Evaluation of Flushing Ability of Cementing Flushing Fluids
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ABSTRACT: During cementing operation with oil-based drilling fluids, the casing and well wall are in the “oil wet” environment, and the cement slurry cannot achieve good cementation with the second interface, which seriously affects the cementation quality of the cementing interface. Therefore, it is very important to select a flushing fluid with good performance and high flushing efficiency for cementing operation with an oil-based drilling fluid. An integrated measurement system of interface parameters and a rotating liquid interface tension tester is selected in the room. A set of evaluation methods of wetting reversal is developed to evaluate the wetting reversal ability of type A and type-B flushing fluids on site to optimize the reasonable type and concentration of the flushing fluid. The experimental results show that under the same concentration, the surface tension and interfacial tension of type A flushing solution are lower than those of type-B; that is, the hydrophilic effect of type A flushing solution is better than that of type-B. The wettability reduction of type A is better than that of type-B. The wetting reversal ability of type A flushing solution with a surfactant concentration of 30% is better than that of type-B. The evaluation methods include surface tension, interface tension, dewetting ability, and oil washing ability of the flushing fluid, and the evaluation of the wetting reversal ability is carried out on the basis of the sandstone, sheet metal (steel for three open casings on site), and slide glass (as a reference). Then, according to the optimal concentration of the flushing fluid, a mixing experiment of the two kinds of flushing fluids is carried out to evaluate the ability of wetting reversal, and compared with the experimental results of A and B, the reasonable flushing fluid formula is finally selected. A proper analysis of the adaptability of the evaluation method of the flushing fluid is conducive to the reliable evaluation of the performance of the flushing fluid and the enhancement of the operator’s ability to grasp the cementing quality. To guide the field practice has a very important practical significance, but it also has a guiding role for the development of new pre-liquid products.

1. INTRODUCTION
With the development of exploration and development of deep oil and gas reservoirs and unconventional oil and gas reservoirs, the numbers of complex deep wells and shale gas wells are increasing year by year, and the requirements for drilling fluid performance are much higher.1,2 Among them, oil-based drilling fluids, with their excellent inhibition and temperature resistance, have obvious advantages in drilling complex wells, especially in high-temperature deep wells and water-sensitive formations, which can more effectively protect water-sensitive oil and gas reservoirs and improve oil and gas production.3,4 As early as the 1960s, scholars attached great importance to oil-based drilling fluids. In the 1970s, a series of high-temperature oil-based drilling fluids were developed and successfully applied to the needs of deep and ultradepth wells.5,6 In the Hunt Energy Cerf Ranch 1–9 well in Texas, the oil-based drilling fluid was successfully drilled to 9046 m. Since then, the oil-based drilling fluid has developed rapidly and has gradually become an important means of drilling high-temperature deep wells, high-angle directional wells, horizontal wells, and various complex well sections and for offshore drilling and reservoir protection.6,7

An oil-based drilling fluid has many advantages, such as strong antipollution ability, strong inhibition, stable performance, and adaptability to high temperature and high pressure and other complex formations, but it also brings challenges for later operations. In the cementing stage, it is required to ensure good first and second interface cementation quality to achieve the ideal cementing quality, while the existence of the oil-based drilling fluid will greatly affect the cementation strength of the wall and the cement slurry or even not.8–11 Therefore, it is necessary to clean the oil-based drilling fluid on the well wall and change the wettability of the wall from oil-wet to water-wet before water injection,12–15 which is an important premise to ensure the cementing quality, and this is the problem that the oil-based mud prepositional fluid should focus on. However, how to determine the flushing effect of preflush on the well wall and the wall wettability after flushing before mud injection is a subject that has been studied.16–18

