparatus was fabricated to measure the impedance characteristics of dynamic emulsions.

Our apparatus consists of three parts:

1. A measurement system (Fig. 1). The system is composed of a sample pool and the frequency response analyzer. To avoid the edge effect, a sample pool, which is a cylindrical vessel made of a resin material, was used as shown in Fig. 1. It was designed to be ϕ110 mm × 148 mm × 5 mm (inside dimensions) in size, supporting the thorough stirring of the liquid samples. The sample pool is equipped with double copper electrode plates of 163 mm × 130 mm × 0.5 mm in size on the inner wall, connecting a PSM1700 Frequency Response Analyzer using twisted pair cabling. The analyzer (PSM1700 Frequency Response Analyzer) reports the complex impedance response of oil–water emulsions, mainly realized through a sinusoidal electric potential with a variable frequency.

2. A stirring system. This system is composed of an agitator, a digital tachometer, and a digital torque meter. The agitator (MR-D Agitator, IKA Labortechnik, UK) can provide a stirring rate of 10–1600 rpm, adjusted using a digital tachometer (RE 162 C Digital Tachometer, IKA Labortechnik, UK), and is equipped with a flattened resin blade of 70 mm × 70 mm × 5 mm. There are 6 special overflow holes of ϕ15mm evenly distributed on the blade to reduce the influence of the fluid resistance. The stability of the emulsion is observed based on a variation in torque using a digital torque meter (D1-S1 Digital Torque Meter, IKA Labortechnik, UK). The stirring system provides a stable shear environment to the sample mixtures, aimed at simulating the shear conditions in a pipeline.

3. HAAKE AC200 Water Bath. Surrounding the sample pool, a 45mm thick water jacket is connected to the water bath system to maintain the temperature of the oil–water mixture samples. The system can operate within the temperature range of 5–90°C.

B. Preparation of W/O emulsions

The oil–water mixtures were obtained by mixing different proportions of crude oil to distilled water, with a volume fraction aqueous phase of 10–70%. Dehydrated oil of the Xinjiang Tarim Oilfield was used as the crude oil samples. The physical properties of the crude oil are shown in Table I. The crude oil was placed in a water bath at 80°C for 2 h to eliminate its shear and thermal histories, and then placed at room temperature (25°C) in shade for 48 h. The oil and water were then emulsified using a stirring system at 500rpm for 10min. After the stirring, the torque of the oil–water mixtures was generally maintained at a nearly constant level, and no layering phenomenon was observed. It can be considered that stable oil–water emulsions were formed through the present study.

![FIG. 1. Schematic diagram of the measurement system.](image-url)
TABLE I. Physical properties of crude oil.

| Properties                     | Results |
|--------------------------------|---------|
| 20°C Density (kg/m³)           | 879.5   |
| Solidification point (°C)      | -10     |
| Asphaltene content (%)         | 3.72    |
| Gum content                    | 7.89    |
| Wax content                    | 3.5     |
| 50°C Viscosity (mPa.s)         | 45.78   |

C. Impedance measurement

After preparation, the oil–water emulsions were immediately transferred to the sample pool. The emulsions were stirred at a rate of 500rpm. After being stirred for 5min, the emulsions were measured using the frequency response analyzer under a frequency sweep of 50 to 10⁶ Hz at a constant voltage level of 2V and without DC bias. The impedance parameters of the samples were recorded and transmitted to the computer within a short period of time (<10s). The data were then analyzed using software (Zview, Scribner Associates, Inc., USA) to obtain the impedance spectroscopy of the emulsion. During this process, a constant temperature of 30°C was maintained using a water bath system.

III. RESULTS AND DISCUSSION

Using the measured parameters, the real part $Z'$ and imaginary part $Z''$ of the impedance spectroscopy were obtained. As shown in the Fig. 2, the imaginary part curves of oil-water mixtures with various water contents have corresponding peak points, respectively. It is interesting to notice that the frequency of the peaks changed with the increase of water content.

