

Problems in the Test Procedure of Hydrated Microsphere Particle Size

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Abstract: Microsphere profile control is a new deep profile control technology in oilfield, which has developed in recent years. The particle size distribution of hydrated microsphere is an important basis for its application in formation pore throat, which has direct influence on the effect of profile control. First, the results of hydrated microsphere particle size were obtained from the current testing method, which was based on the static laser particle size analyzer. And then, the existing problems in current particle measurement of hydrated microsphere were studied. Results showed that there were two main problems in the current test methods of hydrated microsphere particle size. One was the poor reproducibility of the test results, and the other was the unrecognized particle size distribution of the truly hydrated microsphere. Finally, the suggestion of analyzing the influencing factors during the test procedure of hydrated microsphere particle size was given.

1. Introduction

Microsphere flooding is a new deep flooding technology in oilfield, which has developed in recent years. The microsphere technology uses many different methods to synthesize spherical or spherical-like microsphere, including micro-emulsion polymerization method, suspension polymerization, as well as the method of solution polymerization, et al. The microsphere particle size is ranged from tens of hundreds of nanometers to hundreds of microns, which has great significance to improve water flooding development effect and the crude oil recovery.^[1-2]

The particle size of microsphere and the core pore throat should match with each other theoretically.^[3] The hydrated size of microsphere is too small to form a stable and effective pore throat plugging because of the easy through. While the big hydrated size of microsphere will lead to the difficult injection, which cannot achieve the purpose of deep profile control. Therefore, the size and distribution of microsphere size is an important basis for its application in the pore throat size, which directly affects the displacement control effect of microsphere.^[4-5]

At present, there are two standard methods for measuring micron size particles.^[6-7] One is based on dynamic light diffraction (DLS), and the other is based on static light (laser diffraction). However, there is no national standard and industry standard for measuring particle size of microsphere.^[8] The investigation was showed that the main methods of particle size test are transmission electron microscopy (TEM),^[9-10] and laser particle size distribution method^[1-2]. Since TEM must be operated in vacuum, it requires high professional and technical level, which limits its universal application. Laser



particle size analyzer is an instrument by means of laser scattering, which is used to measure particle distribution in emulsion, suspension, as well as the powder sample. It has been widely used in the analysis and research of petroleum and petrochemical industry. ^[1]

The research shows that most domestic oil fields, including Huabei Oilfield, universities, as well as enterprises and other units related to the production of microsphere, ^[11-16] use laser particle size distribution analyzer to obtain the microsphere hydrated size value by testing microsphere water solution sample of microsphere, which is used to guide the microsphere application in oil fields.

In this paper, problems existed in the test procedure of hydrated microsphere particle size was analyzed, which was based on summarizing the experimental results. On the above basis, the suggestion of analyzing the influencing factors during the test procedure of hydrated microsphere particle size was given.

2. The experiment

2.1 Experimental equipment and materials

2.1.1 *Experimental equipments.* LA-950S laser particle size analyzer, Horiba company of Japanese. JK-MSH-PRO-6B magnetic agitator, scientific instrument company of Shanghai. H-101 electrothermal constant temperature drying box, Yao instrument equipment factory of Shanghai.

2.1.2 *Experimental materials.* Technical white oil, Jingshan of Hebei petrochemical plant. Emulsifier A and emulsifier B, industrial grade, Jiebote energy technology Co. Ltd of Beijing. Microsphere samples used in profile control of Huabei oilfield.

2.2 Experimental steps

Shake fully the microsphere samples with distilled water. Formulate aqueous solution of microsphere, whose mass fraction is 1%. Sub-package to seal the stainless steel drums. Take the microsphere solution sample out from the electric thermostatic drying oven for a period of time. Place at room temperature for cooling.