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Flushing fluid is a part of the precementing fluid, mainly composed of clear water, a composite surfactant, a regulator, etc. At present, the most commonly used chemical flushing fluid is a surfactant that can clean and wet the well wall and casing wall by the unique surface activity of the surfactant, such as wetting, emulsifying, penetrating, and dispersing, to ensure the surface well quality by reducing the interfacial tension of the second cementing interface. In this paper, an integrated measurement system of interface parameters and the rotating liquid interfacial tension tester is selected indoors, and a set of wetting reversal evaluation methods is formulated to evaluate the wetting reversal ability of type-A and type-B flushing fluids on site to optimize the reasonable type and concentration of the flushing fluid. The evaluation methods include the evaluation of surface tension, interfacial tension, wetting reduction ability, and oil washing ability of the flushing fluid. The wetting reversal ability is evaluated with on-site sandstone, metal sheet (on-site three split casing steel), and glass slide (as a reference). Then, taking the optimized flushing fluid concentration as the reference, the compound experiment of the two flushing fluids is carried out to evaluate the wetting reversal ability and the wetting reversal effect of the isolation fluid in the oil-based drilling fluid. The contact angle method, Amott method, and USBM method are three commonly used methods for quantitative measurement of wettability. The contact angle method is the most intuitive and simple method to measure the wettability of crude oil and brine on polished minerals, and it is also the best method when it is used for pure fluid. The Amott method and USBM method can measure the average wettability of the core, which is better than the contact angle method when measuring the wettability of an original or complex core; the USBM method is more sensitive than the Amott method when it is close to neutral wettability, but for the system of partial wettability and mixed wettability, the USBM method is unable to measure and the Amott method is more sensitive. The Amott and USBM wettability index methods (improved USBM method) concentrate their advantages. As an alternative method of the Amott method and USBM method, the automatic imbibition method can determine the system that cannot be determined by the Amott method and USBM method. NMR is a simple and rapid method for the quantitative determination of wettability, which can be used to determine the moisture content.

2. EXPERIMENTAL INSTRUMENTS AND EVALUATION METHODS

2.1. Surface Tension Test Method and Principle.

2.1.1. Test Method. The surface tension between gas and liquid is measured by a suspension drop method. The basic principle of surface tension measurement by the drop suspension method is as follows: when the droplet is suspended at the nozzle of a capillary, the shape of the droplet mainly depends on the balance of gravity and surface tension. Therefore, the surface tension of the liquid can be calculated by measuring the shape of the liquid drop; in addition, if the liquid drop is suspended in another immiscible solution, the interfacial tension of the two liquids can also be calculated. In this paper, the surface tension between surfactant solutions of different concentrations and air is measured by the suspension drop method and compared with the surface tension between hard water and air to study the improvement degree of the surfactant on the interfacial tension of gas—liquid two phases.

2.1.2. Test Equipment. In this paper, the surface tension is measured by a KRUSS DSA30S instrument, as shown in Figure 1.

2.1.3. Measurement Steps.

1. Take the water as the reference quantity and respectively configure 10, 15, 20, 25, 30, 35, and 40% concentrations of the rinse solution;
2. Activate the power supply and computer control system of the surface tension tester, take out the microsyringe of the surface tension tester and install it on the tester, adjust the optical lens through the computer so that the syringe needle can be projected into the middle of the display, adjust the lens diaphragm, and adjust the sharpness of the needle;
3. Take off the syringe, draw a certain amount of detergent solution and place it on the tester, push the syringe to push the plug, extrude the solution, and when the droplet is about to drop and the shape is stable, click the surface tension measurement button to display the surface tension results of the droplet;
4. For a solution, carry out measurements for multiple drops of the liquid according to the above experimental steps. The average value is taken as the surface tension value of the solution.
2.2. Test Method and Principle of Interfacial Tension.  
2.2.1. Test Method. The common methods for measuring the interfacial tension of liquid–liquid are the ring removal method, drop volume method, rotating drop method, hanging piece method, bubble pressure method, drop stop method, hanging drop method, etc. In this paper, the rotating drop method is used to measure the interfacial tension of liquid–liquid (surfactant solution and oil).

The basic principle of the rotating droplet method is to fill a rotating sample tube C with a liquid B of high density $\rho$ and add a drop A of low density $\rho$ to suspend it in liquid B, and install it in the sample tube of the rotating droplet instrument in a closed manner. At this time, the sample tube is parallel to the rotating axis D. After starting the machine, take the central axis of the sample tube as the rotating axis and carry the liquid to rotate at an angular velocity $\omega$. Under the action of centrifugal force, gravity, and interfacial tension, a long spherical or cylindrical droplet is formed in the high-density liquid, whose shape is determined by the rotation speed and the interfacial tension between the two liquids. Assuming that droplet A coincides with the axis of rotation after stretching and neglects the influence of gravity and buoyancy, when the angular velocity $\omega$ is large enough, it can be regarded as a cylinder with two semicircle bottoms. Based on the principle that the total energy of droplet A is the smallest when the volume of the droplet is constant, Vonnegut derived the approximate calculation formula of interfacial tension.