To further investigate the universality of the impedance characteristic, the same set of experiments were performed under four different rates (200, 300, 400, and 500rpm). The frequency values of peak points were extracted from the frequency-domain spectroscopy of imaginary part. As shown in the Fig. 3(a), the peak frequency shows a slightly decrease trend with the increasing of rate in the emulsion with high water content. But it is noted that the peak frequency decreases with increasing water content under each rate. In previous works, the peak frequency is always discussed in permittivity and conductivity measurements, to describe of the relaxation of frequency of two-phase system. In this work, we believe that the peak frequency is dominantly affected by the water content in the emulsions, which is caused by the interfacial polarization as we discussed above. And we set the peak frequency as the characteristic frequency ($F_s$). Fig. 3(b) exhibits $F_s$ decreased from 331.34 to

![FIG. 2. Frequency dependent values of real and imaginary parts of the emulsions. Lines with various colours represent the different water contents. The inset displays the position of peak values of the imaginary part.](image-url)
160.30 KHz with the increasing water content, with stirring rate of 400rpm. The characteristic frequency was fitted to a cubic function

$$y = -0.0012x^3 + 0.1065x^2 - 4.4039x + 366.6557$$

(1)

and had a correlation coefficient ($R$) of $\sim 0.99$, which is shown as the red line in Fig. 3(b). This indicates that the cubic function represents the data well.

To study the characteristic frequency, the impedance parameters was displayed in Nyquist diagram. As shown in Fig. 4, a Nyquist diagram for oil–water emulsions with different water contents shows only one semi-circle at a frequency interval of between 50 to $10^6$ Hz. For all cases, one semi-circle is displayed, indicating a homogeneous distribution of the time constants within the frequency range under scrutiny. At frequencies of below $10^3$ Hz, a constant-phase trend starts to take place and rapidly becomes dominant. This is attributed to the electrode polarization, which is related to the inhomogeneous distribution of charges on the electrode surface.\cite{24,25} In this study, a frequency range of $10^3$ to $10^6$ Hz (a subset of the entire range was applied during the experiments) was used for equivalent circuit modeling. Because this frequency range is higher than the threshold where the constant-phase behavior begins, electrode polarization is not a dominant factor in the changes to the modeled resistance and capacitance.\cite{26}

The nonlinear least-squares (NLLS) Marquardt-Levenberg algorithm was implemented in Z-view software (Scribner Associates, Southern Pines, NC) to fit an electrical model to the experimental data and estimate the set of parameters. The equivalent circuit can contribute to an establishment of the correlations between the electrochemical system parameters and the characteristic impedance elements. The data were successfully modeled as a Randles circuit using Z-view software.\cite{24,25} The equivalent circuit contains a resistor (Rs) in series with a parallel R|C, as shown in the inset of

FIG. 3. (a) Rate dependence values of characteristic frequency. The sphere and lines with various colors represent the different water contents. (b) Characteristic frequency dependence of water contents. The red line represents the fitting cubic function of the Fs and water content.
Fig. 4. Nyquist diagram of emulsions with water content of 10 – 70%. The inset illustrates the equivalent circuit model of the oil-water emulsion system.

Fig. 4. Here, R, Rs, and C represent the lead resistance, sample capacitance, and sample resistance, respectively.

Based on the Randles circuit model, the impedance of the emulsion system is as follows:

$$Z = \frac{R(1 + j\omega R_s C)}{1 + j\omega C(R + R_s)}$$

The real and imaginary parts of impedance $Z'$ and $Z''$ of an emulsion can be obtained from Eq. (2):

$$Z' = R_s + \frac{R}{1 + \omega^2 C^2 R^2}$$

$$Z'' = \frac{\omega C R^2}{1 + \omega^2 C^2 R^2}$$

It can be seen that $Z'$ reaches the maximum value $Z_{b\max}$ at $F_s$, which can be seen as the characteristic frequency of the circuit model:

$$Z_{b\max} = \frac{R}{2}$$

$$F_s = \frac{1}{2\pi R t} = \frac{1}{2\pi RC}$$

It is considered that the characteristic frequency of the model is the model value of experimental $F_s$, which can be calculated by the resistance and capacitance parameters.