Turn on the laser particle size, preheat 30 minutes, and then calibrate the instrument according to the operating procedures. Test the distilled water as the empty sample to deduct the background value. Fully shake the microsphere sample after the check amount in the beaker with distilled water, and then adjust the sample concentration to transmittance falls in the range of suitable instruments (red semiconductor laser to meet 80%~90%, the blue LED light meet 70%~90%). Finally, test the size and distribution of dispersion medium in particle samples using laser particle size analyzer at room temperature (refractive index: 1.33; particle refractive index: 1.50).

3. Experimental results and discussion

3.1 Results of hydrated microsphere particle size

The indoor test prepared 4 different aqueous solutions of microsphere samples, which were used in profile control. The microsphere samples were hydrated at 70°C for 4 days, and then were tested the particle size and the distribution by using the laser particle size analyzer. Besides, the median particle size was characterized by D_{50} , which was the corresponding particle size when the cumulative frequency reached to 50%. The D_{50} data of four samples were shown in table 1.

From table 1, it can be seen that the D_{50} repeatability of the same microsphere sample were poor. Besides, the test data even appeared in the order of magnitude of nanometer and micron. Moreover, the relative standard deviation was up to 136%.

Table 1 diameter of 1% solution prepared by different microsphere samples hydrated at the condition of 70°C for 4 days

sample number	particle size of microsphere hydrated at 70°C for 4 days(μm)	deviation
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	determination times						mean value	standard deviation(μm)	relative standard deviation(%)
	1	2	3	4	5	6			
1 [#]	0.158	0.145	3.60	0.146	3.31	0.167	1.25	1.71	136
2 [#]	0.319	0.164	8.06	0.186	8.53	8.27	4.25	4.42	104
3 [#]	0.159	0.158	2.69	2.54	0.153	2.82	1.42	1.39	98
4 [#]	13.41	12.95	18.78	1.48	25.58	2.13	12.39	9.39	76

3.2 problems existing in the test of hydrated microsphere particle size

Taking microsphere sample 1[#] as an example, figure 1 was the particle size distribution curve of the microsphere sample under the same experimental conditions. Figure 1(a) showed that the size distribution of sample 1[#] was 0.100~0.389 μm , and the medium particle size was 0.158 μm . However, figure 1(b) indicated that the particle size distribution range was 1.981~15.175 μm , and the medium particle size was 3.60 μm . The above results showed that the particle size of sample 1[#] was either nanometer or micrometer. That was to say, the size of the microsphere sample showed a difference of magnitude between the nanometer and micrometer particles, even under the same experimental conditions, which led to the fact that the true distribution of the particle size could not be distinguished.

