Characterization of wellbore asphaltene deposition in well Gaotan-1 in Junggar Basin, China

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Abstract
Gaotan-1 is the first high-yield well explored in the southern margin of Junggar Basin, China, which is a major breakthrough in the oil exploration for Xinjiang Oilfield Company. The well produced high-quality light crude oil with very low sulfur content and total acid number. However, serious sedimentation occurred in the wellbore during production, which seriously affects production. In this paper, the sediments from Gaotan-1 were separated into organic soluble and inorganic solid fractions. The organic compounds were characterized by high resolution mass spectrometry and high temperature gas chromatography. It was found that the organic matter was rich in asphaltenes with very high molecular condensation. The high condensation compounds should be formed in the high temperature reservoir environment, which was extracted by the supercritical fluid formed by the light hydrocarbons under the ultra-high temperature and pressure. Due to its super-dissolving ability, the high-condensation hydrocarbons and asphaltenes are brought out of the reservoir, and deposit gradually mixed with silty sand on the wall of the wellbore as the temperature and pressure dropped in the process of oil exploitation.

Keywords Asphaltene · Sediment · FT-ICR MS · Supercritical fluids · Gaotan-1

Introduction
Discovery of the high-yield well Gaotan-1 in the deep strata made a major breakthrough in the lower assemblage of the southern margin of Junggar Basin, China (Du et al. 2019; Zhang et al. 2020). The Gaotan-1 oil was classified as normal mature light oil well preserved from thermal cracking despite its huge burial depth (Zhang et al. 2020). However, deposition on the pipe wall appeared during the production process, which blocked the wellbore and seriously affected normal production. The pipeline pigging was conducted 4 times in a year. This is inconsistent with the inference that crude oil is light and should theoretically be unlikely to cause deposition. Therefore, it is urgent to clarify the formation mechanism of solid deposition, so as to provide a basis for the improvement of on-site engineering processes and the formulation of preventive measures.

In the process of oil exploitation, due to changes in the external environment and its own components, organic matter, inorganic salts, and mud sand impurities in the crude oil were prone to deposit from the reservoir to the wellhead (Rajan Babu et al. 2019). The reasons for the deposition of crude oil in the wellbore are very complicated, causing damage of formation, crude oil production decline, wellbore scaling, great problems for production, and seriously affecting the normal production and economic benefits of oil fields (Rajan Babu et al. 2019; Chen et al. 2013). It is important to determine the type of sediments and to explore the main influencing factors of sedimentation, so as to provide basic data and theoretical support for the development of oil well descaling technology.

Asphaltene deposition is a drastic issue in the petroleum industry and has been subject of many studies (Rajan Babu et al. 2019; Ashoori et al. 2017). Most studies focused on the stability (), aggregation (Chaisoontornyotin et al. 2016; Mohammadi et al. 2015; Nguele et al. 2016; Painter et al. 2015; Rahmani et al. 2005; Rogel 2004), deposition (Al Sultan et al. 2018; Buckley 2012; Kuang et al. 2018; Fávero and
C., Hanpan, A., Phichphimok, P., Binabdullah, K., Fogler, H. S., 2016), and inhibition (Aquino-Olivos et al. 2001; Bagherpour et al. 2020; Hu and Guo 2005; Mohammadi et al. 2011; Raj et al. 2019; Rogel 2011; Smith et al. 2008; Yakubov et al. 2016) of the asphaltene based on molecular solution theory, colloidal solution theory, or temperature–pressure theory. Detailed compositional analysis of asphaltene is beneficial to develop more cost-effective methods to control the deposition of asphaltenes and to increase the overall efficiency of the processing fields with asphaltene problems (Klein et al. 2006). Recent years, the development of Fourier transform ion cyclotron resonance mass spectrometry (FT-ICR MS) enabled molecular characterization of petroleum asphaltenes. FT-ICR MS coupled with various soft ionization techniques, such as electrospray ionization (ESI) and atmospheric pressure photoionization (APPI), most polar and non-polar molecules in the asphaltenes could be unambiguously assigned base on the exact molecular mass (Klein et al. 2006; Zhang et al. 2014; Juyal et al. 2010).

