

Research Article

Experimental Study on Expansion Characteristics of Core-Shell and Polymeric Microspheres

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Traditional polymeric microsphere has several technical advantages in enhancing oil recovery. Nevertheless, its performance in some field application is unsatisfactory due to limited blockage strength. Since the last decade, novel core-shell microsphere has been developed as the next-generation profile control agent. To understand the expansion characteristic differences between these two types of microspheres, we conduct size measurement experiments on the polymeric and core-shell microspheres, respectively. The experimental results show two main differences between them. First, the core-shell microsphere exhibits a unimodal distribution, compared to multimodal distribution of the polymeric microsphere. Second, the average diameter of the core-shell microsphere increases faster than that of the polymeric microsphere in the early stage of swelling, that is, 0–3 days. These two main differences both result from the electrostatic attraction between core-shell microspheres with different hydration degrees. Based on the experimental results, the core-shell microsphere is suitable for injection in the early stage to block the near-wellbore zone, and the polymeric microsphere is suitable for subsequent injection to block the formation away from the well. A simple mathematical model is proposed for size evolution of the polymeric and core-shell microspheres.

1. Introduction

Waterflooding is the most common technique in oil reservoir development. In high-permeability reservoirs, such as Bohai oil field in eastern China, the heterogeneity usually deteriorates sharply in the late stage of waterflooding [1, 2]. In low-permeability reservoirs, such as Changqing oil field in western China, injection water channeling occurs by the interwell fracture network. Therefore, the polymeric microsphere, a profile control agent, is developed to improve the water swept volume [3]. When it meets water in the reservoir, the polymeric microsphere swells and can be migrated, or retained. Following elastic deformation, it may be remigrated and recaptured [4]. Moreover, this profile control agent can adapt to the characteristics of pore throat, avoiding the injection difficulty [5]. Some pilot tests have

been performed in Shengli, Jidong, and Dagang oilfields in China and Mannville Pools in Canada [6], where effective water control and enhanced oil recovery were observed [7–10]. In other cases, transport and retention of microspheres or particles may lead to well productivity impairment [11–13].

There have been many types of polymeric microspheres, such as colloidal dispersion gel, gel microsphere (PPG), bright water, and pH-sensitive cross-linked polymers [14–18]. The major difference among these microspheres is the expansion size versus swelling time [19, 20]. Formation temperature, salinity, and swelling time affect the expansion performance of microspheres. The expansion factor of grain diameter gradually increases with the increasing temperature and swelling time, whereas it decreases with the increasing salinity [21–24]. However, some field applications in high-heterogeneity

reservoirs are unsuccessful because it is fragile after swelling and is prone to deformation under certain pressure [25].

To overcome the disadvantages of the polymeric microsphere, a novel core-shell microsphere is developed as the next-generation profile control agent [26–29], which has a core-shell double-layered structure. It consists of an inner layer and an outer layer. The inner layer has positive charge, whereas the outer layer has negative charge [30, 31]. The core-shell microsphere swells with water, in which the core part swells fast, whereas the shell part expands relatively slowly. Therefore, the core-shell microsphere can easily become a bipolar microsphere with positive and negative charges simultaneously [24, 32, 33].

It is quite difficult to precisely determine the local structure of microspheres using the conventional diffraction techniques because of the nanoscale grain size and surface modification [34]. Research on expansion characteristic differences between the core-shell and polymeric microsphere is not available in the literature.

In the present work, we investigate the size distribution characteristics and average diameter evolution during expansion of the two types of microspheres, that is, the polymeric microsphere and core-shell microsphere. In Section 2, we analyze the main feature of the core-shell microsphere expansion mechanism. In Section 3, laboratory devices and materials are introduced, and the experimental procedure to measure the expansion size variation of the microsphere is presented. In Section 4, size distribution and average diameter evolution of two types of microspheres are analyzed. Section 5 presents two mathematical models for average diameter evolution. Conclusions in Section 6 finalize the paper.

