



Short communication

Characteristics of a crude oil composition and its in situ waxing inhibition behavior



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ABSTRACT

A crude oil containing high paraffin from Yanchang Oilfield was studied for the situ waxing inhibition as an example in this work. The components were separated using silica gel column chromatography by different eluent and were named A1 to A14, and the characteristics of each component and their influence on wax crystallization were studied. It was found that the separated components could interact with each other to decrease the temperature of the wax point to 0.2–1.64 °C, but the effect on the amount of wax was relatively small. Polarized micrographs showed that the components also affect the morphology and distribution of the wax crystals. For example, the component A5 make the wax crystals of A1 larger and make the distribution of wax crystals in a certain direction and more dispersed.

1. Introduction

Crude oil, a complex multi-component homogeneous organic mixture, consists essentially of saturated hydrocarbons, aromatic hydrocarbons, resins, asphaltenes and heterofunctionalized organic compounds containing nitrogen, sulfur and oxygen [1]. Some compounds extracted from crude oil have been investigated at the molecular level. For example, the aromatic hydrocarbon fractions generally includes a range of very low polar heterocompounds, such as benzothiophenes, dibenzothiophenes, and dibenzofurans [2,3]. Individual nitrogen, sulfur and oxygen compound groups occurring in the resin and asphaltene fractions have porphyrins and carboxylic acids [4,5]. However, few researches have been conducted on the interaction between crude oil components, especially the effect of crude oil components on the crystallization of crude oil waxes.

Waxes are a complex mixture of solid (at ambient temperature) hydrocarbons which consist mainly of paraffin hydrocarbons with a small amount of naphthenic and aromatic hydrocarbons as well as polar compounds [6]. The composition of the crude oil varies to varying degrees of the low temperature rheological properties of the crude oil (e.g., pour point, viscosity, etc.). When the temperature decreases to a certain extent, the crude oil will precipitate wax crystals. Wax crystals are combined with each other to form a three-dimensional network structure, the liquid components of crude oil are wrapped, so that the fluidity of crude oil worse or even lost [7]. Resin and asphaltenes are

the average relative molecular mass of the largest and the most polar components in crude oil, the hydrogen bonds between molecules of resin and asphaltenes make the crude oil viscosity high [8], which is the extraction of crude oil and transportation more difficult. Asphaltenes are the heaviest and polar compounds in crude oils with the highest aromaticity and polarity, which consist of fused polynuclear aromatic systems, heteroatoms (S, N, and O), alkyl chains, and heavy metals (e.g., Ni, V). Asphaltenes exist as three-dimensional aggregates due to the strong intermolecular forces such as hydrogen bonding and π -bonding [9] and even at very low concentration as 50–150 mg/L in a good solvent such as toluene [10]. However, resin and asphaltenes are natural surfactants that can be adsorbed on the surface of wax crystals to prevent the growth of wax crystals [11].

The interaction between crude oil components is of great significance to the exploitation and transportation of crude oil. If the relationship between the crude oil components was studied and make clear, the producer can predict the flow of crude oil and the wax precipitation, which can avoid the poor fluidity of crude oil problems and make timely preventive measures.

In this work, the crude oil components were separated by column chromatography, and the main structures of the crude oil were analyzed by UV spectrophotometry and IR spectrophotometry. In addition, differential scanning calorimetry and polarized microscopy were used to study the effect on the wax precipitation, which may be useful to predetermines the change of viscosity and the wax precipitation.

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Table 1
The physical parameters of crude oil from Yanchang Oilfield.

Pour point (°C)	ρ^{20} (g·cm ⁻³)	Saturated HC (%)	Aromatic HC (%)	Resin (%)	Asphaltene (%)
28.2	0.836	70.2	19.7	9.7	0.4

2. Experimental

2.1. Materials

Petroleum ether, ethyl acetate, methanol, trichloromethane were purchased from Xi'an Chemical Reagent Co., Ltd (at AR reagent grade) and were used without further purification. The crude oil sample was obtained from Yanchang Oilfield with no suspended sand particles. The physical parameters of this crude oil was shown in Table 1.

2.2. Thermogravimetric analysis conditions

The TGA (Thermogravimetric Analysis) was carried out using a TGA/SDTA851 instrument over the temperature range of 35–600 °C, at a heating rate of 20 °C/min, nitrogen flow 20 mL/min and other parameters. The quality of the test oil samples ranges from 3 to 6 mg.

2.3. Separation of crude oil components

10 g of the crude oil weighed accurately, dissolved in chloroform and fully adsorbed on the quality of 150–200 mesh silica gel, and then drying at the appropriate temperature. The prepared crude oil sample was loaded into 200 g of the upper layer of the column chromatography silica gel. They were treated with petroleum ether, a mixture of petroleum ether and ethyl acetate, a mixture of chloroform and methanol, and methanol successively. The eluates were collected respectively, the solvent was evaporated, and the corresponding fractions were obtained. The components' names and their contents were shown in Table 2. Due to the strong polar components of crude oil adsorption on the silica gel, the final eluted part is less than 100%.

