Method for Judging the Stability of Asphaltenes in Crude Oil
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ABSTRACT: The colloidal instability index (CII) has been widely used for testing the stability of asphaltenes as a classical method. In this study, five oil samples were tested by the CII method; the results obtained greatly differ from the real field results. In our investigation, we combined the Turbiscan LAB stability analyzer and saturate, aromatic, resin, and asphaltene (SARA) analysis to further investigate the asphaltene stability by heptane titration. The results revealed that there exists a threshold volume ratio before the asphaltenes destabilize. The stability of crude oil is related to the saturation solubility of asphaltenes. By testing the CII value of the crude oil in its current state and the CII value of the dissolved asphaltenes in its saturated state, we were able to propose a new way to judge the oil stability.

1. INTRODUCTION
Oil field production is often accompanied by asphaltene deposition. In China, based on a large survey of crude oil production, researchers have drawn a conclusion that light oil is more susceptible to cause asphaltene deposition as compared to heavy oil, much more so in light oil with a higher gas–oil ratio (GOR). One of the most common explanations for this kind of phenomenon is that the evolution of gas initiates the onset of asphaltene instability. Other parts of research indicate that petroleum stability basically depends on characteristic interaction between asphaltenes and resins in the oil. Other factors include but are not limited to the crude oil fractions content, production conditions, shear stress, gas injection, and other well-stimulated processes. Asphaltene deposition is a serious problem during the production of crude oil. It may occur in oil reservoirs and well tubings depending on the state of the crude oil. When the asphaltenes precipitate and deposit in the reservoirs, they lead to alteration of rock surface wettability and thus lower the relative oil permeability. On the other hand, asphaltenes’ deposition in the well tubing results in pipe plugging and affects the overall efficiency of production and thus additional costs. Therefore, the accuracy of the method to judge the crude oil stability plays an important role in the mitigation of asphaltene deposition.

The colloidal instability index (CII), introduced by Asomaning and Watkinson to evaluate the propensity of residual oil to form sediments, is calculated based on the information about the oil SARA fractions in the oil. Other methods have also been postulated to predict the risk of asphaltene deposition that includes asphaltene instability trend (ASIST), colloidal stability index (CSI), stability index, Sepúlveda stability criterion (SCP), oil compatibility model (OCM), among others. Although these methods are the most widely used to distinguish very stable oils from very unstable ones, experience over the past years has shown that predictions tend to be somewhat pessimistic, indicating asphaltene precipitation in cases where no problems develop in the field.

In our investigation, the SARA analysis experimental test using the thin-layer chromatography and flame ionization detection (TLC-FID) analytical method and the oil stability analysis test using the Turbiscan LAB stability analyzer were performed with the goal of modifying the classical SARA model to provide a more reliable way to determine the stability of oil regardless of the location and methods.

1.1. SARA Analysis. The SARA analysis method is applied to this work. SARA stands for saturates, aromatics, resins, and asphaltenes. By determining the proportions of the aforementioned constituents in the oil, we can calculate the colloidal instability index (CII) of the oil. The CII is a ratio of the sum of quantities of saturates and asphaltenes to the sum of quantities of resins and aromatics. This ratio can be used to estimate the stability of the crude oil with respect to asphaltene deposition. The CII is calculated as per eq 1

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\[
\phi_{\text{oil}} \delta_{\text{oil}} + \phi_{\text{asph}} \delta_{\text{asph}} = \phi_{\text{oil}} \delta_{\text{oil}} + \phi_{\text{asph}} \delta_{\text{asph}}
\]

where \( \phi_{\text{oil}} \) and \( \phi_{\text{asph}} \) are volume fractions of oil and asphaltenes, respectively, and \( \delta_{\text{oil}} \) and \( \delta_{\text{asph}} \) are their respective solubility parameters.

Therefore, a lower value for the Hildebrand solubility parameter signifies a rapid rate of asphaltene precipitation. Studies have also shown that a relationship exists between the amounts of solvent and nonsolvent at the onset condition (eq 5).

\[
\frac{V_n}{V_o} = S \frac{V_s}{V_o} + I
\]

where \( V_n \) and \( V_o \) represent the maximum mixing of oil and heptane at which the crude oil remains stable, and \( V_s \) represents the volume fractions of oil and asphaltenes, respectively; and \( S \) is the slope; and \( I \) is the intercept of a straight line through the \( V_n \) axis. Equation 5 can be rewritten as

\[
V_n - SV_s - ID_0 = 0
\]

Given the total volume \( V_T \), dividing eq 6 by \( V_T \) gives eq 7.

\[
\phi_{\text{oil}} - S \phi_{\text{asph}} - ID_0 = 0
\]

When the solvent is highly diluted, the intercept \( I = \frac{V_s}{V_o} \) represents the maximum mixing of oil and heptane at which the crude oil remains stable, this value can be tested by the near-infrared light.