2.2.2. Test Equipment. In this paper, the liquid–liquid interfacial tension is measured by a rotary drop interfacial tension tester Tx-500c, as shown in Figure 2. The Tx-500c meter/interface tensiometer is controlled and measured by a computer control system.

2.2.3. Measurement Procedure.

1. Prepare 20 mL of white oil and then prepare 10, 15, 20, 25, 30, 35, and 40% of rinse solution with water as the reference volume;
2. The interfacial tension means measuring the interfacial tension between the liquid phase and the incompatible liquid phase; in this experiment, the surfactant solution of different concentrations and white oil are used as the measurement objects;
3. Use the small volumetric flask of the interfacial tension tester and fill a certain surfactant solution in it, then place the volumetric flask horizontally, inject 1–2 drops of oil into the small volumetric flask through a syringe, place the volumetric flask in the interfacial tension tester, at room temperature of 25 °C, rotate for 20 min at a rotating speed of 5000 rpm, adjust the angle of the volumetric flask until the oil drops appear in the display, take photos to measure the surface tension of the oil drops, and measure the oil drops in the forward and reverse directions four times, and take the average value;
4. According to the above steps, measure the interfacial tension between surfactant solution of different concentrations and white oil, for a total of seven groups.

2.3. Test Method and Principle of Wetting Contact Angle. 2.3.1. Test Method. In this paper, direct measurement of the contact angle is used to study the improvement of the wettability of the solid surface. After the glass slide, metal sheet (simulated downhole casing), and core (simulated borehole wall) are treated by lipophilic treatment, an oil film is formed on the solid surface, a drop of the surfactant solution of a certain concentration is dropped on the solid surface, and the projection is magnified on the display by an optical instrument, and the contact angle of the scale calculator is used.

2.3.2. Test Equipment. In this paper, the measuring equipment is the same as the surface tension measuring equipment, i.e., the contact angle is measured by the KRUS DSA30S instrument. Its main performance parameters refer to the above surface tension measuring equipment.

2.3.3. Measurement Procedure. To better understand the change of wetting angle of the surfactant solution after oil washing, the measurement of the wetting contact angle is divided into the following two aspects:

1. Prepare 10, 15, 20, 25, 30, 35, and 40% of rinse solution based on water;
2. Wash the slide with hard water for 3 min, put it into a constant-temperature drying oven at 90 °C for drying, then put it into a carbon tetrachloride soaking oven for 30 min, and then put it into a 90 °C drying oven for drying;
3. After drying, the hydrophilic slides were prepared by soaking in ethanol for 30 min;
4. The hydrophilic slides were immersed in oil for 24 h and then dried in a constant-temperature drying oven at 90 °C, so that the hydrophilic slides were prepared;
5. Inject a certain concentration of detergent solution into the microsyringe of the wetting angle tester and drop it on the surface of the processed slide;
6. Pat the slide after the shape of the drop on the slide no longer changes and take an image and manually adjust the baseline position;
7. When the shape of the water drop no longer changes, click the contact angle measurement button to display the measurement results. That is to say, the wetting angle of the detergent solution of this concentration is measured on the slide;
8. Inject the microinjector into distilled water for cleaning and then use the detergent solution to be measured for wetting. After injecting the solution, the experiment was carried out, and the other groups were measured according to the above wetting angle measurement process. At the same time, the hard water was also measured.

Replace the glass slide with the metal block (steel grade TP140V) and repeat the above experiments on sandstone samples. The flow chart of the experimental device is shown in Figure 3

(2) Soak the slides, metal sheets, and cores after oil wetting in the prepared surfactant solutions of different concentrations. After the samples are dried, determine the wetting angle of water on the samples. Taking the slides as an example, the specific operation steps are as follows:

1. The hydrophilic slides (seven slides) were prepared with 10, 15, 20, 25, 30, 35, and 40% concentrations of the washing solution with water as the reference amount;
2. Hydrophilic slides (seven slides) were prepared according to the above steps 2−3, and the hydrophilic slides were soaked in oil for 24 h and then dried in a constant-temperature drying oven at 90 °C to prepare oleophilic slides;
3. Immerse the hydrophilic slides into seven groups of the washing agent solution, soak them for 10 min, and take them out and dry them;
4. Take water as the liquid phase, take the glass slide dried in step 2 as the solid phase, and use the interface parameter integrated measuring system to measure the wetting angle between water and the glass slide, and for the measurement steps, refer to the above steps 5−8.

