EXPERIMENTAL AND THEORETICAL STUDIES OF THERMODYNAMIC FEATURES AND PHASE BEHAVIOR OF HYDROCARBON MIXTURES

Original article
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Determining the onset of asphaltene precipitation in a model oil system toluene–asphaltene–heptane by ultramicroscopy method

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Abstract. Determining the stability threshold of oils and oil systems is an important task in the oil industry. It is important to be able to detect the precipitation of solid phase from oils at the earliest stages. For the first time, the ultramicroscopy method was used to study the precipitation of asphaltenes from a toluene solution during titration with heptane. The study allowed for the visualization of asphaltene aggregates in the toluene–asphaltene–heptane mixture at the earliest stages of aggregation. The relationship between the numerical concentration of asphaltene aggregates and the heptane concentration was measured. Analysis of this dependence led to the determination of the threshold concentration of heptane, above which asphaltene precipitation and aggregation (referred to as the “Onset point”) occurred. A comparison was made between the capabilities of ultramicroscopy and those of dynamic and static light scattering methods for determining the “Onset point”. It was shown that the ultramicroscopy method has greater sensitivity and can detect the onset of aggregation at lower concentrations of the precipitant.

Keywords: asphaltenes, aggregation, oil, ultramicroscopy, light scattering, disperse systems

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Introduction

Asphaltenes are high-molecular fractions of oil. Together with other oil fractions, such as paraffins and resins, asphaltenes can form asphalt–resin–paraffin deposits (ARPD) during oil field development, as well as during the transportation and processing of extracted oil. The formation of ARPD leads to a decrease in well flow rates, reduces the effective diameter of oil pipelines and may cause the failure of process equipment. Finding a solution to the ARPD problem is a critical challenge in the oil industry. One of the key steps in addressing this issue is studying the processes of asphaltene precipitation from oil and oil systems.

Important information for an oil system containing dissolved asphaltenes is under what conditions and how asphaltenes begin to precipitate from such a system as a solid phase. Precipitation of asphaltenes from solution can occur when the temperature, pressure or composition of the oil system changes. An established method for assessing the stability of oil systems is the process of titrating such a system with an asphaltene precipitant (a liquid alkane, such as heptane or pentane) and determining the concentration of the precipitant, the excess of which leads to precipitation of asphaltenes from solution and the beginning of their aggregation. There are many methods for detecting the onset of aggregation.

Densitometry is the determination of the onset of asphaltene aggregation by measuring the density of the mixture of the oil system during titration with a precipitant [1–3]. A widely used optical method for determining the stability threshold of oil systems is based on measuring the intensity of light transmitted through a sample titrated with a precipitant [4]. The “Spot” method– analysis of the image of a sample spot on filter paper – is also used [1]. Using the method of differential scanning calorimetry (DSC), it is possible to determine the temperature of the onset of precipitation of asphaltenes from a solution [1]. The pressure of the onset of asphaltene precipitation is determined using PVT installations with the ability to detect various physicochemical parameters of the sample. For example, by analyzing changes in the intensity of scattered light in the near infrared range [5], measuring the electrical conductivity of the sample [3], measuring the speed of sound, by the appearance of asphaltene aggregates detected by optical microscopy or using the filtration method, in which a small volume of the sample is filtered through a filter, and then the filter is analyzed for the presence of asphaltene particles on it [3, 5, 6]. Also, the onset of asphaltene precipitation from a sample of an oil system during titration can be determined using a spectrophotometer – by changes in the transmission spectrum [5] or by measuring the interfacial tension at the air–sample boundary [6, 7]. Asphaltene aggregation also leads to changes in the viscosity of the oil system, making viscometry another useful method for detecting aggregation onset [3, 5, 6].

The dynamic light scattering (DLS) method allows measuring the size of nanoparticles in liquid media. This method is also used to determine the threshold of resistance of petroleum systems to asphaltene precipitation [8–10]. At the same time, the DLS method allows studying the kinetics of asphaltene aggregate growth at early stages of aggregation [11, 12].

