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Evaluation of Crude Oil Rheology as a Comprehensive Experimental for the Applied Chemistry Education

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Abstract: In order to further improve the comprehensive experimental skills of applied chemistry undergraduates, a comprehensive experiment of crude oil viscosity, yield value, viscosity temperature curve and viscosity reducer evaluation was designed. Through the determination of different physical parameters of crude oil and the evaluation of viscosity reducer, the characteristics of crude oil and the effect of viscosity reducer are comprehensively evaluated. The design of the experiment has significant pertinence and applicability, effectively deepening students' understanding of some physical properties of crude oil, and further improving students' experimental operation ability, experimental observation ability and problem solving skills. At the same time, the determination of some physical parameters of crude oil makes students realize that physical parameters are indispensable for guiding further development work in the oil fields and gas fields, and at the same time deepen the study of the principle of measuring some physical parameters.

1. Introduction

As the world's demand for oil grows, in order to increase oil recovery, miners must first understand some characteristics of crude oil. China's crude oil resources are abundant, and heavy oils with high viscosity and high freezing point often appear during the extraction process [1]. Therefore, it is particularly important to determine the freezing point and viscosity of crude oil. The rheological data of crude oil can be obtained by measuring the viscosity, which has important guiding value for the process control, transportability of predicted crude oil extraction and transportation, and the operability of crude oil during processing [2-5]. The yield value is the minimum pressure at which the oil-gelling system can flow, and it is the basis for calculating the restart pressure after stopping. During the transportation of waxy crude oil, when faults occurred or pre-stop to transport, as the temperature of the pipeline decreases, wax crystals form a network structure with colloids and asphaltenes in crude oil, showing the characteristics of yield flow [6, 7]. The viscosity-temperature curve has important guiding significance in the research of crude oil heat treatment and the addition of pour point depressant treatment. The viscosity-temperature curve can be used to evaluate the treatment effect; calculate the pressure drop of the pipeline according to the laboratory simulation experiment data, and preselect the process operation plan; foresee process operating conditions by experimental data. Viscosity reducing agents are often added during the process of crude oil extraction and transportation. Viscosity reducing agents can decrease the freezing point of crude oil and change the morphology of wax crystals in crude oil to reduce the viscosity of crude oil. It is particularly important to evaluate the viscosity reducing agent to determine the viscosity reducing

effect of the viscosity reducing agent [8]. Mastering the determination of physical parameters of crude oil is a basic professional knowledge that students of applied chemistry in petroleum colleges must understand and master. Students are required to have in-depth understanding and mastery of experimental principles and experimental processes, and effectively improve students' independent thinking ability, expand thinking ability, practical operation ability, data processing ability, and problem solving ability [9].

Comprehensive experimental design includes a large number of basic professional knowledge that students have learned, and pay attention to the interconnection between these basic knowledge, so that the overall function can be maximized. Students should complete the teaching tasks of comprehensive design experiments according to the requirements, deepen their understanding of theoretical knowledge, and cultivate students' scientific experiments and practical hands-on skills . This research group designed many comprehensive experiments based on the scientific research results of this specialty for many years [10-14]. The experimental design required 4 hours to complete the determination of the physical parameters of the crude oil and the evaluation of the viscosity reducer performance. It is mainly through the operation of different instruments to deepen the understanding of what has been learned. The design of this experiment can not only consolidate the basic knowledge of students, but also enable students to consult data, design experiments, operate experiments, organize data, and write summary reports of experiments to obtain systematic training.

2. Design of the Experiment

2.1. Purpose

(1) Understand the basic properties of crude oil viscosity;
(2) Master the method of measuring the viscosity and yield value of crude oil;
(3) Master the drawing of viscosity-temperature curve;
(4) Understand the screening and evaluation methods of viscosity reducing agents.