2. Core-Shell Microsphere Expansion Analyses

2.1. Core-Shell Microsphere Expansion Mechanism. Figure 1 illustrates the structure of a core-shell microsphere, which consists of an outer layer and an inner layer which is gel core. Since the rock surface usually has negative charge, it is designed on purpose that the outer layer has negative charge, whereas the inner layer has positive charge [30, 31]. The traditional polymeric microsphere, which is mainly made of gel, does not have an outer layer. Polymeric microspheres or the gel core is a viscoelastic plugging agent with 3D structure which can absorb much more water if compared to its own mass [35–37].

The core-shell microsphere has a core-shell double-layered structure. It swells with water, in which the core part swells fast, whereas the shell part expands relatively slowly. The core-shell microsphere can become a bipolar microsphere easily with positive and negative charges simultaneously by hydration [24, 32]. Consequently, multiple microspheres attract to each other, agglomerate, and gradually form into a string or group [24, 32]. Therefore, the size expansion mechanisms of the core-shell microsphere are agglomeration caused by electrostatic attraction and gel swelling, while mainly, the later one applies to the traditional polymeric microsphere.

In the profile control process, the initial and expanded sizes of core-shell microspheres need to match the pore throat sizes of porous media, in order to have remarkable

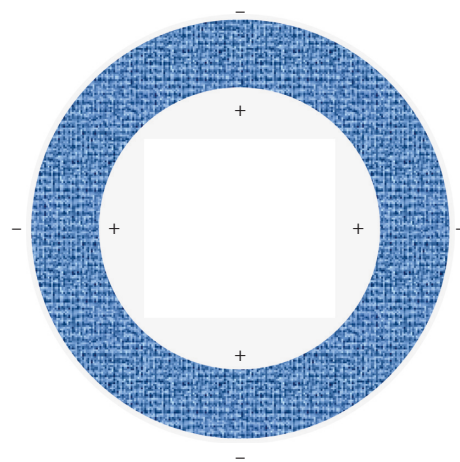


FIGURE 1: Schematic of the core-shell microsphere, which has a core-shell double-layered structure.



FIGURE 2: MICROTRAC S3500 laser particle size analyzer.

influence on pore blocking [8, 13, 31]. To find out the difference of hydrated sizes between novel core-shell microsphere and traditional polymeric microsphere, we design and perform the experimental studies on the size distribution evolution of these two types in Section 3.

2.2. Experiment Principle. The particle size analyzer, MICROTRAC S3500, is the key equipment employed in this study. It uses three precisely placed red laser diodes to accurately characterize particle sizes. The patented Tri-Laser System provides us accurate, reliable, and repeatable particle size analysis for a diverse range of applications. It utilizes the proven theory of Mie compensation for spherical particles and the proprietary principle of modified Mie calculations for nonspherical particles, respectively. The particle size analyzer can measure particle sizes from 0.02 to 2800 μm . In the experiments, polymeric and core-shell microspheres are baked at 70°C to simulate formation condition, and then their size distributions and variations are measured by the particle size analyzer.

3. Laboratory Study

In this section, we describe laboratory setup and materials (Section 3.1) and the procedure of microsphere size measurement (Section 3.2).