In this paper, the crude oil and the sediments from Gaotan-1 were characterized with various analytical techniques. The formation mechanism of sediments from well Gaotan-1 was proposed.

**Experimental section**

**Samples and reagents**

Sediments and crude oil were obtained from oil well Gaotan-1 in the Sikeshu Sag, Junggar Basin, Xinjiang, China. The crude oil was a black liquid with density of 0.824 4 g/cm³, viscosity of 4.2 mPa s at 20 °C. The oil and gas in oil well Gaotan-1 were from coal-derived source rocks in the Jurassic, and the associated gas was with gas wetness of more than 5 (Du et al. 2019; Zhang et al. 2020). Daily oil and gas production of 1213 and 32.17 × 10⁴ m³/day were reported (Du et al. 2019; Zhang et al. 2020). Totally nineteen sediments were collected from the wellbore of Gaotan-1. One of them was obtained from the first time pipeline pigging, labeled as S-0; other 18 samples were sampled from the oil well tubes of the second time pipeline pigging, labeled as S-1 to S-18. The depth of samples was different but could not provide the accurate values. The depth of S-2, S-3, S-4 was the same with increasing radii in the pipeline. The depth of S-14, S-15 was the same with increasing radii in the pipeline. S-2 and S14 were in the center of the pipeline, S-3 was in the halfway around the radius of the pipeline, S-4 and S 15 were in the edge. Dichloromethane and methanol were analytical grade and purified by distillation before use. Ammonium hydroxide (28 wt% solution of NH₃ in water) were purchased from Sigma-Aldrich.

**Analysis process for the sediment**

Sediments were dried at 80 °C in a vacuum drying oven and extracted with dichloromethane and methanol (93:7, v/v) for 48 h by Soxhlet extraction. Solvent was removed by rotary evaporation. The extracts and the crude oil were subjected to elemental analysis, Saturates/Aromatics/Resins/Asphaltenes (SARA) fractionation, gas chromatography (GC), high temperature GC (HTGC), ESI/APPI FT-ICR MS. Eighteen sediment samples, S-1 to S-18 with different unknown depth from well Gaotan-1 were analyzed in the same analysis process.

**FT-ICR MS analysis**

Molecular characterization was subjected to a Bruker apex-ultra FT-ICR MS, which was equipped with a 9.4 T actively shielded superconducting magnet. The organic extracts and crude oil were dissolved in toluene to a concentration of 10 mg/mL, which was further diluted with toluene:methanol(1:3, v/v) to 0.2 mg/mL for negative-ion ESI. Each sample was fortified with 20 μL of NH₄OH. Each sample solution was infused into an Apollo II electrospray ion source at a flow rate of 250 μL/h, which was properly implemented by a syringe pump.

The operating conditions of the negative-ion mode (-ESI) were as follows: The spray shield voltage, 4.0 kV; capillary column front and end voltages, 4.5 kV and -320 V. The ions were accumulated 1 s and the delay was set to 1.1 ms to transfer the ions to the ICR cell by the electrostatic focusing of transfer optics. The RF excitation was attenuated at 13.50 dB. The mass range was m/z 150 − 800. The data size was 4 M words. A total of 64 scans were accumulated to enhance the signal-to-noise ratio and dynamic range. The data processing has been described elsewhere (Shi et al. 2012).