2.2. Source of Viscosity

The composition of crude oil can be divided into four components: saturated hydrocarbons, aromatic hydrocarbons, gums and asphaltenes. At lower temperatures, the molecular weight of saturated hydrocarbons is smaller, and the friction between molecules is smaller. As the temperature increases, saturated hydrocarbons affect the viscosity of crude oil. The effect of viscosity is getting smaller and smaller, and even negligible, the contribution of colloids and asphaltenes to the viscosity of crude oil increases. In addition, colloids can be converted to asphaltenes, and the interaction between macromolecules forms a network structure, which “fixes” oil droplets. In the grid, the crude fluidity is reduced and the viscosity is increased [15-17]. In addition, temperature and pressure also affect the viscosity of crude oil. As the temperature increases, the viscosity of crude oil decreases. The sensitivity of various crude oils to temperature is different. For some crude oils, when the temperature is increased by 10℃, the viscosity of the crude oil is reduced by approximately half. When the pressure is higher than the saturation pressure, the increase in pressure causes the elastic compression of the formation oil, the density of the oil increases, the friction resistance between the liquid layers increases, and the viscosity of the crude oil increases accordingly. When the formation pressure is lower than the saturation pressure, as the formation pressure decreases, the dissolved gas in the oil is continuously separated, and the viscosity of formation crude oil increases sharply [18].

2.3. Determination of Viscosity [19]

At a defined shear rate, the viscosity of the sample is proportional to the shear stress. A rotational viscometer consists of a fixed measuring cylinder and a rotating cylinder (rotor). The resistance of the sample to the rotation of the rotor causes a torsion between the torsion bar between the motor and the drive shaft. The deflection is measured with an electronic sensor, and then processed into data and displayed. Expressed as a formula:

$$\eta = \frac{\tau}{D} = \frac{AS}{M\mathrm{n}} \qquad (1)$$

In the formula:
η——dynamic viscosity, mPa·s;
τ——shear stress, mPa;
D——Shear rate, s^{-1};
A——shear stress factor, which is a constant for a specific sensor system and its viscometer;
S——the signal of the deflection of the torque rod displayed on the viscometer indicator;
M——Shear speed factor, it depends on the ratio of measuring cylinder (cup) and rotor, s^{-1} scale;
N——rotor speed, min^{-1}.

2.4. Yield Value [20]

When the crude oil is lower than a certain temperature, a wax crystal network structure is formed and has a certain strength. In the measurement, the crude oil sample is loaded into the cylinder system of the rotational viscometer, and the temperature is reduced to a certain temperature at a certain cooling rate, and the temperature is kept constant for a certain time to form a network structure. The appropriate shear rate file is used to cut to the sample, the shear stress value τ_y immediately after the rotating cylinder rotated is defined as the yield value of the crude oil sample under this condition. According to the measuring principle of the rotational viscometer, the yield value can be calculated according to formula (2):

$$\tau_y = Z \bullet \alpha \tag{2}$$

In the formula:
τ_y -the yield value of the sample under the measurement conditions, Pa;
Z——Instrument display system constant, Pa;
α ——Indicated value of the instrument.

2.5. Determination of Viscosity Temperature Curve [21]

Rotary viscosity is used to measure the viscosity of crude oil at different temperatures. Viscosity temperature curves are plotted on semi-logarithmic graph paper, and abnormal points are marked.

2.6. Evaluation of Viscosity Reducing Agents

The viscosity-reducing agent is added to the oil sample under demand, and viscosity reducing effect is evaluated by measuring viscosity and freezing point.

3. Materials and Instruments

3.1. Instrument Used for Freezing Point

Round-bottom glass test tube, round-bottom glass sleeve, mercury thermometer and common thermometer (range -30-20 ℃ and -30-100 ℃, graduation value 1 ℃), cooling bath, cooling liquid, refrigerating instrument, holder, ground bottle, Constant temperature water bath.

3.2. Instrument Used for Density

Density measuring cylinder, density meter (in Figure 1), constant temperature bath, thermometer, plastic or glass density measuring cylinder ring rod.

3.3. Instrument Used for Viscosity

RV20 rotary viscometer (in Figure 1), measuring cylinder (D100 / 300 measuring cylinder working temperature 0-300 ℃, working pressure -10.0MPa, viscosity range 10-104 mPa·s; open cup measuring cylinder, working pressure: normal pressure, working temperature below 100 °C, the measurement range is 10-105 mPa·s). Constant temperature circulating oil bath (working temperature -100 ℃, accuracy ± 0.1 ℃), high pressure sample reservoir, high pressure constant speed pump (displacement 0-60mL / min, working pressure 0.0-15.0 MPa).