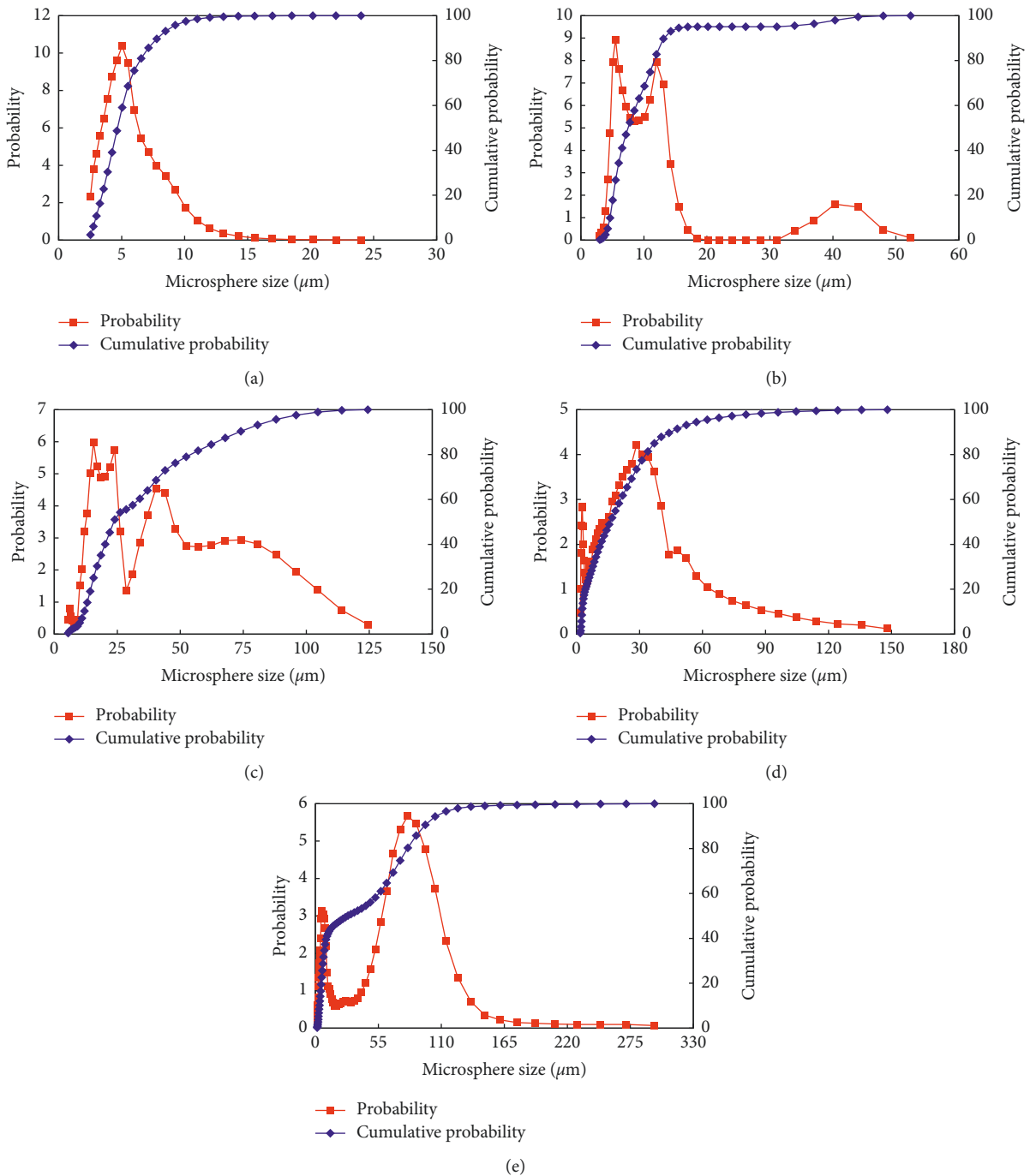


FIGURE 3: Polymeric microsphere size distribution at different periods of hydration: (a) 0 days, (b) 3 days, (c) 7 days, (d) 14 days, and (e) 21 days.

3.1. Laboratory Setup and Materials. In the experiments, the main equipment is the MICROTRAC S3500 laser particle size analyzer (Figure 2). The other tools include a separating funnel, a magnetic stirrer, analytical balance, and thermostat.

The particles used in the experiments are polymeric and core-shell microspheres, respectively. The polymeric microsphere is originated from polymeric nanoparticles with sizes around 300 nm. Because of initial agglomeration, the polymeric microsphere is formed with the average diameter 4.67 μm. The core-shell microsphere has the average diameter

of 8.13 μm. The total salinity of formation water in the test is 5863 mg/L.

3.2. Procedure of Laboratory Study. To measure size distributions and their variation of the two different microspheres, we design the procedure of the laboratory study as follows.

3.2.1. Microsphere-Dispersive Liquid Preparation. To prepare the particle-dispersive liquid with concentration 0.2%, the polymeric or core-shell microspheres are added into the

formation water gradually, keeping the rotating speed of magnetic stirrer at 500 rpm for 30 minutes.

3.2.2. Microsphere-Degreasing Treatment. Due to the limitation of synthesis technology, there are usually some oil and surfactants in the dispersive liquid. In order to observe and measure the microsphere size accurately, we propose the following degreasing operations:

- (1) Mix 600 mL of *N*-hexane into 300 mL particle-dispersive liquid. The volume ratio of *N*-hexane to microsphere-dispersive liquid is 2 : 1
- (2) Put the mixed solution into a conical flask airtight and then stir it for two hours by using the magnetic stirrer at 700 rpm
- (3) Move the stirred mixed solution into the separating funnel and leave it until there appear obvious two layers in the solution
- (4) Collect the lower layer solution, which is microsphere-dispersive liquid
- (5) Repeat steps (1)–(4) twice.