**Results and discussion**

**Extract yields, elemental analysis and SARA composition of S-0**

The extract yield of S-0 was 80.52 wt%. This indicated the main component of sediment was organic compounds. The inorganic component was silty sand (Li et al. 2020). The content of asphaltenes in the extract was 62.3 wt%, which is superhigh when compared with the value of 1.8 wt% in the crude oil. Colloidal instability index (CII) (Ashoori et al. 2017) was 1.7 and 2.4, which
were far more than 0.9, indicating they were very unstable. The relatively low recovery of crude oil was caused by the loss of light hydrocarbons in the solvent evaporation. The crude oil was analyzed by GC and HTGC and sediment extract was analyzed by HTGC to check whether long alkyl paraffins exits in the asphaltenes. GC and HTGC chromatograms are shown in Fig. 1. No long alkyl paraffins were found in the crude oil or sediment extract. The main component of crude oil was normal alkanes with carbon numbers of C5–C30. Content of small molecule compounds (<C10) was very high (more than 42%). The crude oil had geochemical parameters of Pr/Ph, Pr/nC17, and Ph/nC18 with values of 4.2, 0.2, and 0.05, respectively. According to the geochemical characteristics of the crude oil, the kerogen is humic in an oxidation environment and has the typical characteristics of Jurassic hydrocarbon source rocks (Du et al. 2019; Zhang et al. 2020). The HTGC chromatogram revealed that no large molecular normal alkanes in the crude oil.

Elemental analysis of the crude oil, sediment extract is shown in Table 2. The H/C ratios of the crude oil and the extract were 1.93 and 1.04, respectively. The H/C ratio of the extract was far lower than that of the crude oil. The low value of H/C of the extract indicates that the main organic compounds having high degree of condensation.

### Molecular composition of the crude oil and the extract

The crude oil and the extract were subjected to -ESI FT-ICR MS to investigate the molecular compositions. Broad

### Table 1 SARA fractionation results (wt%) of crude oil and sediment extract of S-0

| Sample          | Saturates (wt%) | Aromatics (wt%) | Resins (wt%) | Asphaltenes (wt%) | Recovery (wt%) |
|-----------------|-----------------|-----------------|--------------|-------------------|---------------|
| Crude oil       | 51.5            | 10.1            | 21.6         | 1.8               | 84.9          |
| Sediment extract| 6.0             | 2.7             | 25.7         | 62.3              | 96.7          |

### Table 2 Elemental analysis of the crude oil and the extract of S-0

| Sample          | C (wt%) | H (wt%) | O (wt%) | N (wt%) | S (wt%) | H/C  |
|-----------------|---------|---------|---------|---------|---------|------|
| Crude oil       | 86.39   | 13.89   | 0.69    | 0.06    | 0.04    | 1.93 |
| Sediment extract| 87.84   | 7.65    | 3.23    | 0.70    | 0.39    | 1.04 |
band and mass-scale expanded -ESI FT-ICR mass spectra of the crude oil and the extract are shown in Fig. 2. The mass spectra ranged from m/z 250 to 700, centered at around m/z 450. The odd-mass heteroatom species with a high relative abundance in crude oil were O1 class species (has one and only one oxygen atom in the molecules) which corresponding to alkyl phenols. The abundant peaks around m/z 325 in the extract were identified as alkylbenzene sulfonate surfactant, which should from the contamination of the pipeline pigging. The C16 and C18 fatty acids at m/z 255, 283 were likely contaminants in the negative-ion ESI analysis (Zhang et al. 2011). The expanded mass scale spectra at m/z 374 showed the distinct compositional difference between the crude oil and the extract. The relatively high abundance peaks were O1 and N1 species for the crude oil and N1O1 and N1 species for the extract.

Relative abundance of heteroatom classes assigned from the negative-ion ESI FT-ICR mass spectra of Gaotan-1 crude oil and the extract are shown in Fig. 3. The abundant heteroatom compounds in the crude oil were O1, N1 species with relatively low double bond equivalence (DBE) values. The abundant heteroatom compounds in the extract were N1, O2, N1O1, O1, O3, O4 species with relatively high DBE values. These compound are usually found in source rock extracts (Pan et al. 2018; Ke and Xu 2018) and not abundant in crude oils.

Ion relative abundance distribution of DBE versus carbon number of N1 and O1 class species assigned from the negative-ion ESI FT-ICR mass spectra of Gaotan-1 crude oil and the extract of S-0 are shown in Fig. 4. The relatively high abundance peaks of N1 species in crude oil were carbazoles with DBE of 9 and carbon number ranged from 18 to 50. Besides, carbazoles with DBE of 12–26 and carbon number ranged from 18 to 50 also showed relatively high abundance. Compared with the crude oil, N1 species in the extract had apparently higher DBE values, most of them was more than 15 up to 30, which are distinctly higher than that in regular crude oils (Zhang et al. 2011, 2010, 2016, 2019; Shi et al. 2010; Wang et al. 2013). The O1 class species showed similar variations in the distributions of DBE value and carbon number. The degree of condensation of polar heteroatom species in the extract was higher than that in ordinary crude oils.