Figure 1 RV20 rotary viscometer, electronic scales and density meter

3.4. Instrument Used for Yield Value

Rotary viscometer with shear rate as control variable (measurement accuracy is not greater than 3%, constant temperature circulator, temperature fluctuation between ± 0.1 ℃), thermometer (mercury thermometer or other thermometer should not be greater than 0.1 ℃), Balance (the amount of induction is not greater than 1 g).

3.5. Instrument Used for Wax Analysis

Rotary viscometer, thermostat, thermometer, computer.

3.6. Viscosity Curve

Semi-logarithmic coordinate paper, ruler, pencil.

3.7. Instrument Used for Viscosity Reducing Agent Evaluation

Constant temperature water bath, viscometer, circulating water bath, electronic balance, beaker (5 L, 1 L, 500 mL, 250 mL), plastic cup.

3.8. Experimental Materials

Oil sample, viscosity reducer.

4. Experimental

4.1. Preliminary Treatment of Oil Samples

The oil sample is first filtered and then dehydrated. When filtering, a filter with a diameter of 0.043mm is used. The temperature during filtering is less than 100 °C. The dehydration is performed by a high-temperature dehydrator or centrifugal dehydration 0.5% qualified.

4.2. Determination of the Freezing Point [22]

(1) Put the oil container into the water bath for proper heating, then fill the oil samples into several ground-mouth bottles, and place the ground-mouth bottles in the water-bath to heat 50 °C ± 1 °C. Preheating required that the temperature is higher than 50 ℃, the preheating temperature is heated in a water bath, open the refrigerator or cooling water, and leave the oil sample to cool to 50 ℃ ± 1 ℃. Preheating may not be required under certain conditions.

(2) Insert the cork plug with the thermometer into the test tube, adjust the position of the thermometer so that it is in the center, and place the mercury ball 20mm ± 2mm from the bottom of the test tube, then remove the plug for future use.

(3) The required test tube are heated to 50 °C ± 1 °C. If the temperature of the oil sample is lower than the temperature of the test tube, heat it to the same. With the coolant, the freeze point of the oil sample can be estimated in advance, and multiple cooling baths can be prepared. The temperature of the first stage cool bath is set to 25 °C ± 2 °C, and the lower stage is lowered by 15 °C to control the cooling rate of the oil sample at 0.5-1.0 °C / min. The sleeve is installed vertically in the cooling bath, and the depth of the sleeve in the cool bath is not less than 100mm.

(4) Add the oil sample to the circular mark of the test tube, quickly insert the plug with the thermometer into the center of the test tube, and placing the test tube containing the oil sample into the casing containing the cooling bath smoothly and quickly, and let it stand for cooling. The temperature difference between the oil sample and the cooling liquid is between 10-25 °C. If the temperature difference between the oil sample and the cool liquid is less than 10 °C, the oil sample still does not condense, and it should be transferred to the next cooling bath. Transferring the test tube and the case as a whole, and avoid shaking. If you use a refrigerator to cool the oil sample, the cooling temperature should be set between 0.5-1.0 °C / min.

(5) When the sample temperature is higher than the expected temperature of 8 °C, take the test tube out of the casing and tilt it slightly. Observe the signs of liquid level movement of the oil sample. If the liquid surface moves, place the test tube quickly and smoothly into the casing to cool and taking it out. And put into the casing for less than 3s. In the future, observing the oil sample every 2 °C until the liquid level no longer moves, quickly level the test tube for 5s, and record the freezing point if the liquid level does not move. If the liquid level moves, quickly placing the test tube vertically into the sleeve to continue cooling, repeat the above steps until the liquid level no longer moves, and noting the freezing point. Otherwise, replace the oil sample and reducing the expected freezing point by 4 °C and repeat the above steps.