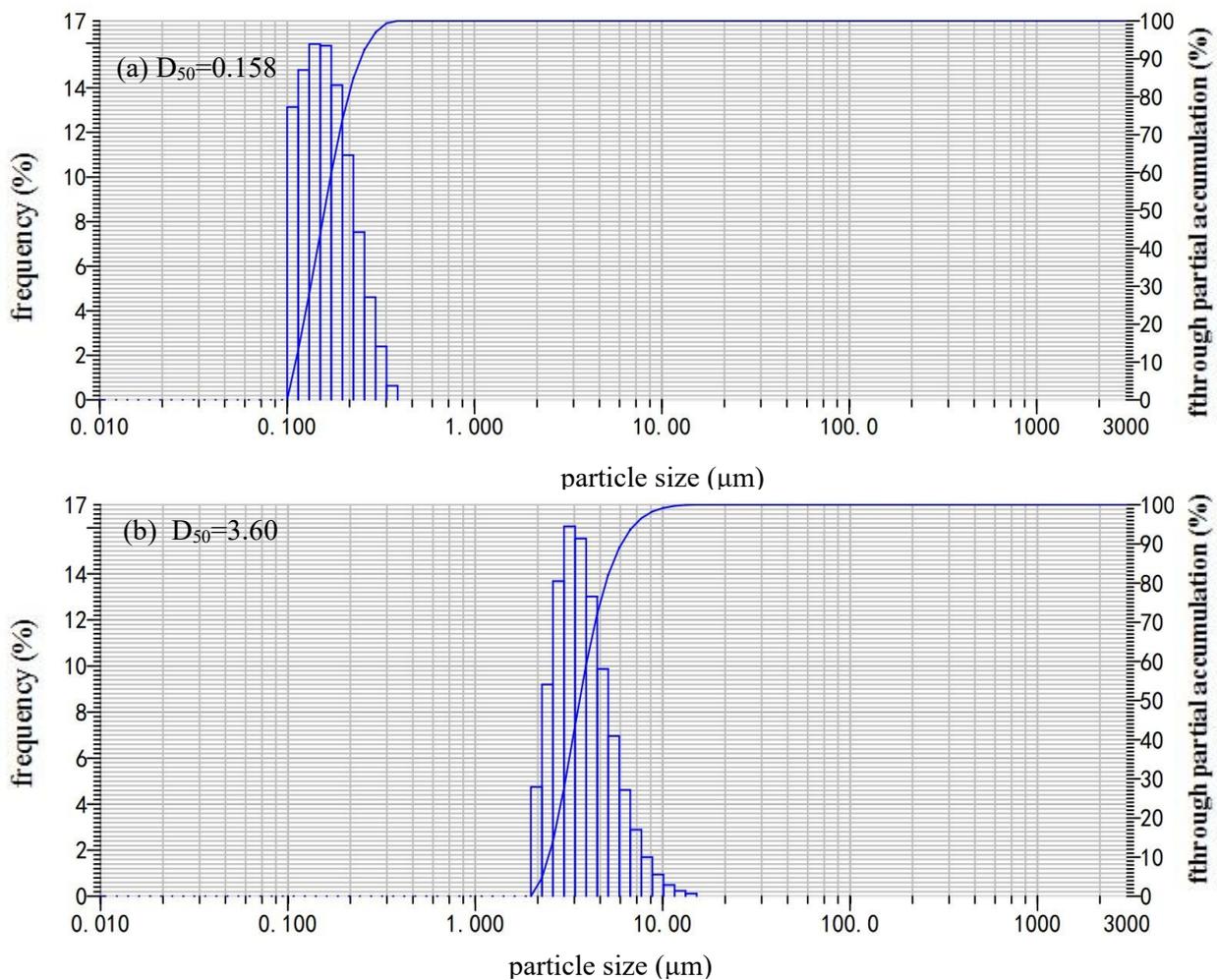


Figure 1. Particle size test results of 1[#] microsphere sample after 4 days at 70°C

Figure 2 showed the size distribution curve of microsphere sample 4[#] under the same test conditions. Figure 2(a) showed that the size distribution of sample 4[#] was 8.816~58.953 μm , and the median particle size was 12.95 μm . Whereas, figure 2(b) indicated that the particle size distribution range was 17.377~678.504 μm ; In addition, the median particle size was 25.58 μm . Based on the above data, it can be seen that the particle size distributions of 4[#] sample were in micrometer scale and overlap in some areas (17.377~58.953 μm). However, the particle size distribution of the two measurements was less than that of overlap, so that the peak value of the particle size distribution was not similar, and the true value of the particle size cannot be obtained.