Fig. 2 Negative-ion ESI FT-ICR mass spectra of Gaotan-1 crude oil and the extract of S-0. Broad band (top) and mass-scale expanded (bottom). The presented structures were proposed by the molecular composition

Fig. 3 Relative abundance of heteroatom classes assigned from the negative-ion ESI FT-ICR mass spectra of Gaotan-1 crude oil and the extract of S-0.
Composition of sediments from different depth

Composition of a series of sediments and their extracts, S-1 to S-18, is shown in Table 3. The total water content of the 8 arbitrarily selected sediments ranged from 8 to 28 wt%. The percent content of organic extract ranged from 20 wt% to 84 wt%, indicating the great difference between the sediments with different depth, even for the same depth but with different radius. All of the samples contained asphaltenes with the range of 45–89 wt%, much higher than that of normal crude oils. Molecular composition of the 18 sediments were very similar with almost the same heteroatom classes and plots of DBE versus carbon number. All the sediments were seriously contaminated by alkylbenzene sulfonate surfactant. In general, with the decreasing of H/C, the content of asphaltenes increased. S-14 had low content of asphaltenes, high H/C, indicating it contains much light crude oil. S-6, S-9, S-12, S-13, S-16 had high content of asphaltenes, low H/C, indicating they contain much asphaltenes. The content of asphaltenes has good negative linearity with H/C as shown in Fig. 5. The difference in H/C ratio can be explained as the difference in oil content. The content of oxygen and nitrogen differ slightly, with the average value of 2.77 wt% and 0.60 wt%

Formation mechanism of asphaltene deposition in well Gaotan-1

The Gaotan-1 oil was normal mature oil well preserved from thermal cracking despite its huge burial depth. The associated gas was coal-type gas with relatively high wetness (gas wetness > 5). Content of small molecule compounds (< C_{10}) was very high (more than 42%). The main component of sediments was asphaltene with H/C of 1.04, indicating a high degree of condensation. Due to the single-phase liquid fluid under the formation conditions of the Gaotan-1 well oil reservoir, the formation is abnormally high pressure and high temperature, the formation energy is sufficient, and the light component has a strong ability to dissolve the high condensation degree asphaltene component, which is easy to carry the asphaltene from the formation into the wellbore.

Based on the above analysis, it is believed that the formation of sediments in Gaotan-1 well is related to the particularity of the reservoir. Highly condensed asphaltenes generated at the high temperature is the material basis for the formation of deposition. Under the reservoir condition of high temperature of 134 °C and high pressure of 133 MPa, petroleum fluid should be in a supercritical state which having high dissolving capacity for the highly condensed...
molecules. The light components in crude oil are preferentially separated and flow out, which destroyed the dynamic stability of the crude oil. The highly condensed molecules (asphaltene) were extracted from the reservoir rock and deposited on the wall of the wellbore as the temperature and pressure dropped along the well (Li et al. 2020). Sand particles in the fluid promoted the deposition. The key factors are the high maturity of oil, the wetness of the associated gas, the high pressure and the high temperature. The supercritical fluid theory connects molecular composition of asphaltene with the maturity of oil, the wetness of associated gas, the high pressure and the high temperature. In addition, it’s consistent with empirical temperature and pressure experience. It has been used to evaluate the risk of asphaltene deposition for other wells under high temperature and high pressure. The results will be discussed in detail in the further work.