In the same way, replace the glass slide with the metal block (steel grade TP140V) and repeat the above experiments on sandstone samples. The flow chart of the experimental device is shown in Figure 4

3. RESULTS AND DISCUSSION

3.1. Analysis of Surface Tension Test Results. In this project, two kinds of flushing solutions, type A and type B, which are commonly used in the field, are proposed to be used to determine the surface tension performance under different concentration conditions. Type-A flushing fluid is viscous, colorless, and tasteless, with a density of about 1.0 g/cm³. It is developed by a company in the United States that is committed to the research and development and application of new technologies and products in oil and gas fields. Its products and services almost cover the fields of drilling, completion, oil production, workover, production increase, and oil and gas collection and transportation in oil and gas fields. Type-B
flushing fluid is developed by a domestic company specializing in the research and development, production, sales and provision of oilfield engineering, and technical services of cementing cement admixture. The flushing fluid is light yellow, has a pungent odor, has a density close to that of type-A flushing fluid, and absorbs a large number of bubbles.

The results of surface tension of type-A and type-B flushing solutions with different concentrations at atmospheric pressure and 20 °C are shown in Table 1.

According to the data in Table 1, the change of surface tension of type-A and type-B solutions with the change of flushing solution concentration is shown in Figure 5.

It can be seen from Figure 5 that the surface tension of type-A surfactant decreases rapidly with an increase of concentration in the range of 10–25% and shows almost no change when the concentration is greater than 25%. However, with an increase of concentration, the decrease of surface tension of the type-B surfactant system is lower than that of the type-A surfactant system. When the concentration of B-type surfactant reaches 40%, the surface tension is 29.10 mN/m, which is higher than that of A-type surfactant at 25%. Because the surfactant molecule is called "amphiphilic molecule", its structure is amphiphilic: one end is a hydrophilic group and the other end is a hydrophobic group. When foreign substances are added to the surfactant, the more the surface tension, the less the surface free energy of the liquid and the lower the surface tension coefficient; that is to say, it can extend in the water. Therefore, the hydrophilic effect of type-A surfactant is better than that of type-B surfactant.

3.2. Analysis of Interface Tension Test Results

In this paper, the interfacial tension values of different concentrations of the flushing fluid and oil (diesel oil, white oil) are measured by two types of flushing fluid systems A and B. The density of white oil and diesel oil is 0.86 g/cm³.

3.2.1. Type A and Interface Tension between Flushing Fluid and Diesel Oil

The measurement results of the interfacial tension between A-type and B-type flushing liquids and white oil with different concentrations, at atmospheric pressure and 20 °C are shown in Table 2.

According to the data in Tables 2 and 3, the average values of interfacial tension of white oil and diesel oil in different concentrations of type-A flushing fluid and diesel oil in different concentrations of type-A and type-B flushing fluids are obtained, as shown in Figure 6.

It can be seen from Figure 6 that when the concentration of the flushing fluid is 10%, the interfacial tension between type-A and type-B flushing fluids and diesel oil is small, but the decreased degree of the interfacial tension between type-A flushing fluid and diesel oil is greater than that of type B, and the interfacial tension between type-A flushing fluid and diesel oil is almost 0, that is to say, the lipophilic group can be well miscible with diesel oil to reduce the interfacial tension between them, while the interfacial tension between type B and diesel oil is slightly higher than that of type-A flushing fluid. Therefore, the results show that A-type surfactant has better lipophilic properties, and its performance is superior to that of the B-type system.

3.3. Test and Evaluation of Wetting Reversal Ability under Different Conditions—Type-A System

There are two experimental methods to evaluate the wetting reversion ability of a flushing liquid system to more accurately evaluate the wetting ability of the flushing agent.

3.3.1. Measurement of Contact Angle between Different Surfactant Solutions and Oil-Wet Samples

The samples (slides, metal sheets, cores) are treated with oil wetting, as shown in Figure 7. Then, the wetting angle of the oil-wet sample
was measured by dropping different washing agent solutions on it.