## 2. RESULTS AND DISCUSSION

SARA analysis of crude oil prior to the addition of asphaltenes is shown in Table 1. Each oil sample was tested four times to minimize errors in the calculation of the colloid instability index (CII).

For CII \( \leq 0.7 \), the oil is stable. When CII is \( \geq 0.9 \), oil is unstable.\(^{22}\) In the CII range \( 0.7 \leq \text{CII} < 0.9 \), the stability status of the crude oil is not clear. However, because stability occurs below 0.7, it is plausible to refer to the oil in the 0.7 < CII \( \leq 0.9 \) range as unstable or at least at the brink of losing its stability. Other studies have suggested that this value can even be lower than 0.7.\(^{25}\)

By these standards, according to the calculated CII results in Table 1 for different types of oils, it is safe to say that the oil is unstable.

However, this was contrary to reality, as field reports showed neither deposition of asphaltenes in the pipes nor its associated bottlenecks. Nevertheless, at this point, it is imperative to emphasize that this does not in any way imply that the CII model is not a suitable method for this kind of oil because asphaltene deposition depends on various factors like temperature, pressure, production rates, composition, and other properties.\(^3\)

The LAB stability analyzer was used to test the stability of the crude oil by adding different volume ratios of n-heptane, and the transformation ratio and the Turbiscan stability index

### Table 1. Fractions of Crude Oil Based on SARA Analysis

| Sample | Saturates/% | Aromatics/% | Resins/% | Asphaltenes/% | CII | Stability | Reality |
|--------|-------------|-------------|----------|--------------|-----|-----------|---------|
| W1-1   | 87.93       | 8.73        | 2.52     | 0.82         | 7.89| unstable  | stable  |
| W1-2   | 87.79       | 10.39       | 1.27     | 0.55         | 7.58| unstable  | stable  |
| W1-3   | 84.56       | 13.03       | 1.43     | 0.70         | 5.90| unstable  | stable  |
| W1-10  | 81.44       | 9.68        | 1.82     | 1.03         | 7.17| unstable  | stable  |
| W1-11  | 84.55       | 11.04       | 1.02     | 0.50         | 7.05| unstable  | stable  |

"Reality" reflects the status of oil based on the results from the oilfield production reports.

According to Ashoori et al.,\(^{22}\) crude oil is said to be stable or unstable when its CII is <0.7 or >0.9 respectively. Other literature sources have suggested low values with Iatroscan TLC-FID and the IFP 9305 methods.\(^{25}\) The proportions of the saturates, aromatics, resins, and asphaltenes in the crude oil can be obtained using the TLC-FID analytical method.\(^{26}\)

1.2. Solubility of Asphaltenes in Crude Oil. In a study by Chandio et al.,\(^{27}\) it was stated that the regular solution theory is mainly applied to explain the solubility and precipitation of asphaltenes. In our investigation, on the basis of this theory, we asserted that asphaltenes are soluble in crude oil. The ability of the crude oil to dissolve the asphaltenes can be explained by the Hildebrand's solubility parameter. The crude oil in this case acts as a solvent, whereas the asphaltenes act as the solute. Based on this theory, when the cohesive energy densities of the asphaltenes and the crude oil are or close to being identical, then maximum solubility of the asphaltenes can be assumed. At this point, it can be said that crude oil is saturated with asphaltenes.

The Hildebrand solubility parameter (\( \delta \)) of a soluble compound with measurable energy of vaporization and molar volume, \( V \), can be calculated from the experiment, since \( \delta \) is defined as

\[
\delta = \sqrt[3]{\frac{\Delta U}{V}}
\]

where \( \Delta U \) is the energy of vaporization to the ideal gas state.

For a mixture of liquids that are mutually soluble, the solubility parameter is proportional to the sum of products of the solubility parameters and volume fractions of the individual liquids in the mixture. Thus, for a given number, \( N \), of liquids in the mixture, the Hildebrand solubility parameter (\( \delta_{\text{mix}} \)) of the mixture is given by eq 6.

\[
\delta_{\text{mix}} = \sum_{i=1}^{n} \phi_i \delta_i
\]

where \( \phi_i \) and \( \delta_i \) are the volume fraction and solubility parameter of liquids in the mixture, respectively.