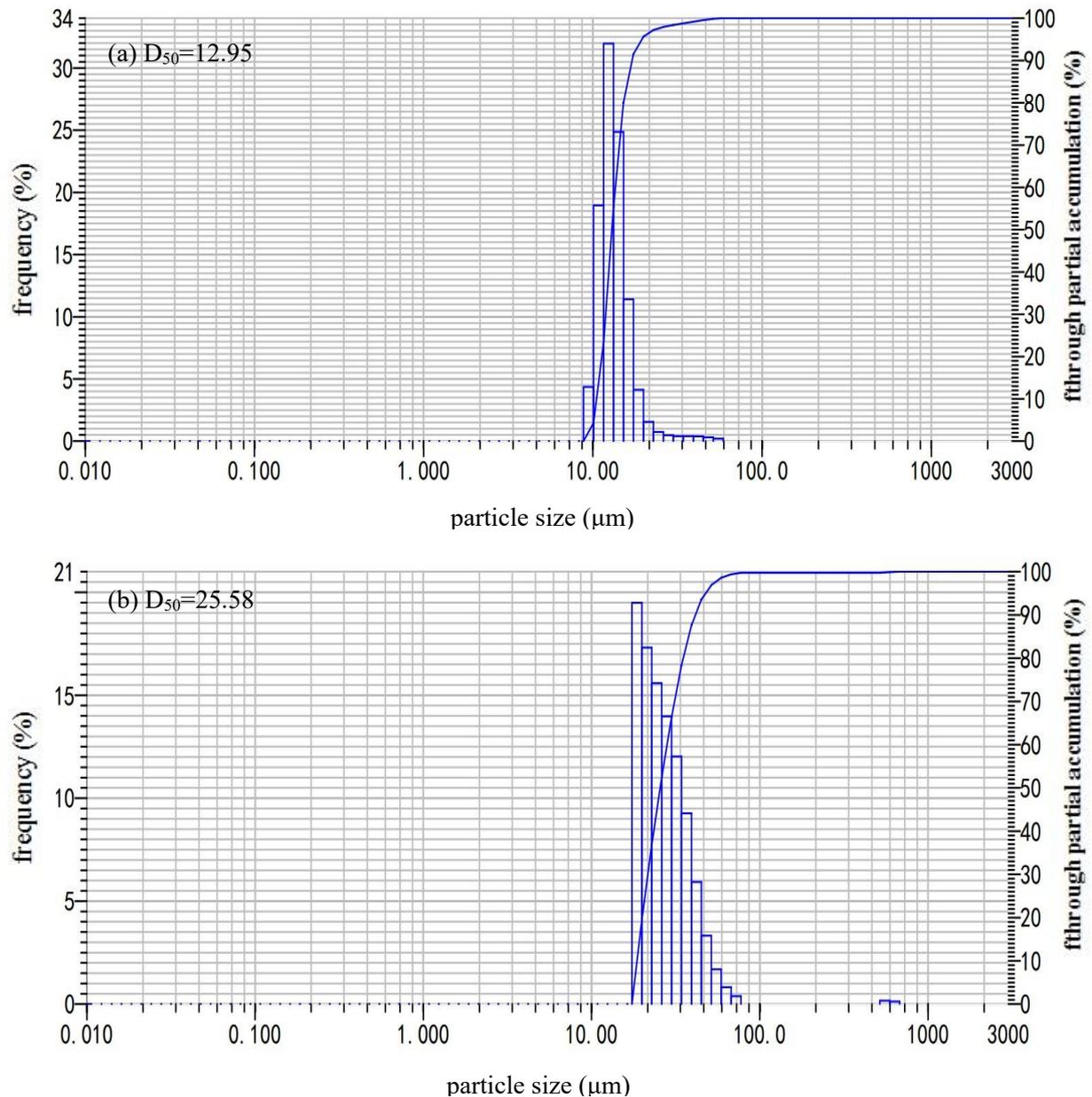


Figure 2. Particle size test results of 4[#] microsphere sample after 4 days at 70°C

In addition to the above, the distribution of microsphere size resulted in the distribution of doublet in figure 3. Figure 3 showed that the particle size distribution of sample 1[#] existed two ranges, 2.269~4.472 μm and 5.122~19.904 μm . It also caused the disturbance of the true particle size distribution of microsphere.