Table 3. Interfacial Tension between A-Type and B-Type Detergent Solution and Diesel Oil at Different Concentrations

| flushing fluid/water | A-type measure value (mN/m) | average value | B-type measure value (mN/m) | average value |
|----------------------|----------------------------|--------------|----------------------------|--------------|
| 10%                  | 0.409 0.401 0.418 0.418    | 0.418        | 2.021 1.995 2.021 1.995    | 1.995        |
| 15%                  | 0.392 0.375 0.384 0.384    | 0.384        | 1.493 1.493 1.473 1.473    | 1.473        |
| 20%                  | 0.368 0.359 0.359 0.367    | 0.366        | 1.267 1.269 1.261 1.267    | 1.261        |
| 25%                  | 0.351 0.343 0.351 0.351    | 0.351        | 1.194 1.212 1.212 1.194    | 1.191        |
| 30%                  | 0.343 0.337 0.343 0.3367   | 0.347        | 1.018 1.034 1.034 1.018    | 1.018        |
| 35%                  | 0.329 0.314 0.322 0.307    | 0.325        | 0.839 0.825 0.839 0.825    | 0.839        |
| 40%                  | 0.307 0.293 0.293 0.307    | 0.303        | 0.798 0.784 0.798 0.798    | 0.798        |
| 0%                   | 12.385 12.283 12.385 12.283| 12.301       | 12.385 12.283 12.385 12.283| 12.182       |

Table 4. Average Value of Wetting Angles of Different Concentrations of A-Type Rinse Solution on the Sample (White Oil Treatment)

| flushing fluid/water | slide  | metal sheet | core   |
|----------------------|--------|-------------|--------|
| 10%                  | 38.84  | 33.31       | 32.33  |
| 15%                  | 33.83  | 31.07       | 33.47  |
| 20%                  | 32.77  | 23.24       | 32.4   |
| 25%                  | 34.34  | 22.46       | 29.43  |
| 30%                  | 30.34  | 21.07       | 24.767 |
| 35%                  | 30.22  | 20.60       | 24.1   |
| 40%                  | 29.63  | 18.62       | 23.57  |
| 0%                   | 61.11  | 64.47       | 92.1   |

change of the wetting contact angle is measured. It can be seen from Figure 8a that the surface roughness of the treated sample is higher than that of the glass slide and the metal sheet, so that the core can be fully in the oil-friendly state, and the wetting contact angle is greater than 90°; the surface of the glass slide

Figure 6. Chart of changes in average values of interfacial tension of the oil phase under different flushing liquid concentrations.

Figure 7. Samples soaked in different solvents.

Figure 8. Change of average wetting angle of A-type flushing liquid with different concentrations on the oil-wet (white oil/diesel oil) sample.
and the metal sheet is relatively smooth, and the oil film on the sample surface is not evenly distributed and dripped after the oil-wet sample is dried. After fixing the water, the contact angle of wetting should be less than 90°.

In addition, titration of the A-type surfactant system with different concentrations on three kinds of samples shows that with an increase of surfactant concentration, the wetting contact angle decreases gradually, which indicates that the surfactant can change the wettability of lipophilic samples. However, when the surfactant concentration is more than 25%, the wetting contact angle of the three kinds of samples changes little with an increase of surfactant concentration. Therefore, it is preliminarily determined that the optimal concentration of the surfactant in the type-A system is 25%.

(2) Determination of the wetting angle of A-type flushing fluid (diesel)

The wettability of the A-type washing agent solution is determined by dropping it on the sample (slide, metal sheet, and core) after diesel oil treatment. The results are shown in Table 9.

From the data in Table 5 above, the wetting contact angle of the diesel-treated sample (slide, metal sheet, core) is obtained after being wetted by diesel, and the A-type surfactant of different concentrations is titrated on its surface. It can be seen from Figure 8b that a small amount of surface activity can quickly change the wettability of the test. When the concentration is 10%, the wettability and wettability of the test are reduced by half. When the concentration is 25%, the wetting contact angle of the three kinds of samples is about 27°, while when the concentration is 30%, the wetting contact angle of the three kinds of samples is about 26°. Therefore, under the condition of diesel wetting, the best surfactant concentration of 25% can change the wettability of the sample.