4.3. Determination of Density [23]

(1) Transfer the oil sample to a stable temperature, clean density meter at the experimental temperature to avoid oil sample splashing and air bubble generation, and reducing the volatilization of light components.

(2) Remove all air bubbles from the surface of the oil sample with a piece of clean filter paper.

(3) Place the measuring cylinder filled with oil sample vertically on a place without air flow. During the entire test, the ambient temperature change should not be greater than 2 °C. When the ambient temperature change is greater than ± 2 °C, a constant temperature water bath should be used.

(4) Use a suitable thermometer or glass rod to stir the oil sample in vertical motion. If a resistance thermometer is used to apply the stirring rod, make the entire cylinder density and temperature uniform. Taking out the thermometer or glass rod from the graduated cylinder when the recorded temperature is close to 0.1 °C.

(5) Put a suitable density meter into the oil sample and release it at the equilibrium position. The density meter floats in the oil sample to avoid wetting the dry pipe above the density meter. Press the density meter to the equilibrium point 1-2mm and letting it return to the equilibrium position and observing the meniscus shape. If the meniscus shape changes, cleaning the densitometer stem and repeat this operation until the meniscus shape does not change.

(6) When use a plastic measuring cylinder, wipe the outer wall of the measuring cylinder with a damp cloth to remove static electricity. When measure the oil sample, making the eyes slightly higher than the liquid surface to observe. The density meter reading is the point where the meniscus on the liquid is tangent to the density scale.

(7) After read the density meter, quickly and carefully take out the density meter and stir the oil sample vertically with a thermometer. The recorded temperature is up to 0.1 ℃. If the temperature differs from the starting temperature by 0.5 ℃, the thermometer and density meter should be read again until temperature within ± 0.5 ℃. If the stable temperature cannot be obtained, put the thermometer, graduated cylinder and oil sample into the constant temperature water bath, and read again. After the lead bullet wax seal type density meter is used above 38 ℃, it should be dried and cooled vertically.

4.4. Determination of Viscosity

(1) Fill the processed oil sample into the high pressure sample reservoir, and squeeze the oil sample into the D100/300 measuring cylinder with the pump through the high pressure sample reservoir until the oil sample overflows from the upper part of the measuring cylinder. Pour the oil sample along the cup wall into the open cup to the marked line.

(2) Plug in the power and turn on the constant temperature circulating oil bath, computer, RC20, and RV20. Turn on the power switch, enter the oil sample name and serial number, select the measurement probe, select the rotor, and set the shear rate and temperature.

(3) Each oil sample to be measured is kept at a constant temperature of 20-40 minutes, and the viscosity is measured from low temperature to high temperature. The viscosity test of each oil sample is not less than 6 temperature points, and one is close to the reservoir temperature. Every time a temperature point is measured, a zero point correction is performed and the data is saved to disk.

4.5. Determination of Wax Precipitation Point [24]

(1) The oil sample is heated to 10-15 ° C above the freezing point of the oil sample, the sample is loaded according to the requirements of the instrument measurement system, the thermostat and the rotational viscometer measurement system are connected, and the sample and measurement system are heated to 70 -80 ℃, and constant temperature for 10min.

(2) Control the constant temperature circulator to continuously cool down at a cool rate of 0.2-1 ° C / min, and at the same time start the rotary viscometer to run at a fixed value at a shear speed within the range of 10-600s-1.

(3) Record the corresponding values of shear stress or viscosity and temperature every 1 °C, and draw a semi-logarithmic curve on the computer until the temperature decreases to a significant change in the slope of the curve, and then continue the temperature measurement 5-7 date.

4.6. Yield Value

(1) Fill the oil sample bottle, plugg the stopper with a thermometer, place the thermometer in the center of the oil sample, and then preheat it to the sample temperature in a constant temperature water bath. At the same time, selecting a suitable cylinder system to fit the rotational viscosity according to the characteristics of the oil sample. Connect the constant temperature circulation system to preheat to the sample loading temperature, and the sample loading temperature is 15-20 °C higher than the freezing point or abnormal point.