Not only experimental methods, but also modeling of petroleum systems is also one of the ways to predict the threshold of their resistance to asphaltene precipitation in the form of a solid phase [13].

It is important to note that at the time of writing this article, it was not possible to find any published works on the study of asphaltene precipitation using the ultramicroscopy method. In the work [14], a device based on the ultramicroscopy method and the method of nanoparticles tracking analysis (NTA) in oil systems were used, but not for studying the stability of oil systems, but for measuring the sizes of particles added to bitumen. An important difference between the ultramicroscopy method and the above-mentioned methods is that this method allows one to visualize and directly count the asphaltene submicron and nanoscale aggregates in the sample, and to study the aggregation process at the earliest stages.

Materials and methods

Asphaltenes for the studies were isolated from bitumen (asphaltenes content – 18 wt%) using the ASTM D6560 method by adding a 40-fold excess of *n*-heptane.

The purity of toluene used to prepare the samples in this work was 99.8%; heptane was of chemically pure grade.

Ultramicroscopy measurements were performed on an NP Counter nanoparticle concentration meter (NP VISION, Russia). This device uses a laser with a wavelength of 650 nm, a power of 50 mW and a digital camera with a matrix size of 5 megapixels, with a shooting speed of up to 52 frames per second. To estimate the sample volume in which the particles are counted, the device was preliminarily calibrated. A micrograph with a frame size of 640×480 pixels was taken for an object-micrometer with a division value of 0.01 mm. Particles in the sample were observed at the same resolution. From the analysis of the obtained micrograph of the object-micrometer, a connection was established between one pixel of the image obtained with this device configuration and the real distances in the image plane in micrometers of 0.619 μm/px. The depth of the part of the sample in which the particles are observed was estimated using the formula proposed by Rudolf Oldenburg and Michael Shribak [15]. For the lens with a magnification of 10 times used in the device, the depth of the field, according to our estimates, is about 15 μm.

Image analysis and particle counting occurs only in the frame area selected by the user (region of interest, ROI) – the central part of the laser beam visible in the frame. When measuring the particle concentration in a sample, a video of the sample under study is recorded at a rate of 30 frames per second. For each such frame, the image is analyzed in the ROI. The algorithms of the device software find all light objects in the ROI and count their number. The particle concentration is measured as the arithmetic mean of the concentration data obtained for each video frame.

DLS measurements were performed on a Photocor Compact-Z particle size analyzer (Photocor, Russia). The measurements were taken at a scattering angle of 90 degrees.

Weighing of components during sample preparation was performed on analytical balance FA2204N (0.1 mg, Jaonlab, China).

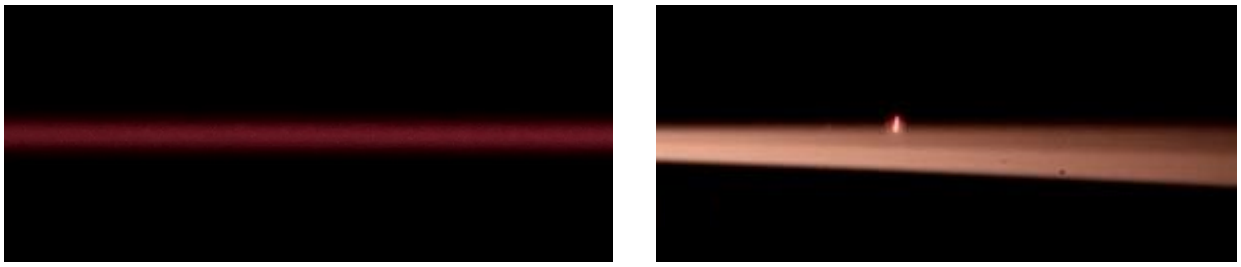
Initially, a solution in toluene with a concentration of 1 g/l was prepared from dry asphaltene powder and kept in a dark place for 24 hours until the asphaltenes were completely dissolved. Then, a sample for research with a concentration of 0.1 g/l was prepared from part of this sample and was also kept in a dark place for 24 hours before measurements.