Seifried et al.\(^{28}\) performed an experiment using the confocal laser scanning microscopy to determine the solubility parameter for a mixture of oil, solvent, and titrant.

Mutelet et al.\(^{29}\) using eq 4 were able to estimate the solubility parameter of the crude oil.

\[
\delta_{\text{crudeoil}} = \frac{\phi_{\text{oil}} \delta_{\text{oil}} + \phi_{\text{asph}} \delta_{\text{asph}}}{\phi_{\text{oil}} + \phi_{\text{asph}}}
\]
(TSI) of the mixture were recorded; the results are shown in Figure 1.

The threshold heptane–oil volume ratio represents the onset of asphaltene precipitation (OAP). Take W1-1 for instance, asphaltenes of W1-1 start to precipitate when the heptane–oil volume ratio is 1:1 (Figure 1A); below this volume ratio, the asphaltenes can exist in the crude oil as stable components. This indicates that the asphaltenes are not at their saturation point in the crude oil. Another important observation from Figure 1A is that the higher the heptane–oil volume ratio, the faster asphaltenes start to precipitate in the oil and the higher the TSI value. Other oil samples also exhibited a similar trend, as shown in Figure 1B.

In Figure 1A, we marked two volume ratios, 0.6:1 and 1:1, as A and B, respectively. We compared the backscattering signal of these two ratios with the Turbiscan LAB stability analyzer so as to further help us understand the asphaltene precipitation phenomenon (Figure 2). The dispersion stability of the asphaltenes in the two ratios of heptane was characterized.

Figure 2 shows the delta backscattering profiles obtained from the asphaltenes with the two heptane–oil volume ratios over a period of 30 min. In the delta backscattering profiles, we found that the curves of the oil with low heptane–oil volume ratio (0.6:1) were almost constant with time (Figure 2A), whereas those of crude oil with a high ratio (1:1) changed dramatically (Figure 2B). From the results of Figure 2B, we distinguished the tube axis into three regions: the bottom 1–10 mm, the middle 11–40 mm, and the top 41–50 mm (Figure 9). At the start of the test, the backscattering signal mostly is constant, this is because asphaltenes need time to precipitate. As time progresses, the signal becomes unstable because much of the asphaltenes have started to precipitate.
from the solution and coalesce. At the end of the experiment, because the asphaltenes settle from the solution and fall from the top to the bottom, the backscattering signal was the strongest at the bottom, moderate in the middle, and weakest at the top. The Turbiscan LAB stability test results are shown in Figures 3–6.

These five oil samples exhibited the same behavior (Figures 2–6), and as such these results confirm that the oil has the ability to maintain its stability upon addition of trace amounts of n-heptane. We confirmed that the threshold volume ratio of the heptane–oil can reflect the solubility of the asphaltenes in the oil. When the asphaltenes reach their saturation point in the crude oil, the addition of any trace amount of n-heptane will lead to their immediate precipitation. Thus, at this point, when the SARA analysis of crude oil was performed and a new CII calculated (Critical CII), its value provided a more accurate benchmark than the originally suggested CII benchmarks.

To saturate the crude oil with asphaltenes, sufficient amounts of asphaltenes obtained under the ASTM D3279 procedure were dissolved for 1 hr in their respective crude oil samples with the aid of ultrasonic vibration (Table 2). Then, the TLC-FID analyzer was used to determine the SARA composition and the critical CII calculated; results are shown in Table 3.

Table 2 shows the ability of the crude oil samples to dissolve extra solid asphaltenes. The ultrasonic vibration was helpful in dispersion and dissolution of solid asphaltenes, and the amount
of asphaltenes dissolved by crude oil is related to the content of resins and aromatics.

In Table 3, the obtained critical CII for the five oil samples is reported; this critical CII can be used to evaluate the stability of the oil. Take W1-1 for instance, if the CII value of W1-1 is below 9.01, then the oil is stable and above this value, the oil loses its stability.

Thus, from these results, it can be said that for different types of crude oils, the saturation of asphaltenes in the oil is a very important factor and this can be reflected by the threshold volume ratio of the heptane–oil. Therefore, in this case, the classical standards for the CII value can only be used as a spot reference for determining the oil stability but not as a representation of the actual field situation.