(2) Calculate the quality of the oil sample at the measurement temperature based on the volume of the measure cylinder and the density of the oil sample. Then remove the outer cylinder and quickly weigh the oil sample and installing it on the viscometer. Control the constant temperature circulation system to select the cooling speed from 0.3-0.5 ℃ / min to the measurement temperature with the accuracy of ± 0.1 ℃.

(3) Manually changing the shear rate of the viscometer to the minimum shear speed. For viscometers with continuous shear rate, select $0.2s^{-1}$ for the shear rate. Start the viscometer and record the shear stress when the rotating cylinder starts to turn. When the cylinder system is not selected properly, it cannot rotate or the stress value is not displayed, repeat the above steps to measure again.

4.7. Draw Viscosity-temperature Curve

(1) Start the rotational viscometer at the selected shear rate, and record the first value until the reading of the instrument is basically stable. Then recording it every 5 minutes. If 4 a values are recorded continuously, the difference between the arithmetic average value of next 3 a values and the previous one is not more than 5%, it is considered to reach an equilibrium value, and the determination of a value at the shear rate is completed.

(2) When the oil sample is a non-Newtonian fluid, at this temperature and shear rate interval, select at least 5 shear rates to measure its apparent viscosity, and its temperature interval is 2 °C.

(3) When the oil sample is Newtonian fluid, the temperature interval is 5 °C.

(4) When the oil sample exhibits Newtonian fluid characteristics, the lowest temperature is determined as the abnormal point.

4.8. Viscosity Reducing Agent Evaluation

(1) Take 25mL oil in multiple measuring cups, and take the viscosity reducing agent to prepare liquid A with a certain concentration;

(2) Add the diluted viscosity reducing agent A to the oil sample, prepare 0, 100, 300, 500, 700, 900

mg/L oil sample to be tested, label it, heat the measuring cup to 50 °C, and stir it well;
(3) Measure the viscosity between its freezing point and 70 °C according to 4.2 and 4.4.

5. Results Processing and Discussion

5.1. Crude Oil Viscosity Determination

To the end of the test, the viscosity data of each pressure and temperature were processed with the RV20 software package.

5.2. Yield Value

(1) Manually changing the shear rate range of viscometer, and the yield value is calculated according to the formula;
(2) When the program computer controlled is used to control the measurement, the shear stress can be directly recorded, and the maximum value is the yield value of crude oil.

5.3. Draw the Viscosity Temperature Curve

(1) On semi-logarithmic coordinate paper, the ordinate is viscosity and the abscissa is temperature. Data points are marked with viscosity values at different temperatures;
(2) Mark abnormal points on the abscissa;
(3) Non-Newtonian fluid characteristic temperature interval data points are connected by a smooth curve, and the corresponding shear rate of the curve is marked;
(4) The data points between the abnormal point and the wax analysis point are connected by a straight line;
(5) The data points between wax analysis point and 80 ℃ are connected by a straight line. When the wax sample points of oil sample cannot be measured, the data points between abnormal point and 80 ℃ are connected by a straight line or smooth curve.

5.4. Viscosity Reducing Agent Evaluation

The viscosity reduction and freezing point of oil samples with reduced viscosity and blank oil samples were compared at low temperature.

6. Questions

(1) What are the factors that affect the viscosity of crude oil?
(2) What's the mechanism of viscosity reduction?
(3) How to use these data to screen and select viscosity reducer?

7. Conclusion

The design of the experiment is applied to the basic knowledge in the textbook "Oil Field Chemistry", which mainly determines the physical parameters of crude oil and evaluates the performance of the viscosity reducer. In the experiment, different instruments were used to measure different physical parameters, and the viscosity-reducing agent was evaluated by the viscosity-reducing rate and the change of the freezing point of the viscosity-reducing agent. On the one hand, students 'experimental operation ability is trained; on the other hand, students' experimental observation ability is improved. And deepen students' understanding of comprehensive chemistry experiments. Comprehensive chemistry experiments are not only a study of students' ability to mechanically complete experiments in accordance with the requirements of the experimental instruction book, but also more importantly through the experimental design of different results produced by different experimental steps and content, Can improve students' creative thinking ability.

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