The threshold concentration of heptane was determined using the following method. 0,3 ml of the solution was collected into a 2 ml sapphire cuvette using variable volume dispensers and then heptane was added step by step (by titration). First, 100 μl was added three times, then the step was reduced to 30 μl.

After each addition of heptane, measurements were carried out using the DLS and ultramicroscopy methods. Accordingly, after each titration step, information on the scattered light intensity, particle size (if particles were detected by DLS) and particle number concentration was obtained for the sample.

Results and discussion

Figs. 1a and 1b show the field of view of an ultramicroscope when observing pure toluene and a 0.1 g/l solution in toluene respectively. Due to light scattering on toluene molecules (Rayleigh scattering), a focused laser beam is visible in Fig. 1a, while no particles are observed in toluene. In the sample of asphaltene solution in toluene (1b), the laser beam is slightly defocused, apparently due to local heating of the sample by laser radiation (thermal lens effect). Several submicron-sized particles can also be observed. Due to the heating of the sample by the laser beam, the particles perform a constant convective movement upward. For this reason, the images of luminous objects in the photo are slightly blurred. At this stage of the research, it is not possible to establish the exact size and nature of these particles. Apparently, this sample of asphaltene solution contains components insoluble in toluene. Their concentration can be estimated at about 10^6 pcs/ml.



а/a б/б

Fig. 1. Field of view of an ultramicroscope (NP Counter) when observing pure toluene (a) and a sample of 0.1 g/l asphaltene solution in toluene (b)

Рис. 1. Поле зрения ультрамикроскопа (NP Counter) при наблюдении чистого толуола (а) и образца 0,1 г/л раствора асфальтенов в толуоле (б)

A 0.1 g/l asphaltene solution in toluene was titrated with heptane, measuring the scattered light intensity, the correlation function of scattered light intensity fluctuations, and the particle number concentration in it after each addition of the next portion of heptane. Fig. 2 shows the dependence of the particle number concentration (C_N) in the sample on the amount of added heptane.

Fig. 2 shows that the particle concentration increases very slightly from 0% to 65%. Such increase in concentration is due to the short-term formation of a local high concentration of heptane with each addition to the sample during titration and the appearance of a small number of aggregates in this, briefly supersaturated with heptane, region. At the same time, starting from 69%, a significant increase in the concentration of asphaltene particles is already observed. At concentrations of 75–76%, the ultramicroscope field of view changes – there are a lot of particles and their images overlap each other. At such and higher concentrations of particles in the sample, it is no longer possible to measure the concentration with good accuracy using the ultramicroscopy method without additional dilution of the sample.

Due to the overlapping of the images of two particles in the frame, such objects will be perceived by the program algorithms as a single object and the measured concentration will be underestimated. Fig. 3 shows an image of the field of view of an ultramicroscope when observing a sample of asphaltene solution of 0.1 g/l at 74 vol%.

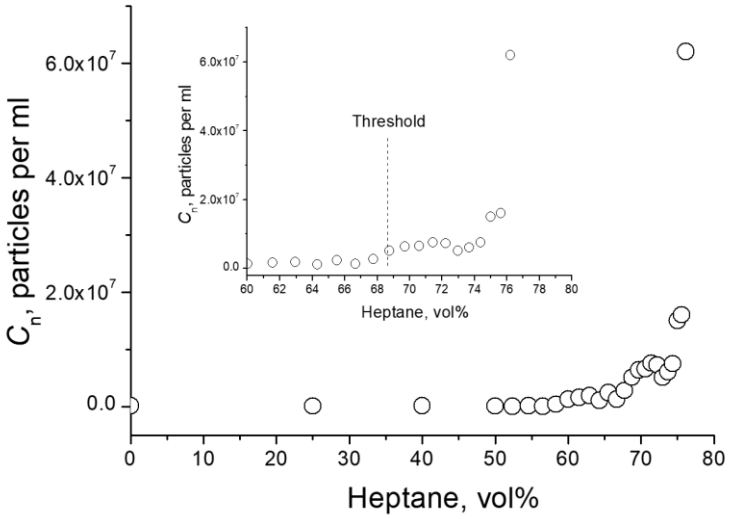


Fig. 2. Dependence of the numerical concentration of particles in a 0.1 g/l sample of asphaltenes in toluene on the volume concentration of heptane during titration