3. CONCLUSIONS

In summary, the composition of oil and characteristic behavior of asphaltenes precipitation in crude oil were investigated. The results showed that the classical CII value calculated from the SARA analysis can only be considered as a spot determination of stability of the oil rather than a deterministic value. We also confirmed that crude oil cannot lose its stability until its asphaltene composition is close or approximately equal to its saturation point. This was determined through the use of varying heptane–oil volume ratios for which a ratio was attained at which the oil is saturated with asphaltenes. This ratio marked the onset of asphaltene deposition and was referred to as the threshold heptane–oil volume ratio. At this ratio, SARA analysis was performed and produced a new CII value, which we referred to as critical CII in this paper. We determined that for any CII value below this critical value, the oil is stable and above this value, its unstable. This is the key to remedying asphaltene-related problems during oil production because by monitoring the CII of the crude oil, asphaltene remediation measures can commence when the CII approaches this critical value. Therefore, we conclude that this method is a more deterministic way for judging the stability of crude oil and reduces the level of uncertainty in the classical CII determination method.

4. EXPERIMENTAL SECTION

4.1. Materials

4.1.1. Crude Oil Samples. To make the correction model more accurate, five depressurized oil samples were used in this study. They were obtained from oil fields located in the northwest of China. The test oil samples were named as follows: W1-1, W1-2, W1-3, W1-10, and W1-11. These samples represent crude oil from the F1-1 fault reservoir with an average depth of more than 7000 meters. The reservoirs are characterized by high temperature, high pressure, and high gas oil ration (GOR). Detailed information is shown in Table 4.

4.1.2. Solvents. n-Heptane was available in 99.9% purity and obtained from Shanghai Macklin Biochemical Technology Co.

4.1.3. Asphaltenes. Asphaltenes are obtained from crude oil samples using the separation procedure ASTM D3279.

4.2. Apparatus.

4.2.1. TLC-FID Analysis. The evolution of SARA fraction was tested by the TLC-FID analytical apparatus. A detailed description of the experimental apparatus can be found in the earlier publications. The schematic diagram of the apparatus used in this study is illustrated in Figure 7. The main part of the apparatus consists of oxygen and hydrogen flow rate ratio, which influences the stability of test results and causes deviation peaks. SARA analysis of crude oil is shown in Tables 1 and 3.

4.2.2. Turbiscan LAB Stability Analyzer. The Turbiscan can be used to test the stability of colloid and demonstrate the instability process (flocculation, aggregation, and deposition). The Turbiscan mainly contains a near-infrared light source, transmission detector, and a backscatter detector. The wavelength of near-infrared is 880 nm, which allows the detection of particle 0.1 μm to 1 mm in size. Figure 8 shows a simple schematic of Turbiscan using multiple light scattering methods.

The near-infrared light is periodically scanned along the height of the sample cell during the set time interval, and both the backscattered light signal and the scanning time are recorded. The method is based on multiple light scattering mechanisms. The backscattered flux (BS) and the Turbiscan stability index (TSI) were employed to judge the stability of the crude oil. These parameters are calculated as per eqs 8 and 9, respectively.

\[ BS = \frac{1}{\sqrt{\lambda^*}} \]  

(8)

where \( \lambda^* \) is the phototransport mean free path that is given by the equation

\[ \lambda^*(\phi_i, d) = \frac{2d}{3d(1 - g)Q_i} \]  

(9)

The parameter \( g(d) \) is the asymmetric factor and \( Q_i(d) \) is the ratio of extinction cross section to the geometrical cross section.

Table 3. Critical CII of Crude Oil Saturated with Asphaltenes

| sample | saturates/% | aromatics/% | resins/% | asphaltenes/% | critical CII |
|--------|-------------|-------------|----------|---------------|--------------|
| W1-1   | 88.97       | 8.94        | 0.15     | 0.03          | 9.01         |
| W1-2   | 87.97       | 10.47       | 0.82     | 0.73          | 7.86         |
| W1-3   | 87.28       | 9.86        | 0.16     | 0.99          | 8.81         |
| W1-10  | 88.44       | 11.43       | 0.84     | 1.58          | 7.33         |
| W1-11  | 87.68       | 11.69       | 0.26     | 0.68          | 7.39         |

Table 4. Characteristics of Five Crude Oil Samples

| sample | comment                   | depth/m | reservoir temperature/°C | reservoir pressure/MPa | saturation pressure/MPa | GOR | density/(g/cm³) |
|--------|---------------------------|---------|--------------------------|------------------------|-------------------------|-----|----------------|
| W1-1   | oil belongs to F1-1 fault in northwest of China | 7458.00 | 157.44                   | 86.88                  | 34.87                   | 423 | 0.7960         |
| W1-2   |                                          | 7357.98 | 156.20                   | 85.69                  | 36.03                   | 457 | 0.7972         |
| W1-3   |                                          | 7639.71 | 162.4                    | 79.48                  | 34.31                   | 407 | 0.8008         |
| W1-10  |                                          | 7607    | 158.4                    | 86.35                  | 34.41                   | 409 | 0.8004         |
| W1-11  |                                          | 7630    | 155.9                    | 86.24                  | 35.60                   | 442 | 0.7988         |
section. They are both optical parameters that can be determined by the Mie theory.35−37

The parameter, \( \phi \), is the volume fraction of the colloids in the sample (asphaltenes).