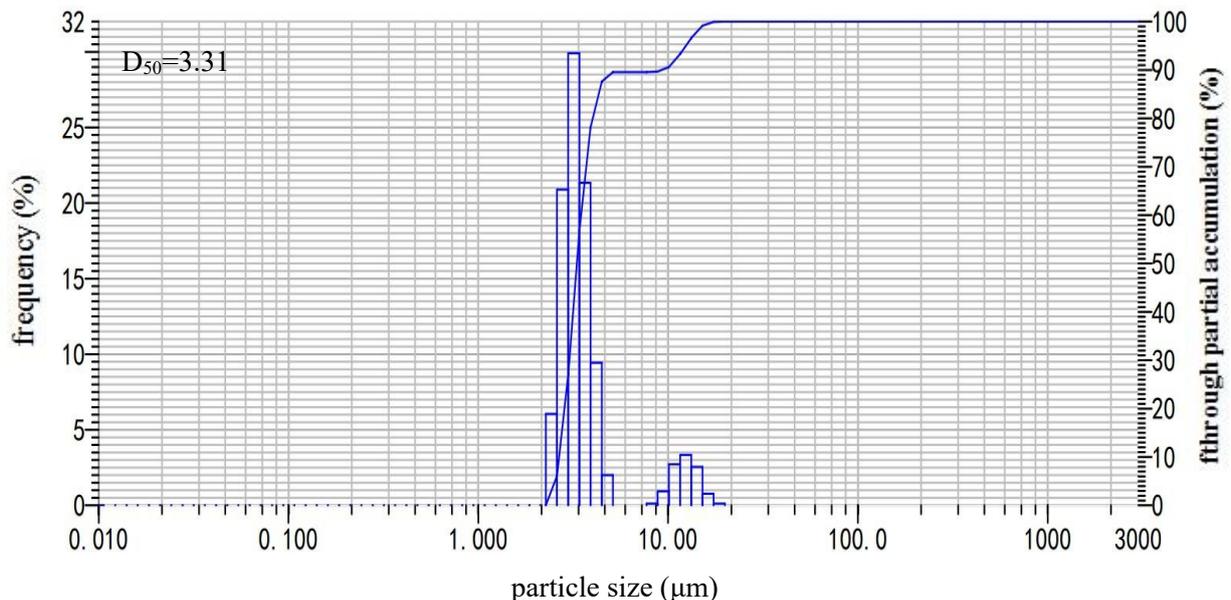


Figure 3. Doublet particle size distribution of 1# hydrated microsphere after 4 days at 70°C

Based on all the above particle size results of the hydrated microsphere samples, it showed that there were some problems in the current test methods of hydrated microsphere particle size of microsphere, including the doublet particle size distribution, and the phenomenon of different magnitude. It resulted in the poor reproducibility of the test results, which led to the unrecognized particle size distribution of the truly hydrated microsphere.

3.3 The suggestions about the test of hydrated microsphere particle size

Since the current testing method of the static laser particle size analyzer had some problems, which were the poor reproducibility of the test results, and the unrecognized particle size distribution of the truly hydrated microsphere. It was extraordinarily important to analyze the test factors of hydrated microsphere particle size, which was suggested to study through analyzing the morphology and the composition of microsphere, as well as the testing process of the static laser particle size analyzer.

4. Conclusions

- i. The median particle sizes of four different microsphere samples were obtained through the static laser particle size analyzer. The D_{50} repeatability of the same microsphere sample was poor. Besides, the test data even appeared in the order of magnitude of nanometer and micron. Moreover, the relative standard deviation was up to 136%.
- ii. The existing problems in current particle measurement of hydrated microsphere were studied through analyzing the results of the hydrated particle size. Results showed that there were two main problems in the current test methods of hydrated microsphere particle size. One was the poor reproducibility of the test results, and the other was the unrecognized particle size distribution of the truly hydrated microsphere.
- iii. The suggestions about the test of hydrated microsphere particle size were given, which were analyzing the morphology and the composition of microsphere, as well as the testing process of the static laser particle size analyzer.

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