3.3.2. Determination of Wetting Contact Angle between Oil-Wet Sample and Water after Immersion in the Washing Agent Solution

The samples (glass slide, metal sheet, core) were treated by oil wetting and then soaked in different washing agent solutions, and the wetting angle of the samples was measured by dropping water on the dried samples.

(1) Contact angle between sample and water after immersion in A-type washing solution (white oil treatment)

The contact angle between the white oil-wet sample (slide, sheet metal, core) and water was measured after immersion in A-type washing solution. The results are shown in Table 6.

According to the data in Table 6, Figure 9a is obtained; that is, after the white oil treatment, the glass slide, metal sheet, and core are soaked in A-type washing solution of different concentrations, then the water is titrated on the surface, and finally, the wettability is determined to evaluate the oil washing ability of A-type washing solution. It can be seen from Figure 9a that the wetting contact angle between the sample and water decreases gradually with an increase of surfactant concentration. When the concentration of the surfactant is more than 30%, the change of the contact angle is small for the metal chip, while for the glass slide and core, the contact angle is similar when the concentration is 25 and 30%. For this reason, the surfactant concentration is recommended to be 25−30% for the formation of casing and sidewall rock in the flushing section of the isolation fluid during cement injection.

(2) Contact angle between sample and water after immersion in A-type flushing solution (diesel)

The contact angle between diesel oil and water is determined by dropping it on the sample (slide, metal sheet, and core) and soaking it in A-type washing solution. The results are shown in Table 7.

According to the data in Table 7, Figure 9b is obtained; that is, after diesel oil treatment, the sample (glass slide, metal sheet,
core) is soaked with A-type washing solution of different concentrations, and then, the water is titrated on the surface to determine its wettability to evaluate the oil washing ability of A-type washing solution. It can be seen from Figure 9b that with an increase of type-A surfactant concentration, the change of wetting contact angle between the glass slide and metal sheet is similar; for the core, when the concentration is greater than 25%, the wetting contact angle decreases greatly, from 32.1° to 25°. The reason for this phenomenon is that the oil on the surface of the core is more difficult to remove than that on the slide and the metal sheet because of the roughness of the rock surface and the characteristics of porosity and permeability. Only when the concentration is high, the wetting reversal gradually occurs.

3.4. Test and Evaluation of Wetting Reversal Ability under Different Conditions

3.4.1. Determination of Wetting Angle of Type-B Flushing Fluid (Diesel). The wettability of B-type washing agent solution is determined by dropping it on the sample (slide, metal sheet, and core) after diesel oil treatment. The results are shown in Table 8.

According to Table 8, the relationship between the average value of the wetting angle and the concentration of the flushing solution is shown in Figure 14 after the samples (slide, metal sheets, and core) after diesel oil treatment. The results are shown in Table 8.

| flushing fluid/water | slide | metal sheet | core |
|----------------------|-------|-------------|------|
| 10%                  | 38.32 | 36.73       | 37.25|
| 15%                  | 35.53 | 34.67       | 34.93|
| 20%                  | 34.9  | 33.1        | 32.7 |
| 25%                  | 33.23 | 32.7        | 32.1 |
| 30%                  | 32.93 | 31.6        | 29.7 |
| 35%                  | 32.37 | 31.47       | 26.8 |
| 40%                  | 30.67 | 29.83       | 25   |

Table 8. Wetting Angle of Different Concentrations of B-Type Detergent Solution on the Sample (Diesel Treatment)

According to Table 8, the relationship between the average value of the wetting angle and the concentration of the flushing solution is shown in Figure 14 after the samples (slides, metal sheets, and cores) treated with diesel oil are washed with different concentrations of B-type flushing solution.

It can be seen from Figure 10 that the change of wetting angle of the three samples is the same. When the concentration of the washing liquid is 10%, the decrease of wetting angle tends to be gentle, but at the same concentration, the values of the wetting angle are all higher than the lubricating angle of type-A washing liquid. When the surfactant concentration is 40%, it reaches the minimum value, of about 33°.

3.4.2. Contact Angle between Sample and Water after Immersion in B-Type Flushing Solution (Diesel). The contact angle between diesel oil and water is determined after wetting the sample (slide, metal sheet, core) and soaking it in B-type washing solution. The results are shown in Table 9.

According to the data in Table 9, Figure 11 is obtained; that is, after diesel oil treatment, the sample (glass slide, metal sheet, core) is washed with B-type flushing fluid of different concentrations, and its wettability is finally determined to evaluate the oil washing ability of B-type flushing fluid.