Рис. 2. Зависимость численной концентрации частиц в образце 0,1 г/л асфальтенов в толуоле от объемной концентрации гептана при титровании

Comparison of Figs. 1b and 3 clearly shows the qualitative difference in particle concentration during this experiment, while Fig. 2 provides a quantitative estimate of the concentration change during titration. This allows one to determine the threshold heptane concentration above which asphaltenes begin to precipitate from the solution and aggregate.

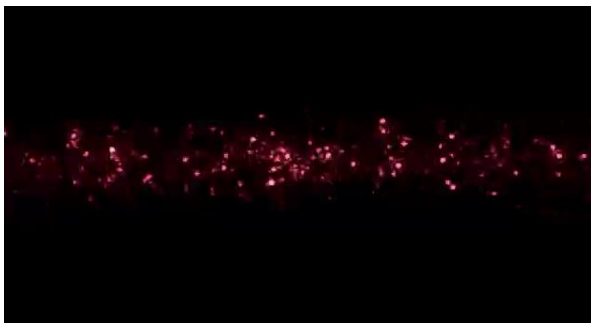


Fig. 3. Ultramicroscope field of view image of a 0.1 g/l asphaltene solution sample in toluene after adding 74 vol% heptane by titration

Рис. 3. Изображение поля зрения ультрамикроскопа при наблюдении образца раствора асфальтенов 0,1 г/л в толуоле после добавления титрованием 74 об.% гептана

During titration of a 0.1 g/l asphaltene solution with heptane, the dependences of scattered light intensity and particle size (if detected by DLS) on the heptane concentration were measured simultaneously with ultramicroscopy measurements. Fig. 4 shows the dependence of scattered light intensity on the heptane concentration during titration.

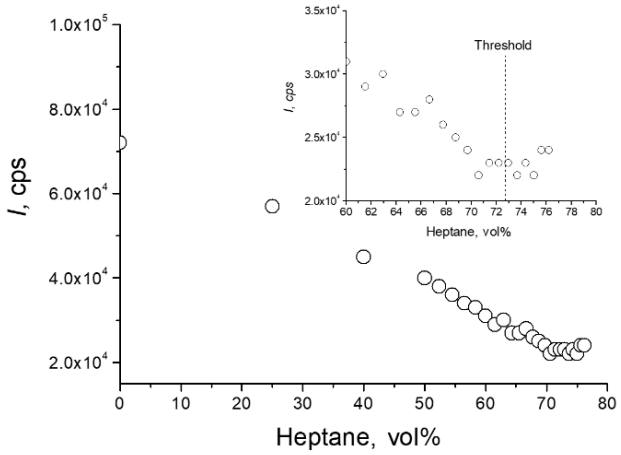


Fig. 4. Scattered light intensity vs. heptane volume concentration for a 0.1 g/l asphaltene solution in toluene during titration, where cps is counts per second

Рис. 4. Зависимость интенсивности рассеянного света от объемной концентрации гептана для раствора 0,1 г/л асфальтенов в толуоле при титровании, где cps – counts per second (импульсы в секунду)

Fig. 4 clearly shows that as heptane was added to the sample to 71–72%, the scattered light intensity decreased due to the dilution of the asphaltene solution (brown solution) with a transparent liquid (heptane). Starting from 72–73%, the scattered light intensity begins to slowly increase with increasing heptane concentration. For a sample containing 76% heptane, the scattered light intensity will increase without adding heptane, since the asphaltene aggregation process has begun in the sample. For a sample with 76.2% heptane, the intensity changed from 24,000 cps to 36,000 in 15 minutes after titration was stopped.