Table 3 Composition of the sediments and their extracts from well Gaotan-1

| ID  | Water (wt%) | Extract (wt%) | Asphaltenes (wt%) | Oxygen (wt%) | Nitrogen (wt%) | H/C  |
|-----|-------------|---------------|-------------------|--------------|----------------|------|
| S-1 | 18.6        | 20.88         | 49.6              | 3.04         | 0.58           | 1.45 |
| S-2 | 14.5        | 23.00         | 54.5              | 2.72         | 0.56           | 1.42 |
| S-3 | 16.0        | 25.82         | 50.5              | 2.88         | 0.60           | 1.34 |
| S-4 | 22.3        | 28.48         | 65.4              | 2.19         | 0.59           | 1.19 |
| S-5 | 10.3        | 46.24         | 64.2              | 2.67         | 0.55           | 1.28 |
| S-6 | 27.8        | 84.17         | 82.3              | 2.71         | 0.66           | 1.04 |
| S-7 | 8.2         | 36.21         | 61.6              | 2.87         | 0.54           | 1.33 |
| S-8 | 23.4        | 75.48         | 67.9              | 3.32         | 0.58           | 1.15 |
| S-9 | –           | 83.80         | 89.5              | 2.58         | 0.64           | 1.09 |
| S-10| –           | 77.96         | 82.0              | 2.71         | 0.67           | 1.05 |
| S-11| –           | 48.26         | 70.4              | 3.03         | 0.65           | 1.13 |
| S-12| –           | 74.50         | 71.6              | 2.55         | 0.63           | 1.11 |
| S-13| –           | 72.23         | 81.8              | 2.62         | 0.65           | 1.06 |
| S-14| –           | 73.74         | 45.9              | 3.78         | 0.67           | 1.53 |
| S-15| –           | 75.65         | 71.8              | 2.31         | 0.60           | 1.19 |
| S-16| –           | 74.78         | 82.3              | 2.57         | 0.64           | 1.08 |
| S-17| –           | 29.38         | 57.2              | 2.76         | 0.53           | 1.34 |
| S-18| –           | 79.85         | 59.7              | 2.46         | 0.54           | 1.26 |

Fig. 5 Plot for contents of asphaltene versus H/C ratios

It’s necessary to choose polar asphalt dispersants to increase the stability of the crude oil system, and use polar reagents for chemical cleaning of the scaled wellbore. More bottom hole filters are required to reduce solid particles. Moreover, it helps recognize supercritical carbon dioxide EOR flooding mechanism and prevent asphaltene deposition.

Conclusions

The crude oil and the sediments from Gaotan-1 were characterized with various analytical techniques. Asphaltenes is considered as the main organic component of sediment, excluding the possibility of paraffins. The low value of H/C for sediment extract indicates that the main organic components having high degree of condensation. The abundant heteroatom compounds with relatively high DBE values and polarity in sediment extract are usually found in source rock extracts, which are hardly exploited from the stratum. There are distinct difference in the content of asphaltenes between the sediments with different depth, even for the same depth but with different radius. However, molecular composition of the 18 sediments were very similar. In general, with the decreasing of H/C, the content of asphaltenes increased. The content of asphaltenes has good negative linearity with H/C. The difference in H/C ratio can be explained as the difference in oil content. Based on the molecular characterization of the sediments and the background information of well Gaotan-1, formation mechanism of asphaltene deposition was proposed. The formation of sediments in Gaotan-1 well
is related to the particularity of the reservoir. Highly condensed asphaltenes generated at the high temperature is the material basis for the formation of deposition. Under the reservoir condition of high temperature and high pressure, and high gas-oil ratio, petroleum fluid exists in the form of supercritical state. The light components in crude oil are preferentially separated and flow out, which destroyed the dynamic stability of the crude oil. Due to its super-dissolving ability, the high-condensation hydrocarbons and asphaltenes are brought out of the reservoir, and deposit gradually mixed with silty sand on the wall of the wellbore as the temperature and pressure drop in the process of oil exploitation. The supercritical fluid theory for the formation mechanism of asphaltene deposition has been used to evaluate the risk of asphaltene deposition for other wells under high temperature and high pressure, and helps recognize supercritical carbon dioxide EOR flooding mechanism and prevent asphaltene deposition.

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Conflict of interest There are no conflicts of interest.

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