It can be seen from the oil washing ability of the flushing solution in Figure 11 that the reduced amplitude of the wetting angle of different samples is different after the sample is treated with diesel oil. Under the same concentration, the wetting angle of the core is smaller than those of the glass slide and the metal slide, that is, the oil-wet core is easier to be washed. Compared with A-type flushing solution, the wetting angle of the sample is still higher after oil washing, i.e., the wettability changes slightly.
Indeed, the washing capacity, A-type flushing solution is still better than B-type flushing solution.

3.5. Analysis of the Compatibility of Surfactants in Different Systems. 3.5.1. Test Results of Surface Tension of the Coordination System. To select the best flushing solution, a mixing experiment of type-A flushing solution and type-B flushing solution was carried out with a total concentration of 30%. The surface tension of the flushing solution was measured in the proportions of 5:95, 25:75, 50:50, 75:25, and 95:5%, respectively. The experimental results are shown in Table 10.

According to the data in Table 10, with the change of the concentration of the flushing solution, the change of the surface tension of the mixed flushing solution is shown in Figure 12.

Table 10. Surface Tension of 30% Surfactant Solution

| A-type:B-type | measure value (mN/m) | average value |
|---------------|----------------------|---------------|
| 5:95%         | 32.25                | 32.43         |
| 25:75%        | 32.11                | 32.32         |
| 50:50%        | 31.99                | 32.11         |
| 75:25%        | 32                    | 32.08         |
| 95:5%         | 31.87                | 31.97         |

From the data distribution in Figure 12, it can be seen that the surface tension of the mixed solution decreases with an increase of type-A surfactant dosage. Therefore, the addition of the A-type system is helpful to reduce the surface tension of the mixed fluid. According to the surface tension data of 30% concentration of two kinds of surfactants, the surface tension of type A is 22.98 mN/m and that of type B is 32.32 mN/m. The surface tension of the mixture is about 32 mN/m. The surface tension of the A-type system with 25% concentration is 23.37 mN/m. Therefore, although the A-type surfactant system can reduce the surface tension of the mixture, the effect is not very significant, which is worse than the use of the A-type surfactant system with a lower concentration. It is suggested that the two surfactants do not match.

3.5.2. Test Results of Interfacial Tension of the Coordination System. The interface tension of type-A and type-B flushing fluids was measured by the experiment of mixing type-A and type-B flushing fluids with a total concentration of 30%. The experimental results are shown in Table 11.

According to the data in Table 11, the average values of the interface tension of diesel oil in different concentrations of A-type flushing solution and diesel oil in different concentrations of A-type and B-type flushing solutions are obtained, as shown in Figure 13.

Table 11. Interfacial Tension between 30% Detergent Solution and Diesel Oil

| A-type:B-type | measure value (mN/m) | average value |
|---------------|----------------------|---------------|
| 5:95%         | 0.839                | 0.825         |
| 25:75%        | 0.668                | 0.668         |
| 50:50%        | 0.499                | 0.499         |
| 75:25%        | 0.337                | 0.329         |
| 95:5%         | 0.314                | 0.311         |
| water         | 12.385               | 12.283        |

It can be seen from Figure 13 that with an increase of type-A surfactant in the mixture, the interfacial tension of the mixture decreases continuously, from the initial 0.831 to 0.321 mN/m. The interfacial tension between type-A and type-B surfactants with 30% concentration and diesel oil is 0.34 and 1.02 mN/m, respectively. Therefore, the interfacial tension of the mixed solution is lower than that of B-type solution, which is similar to that of A-type surfactant at a concentration of 35%. The results show that the effect of mixing of two kinds of surfactants on the improvement of the interfacial tension is not very significant. It is suggested that only A-type surfactant should be used.

3.5.3. Evaluation of the Wetting Reversal Ability of the Mixed System. (1) Measurement of the contact angle between different surfactant solutions and oil-wet samples

The samples (slides, metal sheets, and cores) were wetted with 30% of the total concentration of the washing agent solution by diesel oil, and then, the mixed washing agent solution was dropped on the oil-wet sample to determine its wetting angle.

The wettability of the mixed detergent solution drops on the slide after diesel treatment is determined, and the results are shown in Table 12.