2.4. IR analysis

After the separation of the crude oil by the column separation method, some of the components in the crude oil were deposited or

Table 2
Components and contents of the crude oil.

Eluents	Labels of component	w/%
Petroleum ether	A1	65.01
Petroleum ether: Ethyl acetate (volume ratio 100:1)	A2	8.42
Petroleum ether: Ethyl acetate (volume ratio 50:1)	A3	4.26
Petroleum ether: Ethyl acetate (volume ratio 30:1)	A4	2.18
Petroleum ether: Ethyl acetate (volume ratio 20:1)	A5	1.39
Petroleum ether: Ethyl acetate (volume ratio 15:1)	A6	1.39
Petroleum ether: Ethyl acetate (volume ratio 10:1)	A7	2.38
Petroleum ether: Ethyl acetate (volume ratio 3:1)	A8	1.59
Petroleum ether: Ethyl acetate (volume ratio 2:1)	A9	0.79
Petroleum ether: Ethyl acetate (volume ratio 1:1)	A10	2.97
Methanol: Trichloromethane (volume ratio 30:1)	A11	1.29
Methanol: Trichloromethane (volume ratio 10:1)	A12	1.39
Methanol: Trichloromethane (volume ratio 5:1)	A13	0.40
Methanol	A14	0.50

adsorbed on the surface of the silica gel. The silica gel was dried, grinded and then analyzed by infrared spectroscopy. FTIR spectra were recorded in KBr pellets with a Bruker Tensor 37 spectrometer in the 400–4000 cm⁻¹ region. FTIR spectrometer was calibrated by 1.5 mil and 55 μm thick polystyrene (PIKE Technologies) as reference standard before the analysis.

2.5. UV analysis of the crude oil components

The separated crude oil constituents were solve in trichloromethane respectively at a concentration of 10 mg/L. Electronic spectra were recorded in the range 200–400 nm, with spectral on as standard, on a UV-2600 spectrophotometer.

2.6. Differential scanning calorimetry analysis

The differential scanning calorimetry (DSC) analysis of the samples are performed using a Mettler-Toledo DSC822e DSC apparatus. The chemicals, suppliers and purities used for the DSC calibration were: *n*-C7 (Scharlau, 99%), *n*-C8 (Panreac, 99%), *n*-C12 (Alfa-Aesar, > 99%), *n*-C16 (Alfa-Aesar, > 99%), *n*-C18 (Fluka, P99.8%) and Indium (Mettler Toledo). Repeatability and reproducibility tests shows that this calorimeter is capable of producing data of enthalpies of fusion with an uncertainty of 1.5%, temperatures of fusion between 0.1 and 0.2 K, and heat capacities with an uncertainty of 1.5%, all at a 95% confidence level. The temperature profile follows two steps: (1) Previous step: Sample is heated at 3 °C/min from room temperature to 40 °C to completely dissolve possible solid phase and to remove any thermal history; (2) Cooling step: Sample is cooled down from 40 °C to –20 °C at 3 °C/min.

2.7. Optical microscopy analysis

The saturated hydrocarbon component was separated from the crude oil using the standard method for the optical study. Wax crystal morphologies were observed using a BX41-P OLYMPUS polarizing microscope. Samples were initially heated to 50 °C and then cooled to 10 °C for 5 min. A small amount of wax crystal was loaded onto the glass slide inside a copper stage with a central window. During the measurement, the temperature of the copper stage was controlled at 10 °C in a circulating bath.

3. Results and discussion

3.1. Thermogravimetric analysis of crude oils

In order to understand the content of the crude oil, thermogravimetric analysis was carried out. The plots of mass fraction (W/%) versus the temperature (T/°C) yield curve as shown in the Fig. 1. As can be seen from the data in the Fig. 1, from 35 to 500 °C, the oil sample is continuously evaporated to lose weight, until the temperature reaches to 500 °C, it almost becomes a horizontal line, only 0.1% residue left. When the temperature rises from 40 °C to 200 °C, the weight loss rate of the crude oil reaches 44.28%, which corresponds to the petrol ingredients of the crude oil. As the temperature ranges from 150 °C to 350 °C, the weight loss rate is 61.04%, which corresponds to the diesel ingredients [12]. The TGA result shows that this crude oil is a good feedstock for oil refining.

3.2. IR analysis

After the separation of the crude oil in 2.2, the silica gel turned from white to gray, because some of the components in the crude oil were deposited or adsorbed on the surface of the silica gel. The silica gel with the sediments were dried and then analyzed by infrared spectroscopy (IR), and it was shown in Fig. 2. As can be seen, the absorption at

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