The non-linear regression partial least square program is used to adjust the parallel elements R and C with an error of less than 1.5%, and the Chi-squares show a magnitude of $10^{-4}$. The variation of the parameters with the water content is shown in Fig. 5(a). With an increase in water content, it is observed that the R of the emulsion decays from 5372 to 5114 ohm, with a relative variation of 4.80%. C has an upward trend from 92.57 to 199.60 pF, with a relative variation of 115.62%. The changes of the impedance parameters of the emulsions are related to the electrical properties of the crude oil and distilled water. Comparing with the crude oil, the distilled water has higher conductivity at the same temperature. The higher proportion of water phase is, the lower value of resistance of the emulsions remains. At ambient temperature, the distilled water’s permittivity is $\sim$80, which is much higher than that of crude oil (2.0$\sim$2.6). With the water content increasing, the permittivity and the capacitance increase. More importantly, it is obvious that the variation of characteristic frequency is mainly caused by the variation of capacitance with the increasing water content. It is also noted that the changing rate of capacitance is increasing with the increasing of the water content. The trend can be explained by the dielectric characteristics of the emulsions. In the case of water in oil emulsions, it is assumed that the conductivity of the oil phase is much lower than that of the water droplets. For the concentrate suspensions of spherical droplets, the following approximate equations are obtained from Hanai’s equation.
FIG. 5. (a) Resistance and capacitance of emulsions vs. water content. (b) Resistances and capacitances of emulsions vs. stirring rates. The sphere and lines with various colors represent the different water contents.

\[ \phi = 1 - \left( \frac{\varepsilon_a}{\varepsilon_l} \right)^{\frac{1}{3}} \]  

(5)

Where \( \phi \) is the water content of the emulsion system, \( \varepsilon_a \) is the relative permittivity of the continuous medium, and \( \varepsilon_l \) is the limiting values of the permittivity at low frequency, which can be seen as the static permittivity. In this work, the capacitance of the emulsions can be describe by the static permittivity

\[ C = k \varepsilon_l \]  

(6)

Where the \( k \) is constant parameters related to the shape and size of the sample pool. Thus the capacitance can be described as

\[ C = \frac{k \varepsilon_a}{(1 - \phi)^3} \]  

(7)

Eqs. (7) illustrates that the increasing water content can lead to an increase capacitance, which caused the characteristic frequency increases with water content in a cubic trend.

The R and C of the emulsions with different water contents (10%, 30%, 50%, 70%) are studied under different dynamic conditions (200, 300, 400, 500 rpm). As shown in Fig. 5(b), the R keeps unchanged with the increasing rate, while the C has an increasing trend in the emulsions with high water content. For the emulsion with water content of 70%, the C increases from 173.9 to 199.8 pF with a relative variation of 15.0%. The stirring rate increases the number of dispersed droplets and decreases the size of dispersed droplets, thus it leads to the increasing of C. Consequently, the results show that the rate has a significant influence on the electrical parameters, especially the C of the emulsions with high water content.

In the petroleum industry, the secondary exploitation strategies lead to the generation of water content in crude oil. The water content of crude oil has a major impact on the process of exploitation, storage, and transportation of an oilfield. The dynamic emulsions always maintain more stable water
content under the shear conditions, which caused the low accuracy of the static testing method. However, there is few researches focus on the dynamic emulsions. Basing on the frequency dispersion of the oil-water two phases system, the impedance spectra technology can be served as an appropriate method to promote the development of oil industry. In this work, the impedance characteristics of emulsions with different water contents were studied by a dynamic testing apparatus. The method provides the possibility of characterizing the water content under dynamic conditions.

IV. CONCLUSION

In this work, a laboratory testing apparatus successfully characterized the water content of dynamic emulsions. Based on this, we investigated the potential of impedance spectroscopy to determine the water content in emulsions under the influence of a shear force.

The results show that the characteristic frequency acquired from impedance spectra exhibits a decrease with an increase in water content with a cubic function. An equivalent circuit model was provided to understand the impedance characteristics of the emulsion systems. Basing on an equivalent circuit model, the capacitance has an obvious increasing trend with the increasing water content, which can be explained by the dielectric equations related to the water content. In addition, the shear force has an influence on the electrical parameters, thus the dynamic testing is necessary for the characterization of the water content. Further investigation on the behaviour of droplets will be performed to understand the effect of the dynamic conditions on the impedance properties of the emulsions. To summarize, a strong cubic correlation exists between the characteristic frequency and the water content. This correlation can significantly reduce the complexity of dynamic measurements of the water content of crude oil emulsions, and contribute to a characterization of the water content in flowing emulsions.

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