Depending on the BS profile (backscattering flux), the TSI compares each and every scan to its previous one and the whole result is divided by the column height of the sample bottle (eq 10).

\[
TSI = \sum \frac{\sum_{i} \text{scan}_{i}(h) - \text{scan}_{i-1}(h)}{H}
\]

where \( \text{scan}_{i}(h) \) is the \( i \)th backscattered signal from the cell bottom to top, \( H \) is the height of the cell. TSI values can directly reflect the stability status of crude oil; the smaller values, the more stable the crude oil.33,38 The diagram of the experimental setup is presented in Figure 9.

4.3. Procedure. 4.3.1. Separation of Asphaltenes. Asphaltene separation followed the standard procedure recommended in ASTM D3279 (n-heptane as solvent). A solvent-to-oil ratio of 60:1 was used, which has been confirmed to have less effect on the physical and chemical properties of the asphaltenes deposited.39 This mixture was allowed to age for 8h before filtration with 0.22 \( \mu \)m filter papers, followed by oven-drying and weighing.

4.3.2. SARA Analysis. The SARA composition of five samples oil was analyzed using the TLC-FID apparatus according to the Industrial Specification of China Petroleum Standard SY/T 5119-2008.40 The procedure for sample preparation was in accordance to the standard suggested by Karlsen et al.31 The CII of each sample was calculated with the SARA data.

4.3.3. Determining the Oil Stability with the Turbiscan LAB Stability Analyzer. To further analyze the real stability of the asphaltenes, n-heptane was added to the oil and LAB stability analyzer was used to test the stability of the mixture. First, crude oil is placed in the sample cell at a stable temperature (25°C) and the emitted light only partially passes through the cell. In the next phase, a certain amount of n-heptane is titrated with the crude oil samples in the heptane−oil volume ratio of 0:1, 0.3:1, 0.5:1, 1:1, 1.5:1, and 3:1 which precipitates asphaltenes. Each volume ratio was tested for 30 min.

When the asphaltenes start to precipitate and coalesce, the turbidity of the oil becomes stronger, and it was apparent at this point that the backscattered light over the whole height increased due to asphaltene particles in the crude oil. The results are presented in Figure 1.
Obviously, our analysis is based on the assumption that in crude oil there exists a limit that is not affected by the addition of 2-n-heptane. Implying that when a low volume of n-heptane is titrated with oil, it would not cause the asphaltene precipitation and the LAB stability analyzer detects colloidal particles in the oil. This limit is determined by the ability of the crude oil to dissolve asphaltenes, thus a relationship between the solubility of asphaltenes and the ability of crude oil to dissolve asphaltenes.

Rahimi et al.\textsuperscript{41} pointed out in the colloidal dissolution model of the petroleum system that asphaltene aggregates are the core of the group of colloids in a petroleum system. Resins in the oil concentrated on the periphery of these colloids act as a protective layer around the asphaltenes, aromatics, and saturates. According to the colloidal dissolution theory, the solubility of asphaltenes in crude oil depends on the amount of resins, aromatics, and saturates. If the asphaltenes cannot dissolve anymore, it means that in this state, the asphaltenes are at their critical solubility in crude oil.

We added the asphaltenes to their respective untampered test oil samples from which their preparatory samples were obtained, 10 g of asphaltenes was put in the 40 g of crude oil in a cell, and the asphaltenes were allowed to dissolve for 1 h in the oil with the aid of ultrasonic vibration. By observing the bottom of the cell, we were able to judge that the asphaltenes had reached their critical solubility in the oil. The oil dissolved a certain amount of asphaltenes and the rest were deposited to the bottom of the cell. After standing for 45 min, we took the supernatant for new sample preparation, and the rest was filtered. n-Heptane was used to wash the undissolved asphaltenes on the filter paper until the washing solution becomes clean and colorless. Finally, the undissolved asphaltenes were dried and weighed.

The newly prepared sample was divided into two parts: the TLC-FID analyzer was used to test the SARA composition of oil, whereas the Turbiscan LAB stability analyzer was used to analyze the stability of the crude oil. Then, we were able to relate the results of SARA analysis and those of the Turbiscan stability analyzer.

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Notes

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