At the penultimate titration step (75.6%), no particles were detected in the sample by the DLS method – processing of the correlation functions of the scattered light intensity did not provide reliable information on the particle sizes. At a concentration of 76.2% (the last iteration of titration), processing the correlation functions measured by the DLS method allows us to detect the presence of particles in the sample and measure their hydrodynamic radius. The size of asphaltene aggregates (hydrodynamic radius) in the sample after completion of titration (76.2%) measured by the DLS method was about 60 nm. Since the concentration of heptane in such a sample is close to the threshold, the growth rate of the average size of aggregates is quite low. 15 minutes after the end of titration, taking into account the accuracy of the DLS measurement, the average particle size in the sample did not change, despite the fact that the intensity during this time increased from 24,000 cps to 36,000 cps. At heptane concentrations significantly higher than the threshold, the growth rate will be high [11].

As seen from the conducted studies, the threshold concentration values of heptane for the studied sample, determined by two different methods (DLS and ultramicroscopy), are very similar. The ultramicroscopy method detects the onset of asphaltene precipitation from the solution at lower concentrations compared to the DLS method, which measures the size of the precipitated asphaltene aggregates. This is because, for DLS to accurately measure particle size in a liquid, the concentration of the particles must be sufficiently high.

Conclusion

For the first time, the ultramicroscopy method was used to determine the threshold concentration of heptane that leads to the onset of asphaltene precipitation from a solution in toluene. It was shown that this method allows one to measure the concentration of asphaltene aggregates in model oil systems and, from the analysis of such measurements during titration, determine the threshold value of the precipitant (heptane). Comparison of the results of determining the threshold concentration of heptane by the ultramicroscopy method and the DLS and SLS methods showed that the ultramicroscopy method is not inferior to these two methods in sensitivity. Moreover, it can be said that the ultramicroscopy method provides the ability to study earlier stages of asphaltene aggregation than the DLS and SLS methods, since it allows one to detect individual nanoparticles in a liquid.

Conflict of interests

The author declares no conflict of interests.

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ЭКСПЕРИМЕНТАЛЬНЫЕ И ТЕОРЕТИЧЕСКИЕ ИССЛЕДОВАНИЯ ТЕРМОДИНАМИЧЕСКИХ СВОЙСТВ И ФАЗОВЫХ ПРЕВРАЩЕНИЙ УВ СМЕСЕЙ

Оригинальная статья
УДК 541.182
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Определение порога начала осаждения асфальтенов в модельной нефтяной системе толуол–асфальтен–гептан методом ультрамикроскопии

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Аннотация. Определение порога устойчивости нефтей и нефтяных систем – важная задача в нефтяной отрасли. Важно быть способным детектировать выпадение твердой фазы из нефтей на самых ранних стадиях. Впервые методом ультрамикроскопии проведено исследование выпадения асфальтенов из раствора в толуоле при титровании гептаном. Проведенные исследования позволили визуализировать появление асфальтеновых агрегатов в модельной системе толуол–асфальтены–гептан на самых ранних стадиях агрегации. Измерена зависимость численной концентрации асфальтеновых агрегатов от концентрации гептана. Анализ такой зависимости позволил определить пороговую концентрацию гептана, при превышении которой в образце начинается выпадение асфальтенов из раствора и их агрегация («порог устойчивости»). Проведено сравнение возможностей методов ультрамикроскопии и метода динамического и статического рассеяния света для определения «порога устойчивости». Показано, что метод ультрамикроскопии обладает большей чувствительностью и позволяет детектировать начало агрегации при более низких концентрациях осадителя.

Ключевые слова: асфальтены, агрегация, нефть, ультрамикроскопия, светорассеяние, дисперсные системы

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Конфликт интересов

Автор заявляет об отсутствии конфликта интересов.

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