According to the data in Table 12, the relationship between the average value of wetting angle and the concentration of washing solution is shown in Figure 14; after diesel treatment,
slides, metal sheets, and rock cores are washed by the mixed washing solution.

It can be seen from Figure 14 that with an increase of the amount of type-A surfactant, the contact angle between the mixed solution and the three types of oil-wet samples decreases gradually. When type A accounts for 95% of the volume of the mixture, the wetting contact angle measured by the three kinds of samples is about 29° and its reduction is lower than that of pure type-A surfactant with a concentration of 30% acting on the three kinds of oil-wet samples (27°), but the influence of the mixture on the wetting contact angle is due to the separate type-B system, and the wetting contact angle under this condition is about 36°. Therefore, the effect of A-type surfactant is better than that of B-type surfactant.

(2) Determination of wetting contact angle between the oil-wet sample and water after immersion of washing agent solution

Mix type-A and type-B surfactants, wet the sample (slide, metal sheet, core) with diesel oil, then put it into the mixed washing agent solution for immersion, and measure the wetting angle of the water drop on the dried sample.

The contact angle between diesel oil and water was measured after the sample was wetted and soaked in the washing solution. The results are shown in Table 13.

Table 13. Average Value of the Wetting Angle of Lipophilic Sample with Water after Immersion in the Mixed Washing Solution (Diesel Treatment)

| A:B | slide  | metal sheet | core   |
|-----|--------|-------------|--------|
| 5:95% | 38.96  | 38.43       | 36.66  |
| 25:75% | 37.25  | 37.05       | 33.73  |
| 50:50% | 35.58  | 35.45       | 32.33  |
| 75:25% | 35.26  | 34.4        | 29.7   |
| 95:5%  | 34.27  | 33.33       | 28.71  |

According to the data in Table 13, Figure 15 is obtained; that is, after diesel oil treatment, the glass slide, the metal sheet, and the core are washed with the washing liquid (30% of the total concentration), and finally, the wettability is determined to evaluate the oil washing ability of type-A washing liquid.

It can be seen from Figure 15 that with an increase of the volume of the type-A surfactant system, the wetting contact angle between the three kinds of samples treated with the surfactant and water decreases gradually. Among them, the decreasing range of the wetting contact angle of the mixed solution to the core sample is larger than those of the glass slide and metal slide. By comparing the wetting contact angle between water and the sample treated with A-type and B-type systems, it can be found that the wetting contact angle between the sample treated with 30% B-type system and water is 32−38°, the wetting contact angle between the sample treated with the A-type system and water is 29−32°, and the wetting contact angle between the sample treated with the mixed solution and water is 28.2−34.2°. The comprehensive analysis shows that the wetting reversal ability of the single A-type surfactant system is better than that of the mixed liquid system, and the wetting reversal ability of the mixed liquid system is better than that of the single B-type system. It is suggested that only the A-type surfactant system can achieve the effect of wetting reduction.

4. CONCLUSIONS

The content of this chapter is mainly based on the investigation of the existing evaluation methods of wetting reversal. An integrated measurement system of interface parameters and a rotating liquid interface tension tester are selected to develop a set of evaluation methods for wetting reversal. According to the evaluation of wetting reversal of A, B, and the mixed solution, the following conclusions are obtained:

1. The experimental results of surface and interface tension of the two kinds of flushing fluids show that the surface tension and interface tension of type-A flushing fluid are lower than those of type B at the same concentration; that is to say, the hydrophilic effect of type-A flushing fluid is better than that of type B.

2. Evaluation of dewetting ability: a small amount of the surfactant can better change the wettability of oil-wet samples; the change of wetting angle tends to be gentle at
30% surfactant concentration, and the dewetting ability of type A is better than that of type B.

3. Oil washing ability evaluation: the oil washing effect is better for core oil washing than that for sheet metal and worst for slide oil washing. In terms of oil washing ability, 30% surfactant concentration is recommended.

4. Based on the surfactant 30% flushing solution, solutions A and B are mixed in different proportions. The evaluation result of the wetting reversal ability of the mixed solution is that the wetting reduction ability of the mixed solution is equivalent to that of type-A flushing solution, which is better than that of type-B flushing solution, while the test results of surface tension, interface tension, and oil washing ability are all equivalent to that of type-A flushing solution.

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Notes
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