3.2.3. Initial Microsphere Size Measurement. Use a disposable pipette to aspirate a small amount of degreased microsphere-dispersive liquid and then measure the initial microsphere size by the particle size analyzer.

3.2.4. Microsphere Expansion Size Measurement

- (1) Place the degreased microsphere-dispersive liquid into a thermostat at 70°C, bake it, and then take samples at various times: 3 days, 7 days, 14 days, and 21 days
- (2) Put the samples on the magnetic stirrer and stir it continuously for 5 minutes
- (3) Use a disposable pipette to aspirate a small amount of degreased microsphere-dispersive liquid and then measure the microsphere expansion size distribution by the particle size analyzer.

4. Experimental Results

4.1. Polymeric Microsphere Size Distribution. Following the experimental procedure in Section 3, we obtained the polymeric microsphere size distribution at different periods of hydration, that is, 0 days, 3 days, 7 days, 14 days, and 21 days, as shown in Figure 3.

Under the conditions of constant salinity of 5863.27 mg/L and temperature of 70°C, we observed the multimodal distribution behavior in polymeric microsphere swelling process (Figure 3). The maximal probability of microsphere size is less than the initial value, that is, 10%.

The initial average diameter of the polymeric microsphere is 4.67 μm (Figure 3(a)). It results from agglomeration of polymeric nanoparticles [6, 9, 37]. After hydration for 3 days, it increases 2.04 times to 9.41 μm (Figure 3(b)). After 7 days, it increases to 25.57 μm (Figure 3(c)), expanding by

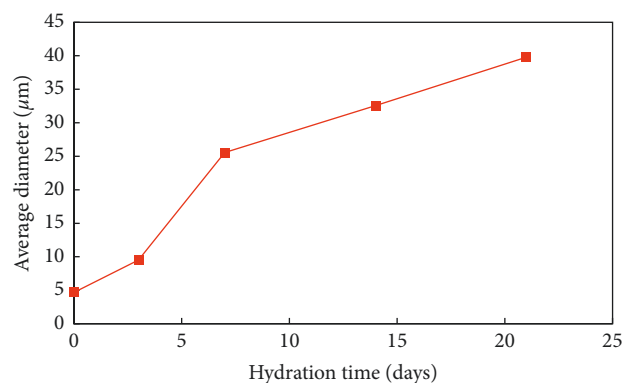


FIGURE 4: Average diameter of polymeric microsphere versus hydration time.

5.41 times. Since then, the polymeric microsphere keeps constant expansion velocity (Figure 3(d)). On hydration time of 21 days, the average diameter continues to increase to 40.33 μm by 8.6 times, compared with the initial size (Figure 3(e)). The size distribution of polymeric microspheres may be used to calculate the fractal dimension [38, 39], which can then be applied to evaluate the agglomeration degree at different periods of hydration [40].

The average diameter of polymeric microsphere increases monotonically (Figure 4). The polymeric microsphere sizes increase relatively slowly in the early stage, that is, 0–3 days, which coincides with the results from previous research [24]. This is because the polymeric microsphere needs time to unfold the polymer structure before the agglomeration. From the point of view of profile control, the polymeric microsphere can be injected into low permeability formation easily because of its small initial size. Moreover, the polymeric microsphere is able to filtrate into deep formation because of its slow expansion speed in the early stage.

4.2. Core-Shell Microsphere Size Distribution. Following the experimental procedure in Section 3, we also obtained the core-shell microsphere size distribution at different periods of hydration, that is, 0 days, 3 days, 7 days, 14 days, and 21 days, as shown in Figure 5.

Under the conditions of constant salinity of 5863.27 mg/L and temperature of 70°C, we observed the unimodal distribution behavior in the core-shell microsphere expansion process (Figure 5). The maximal probability of microsphere size increases with time, which is different from the polymeric microsphere behavior shown in Figure 3. This difference indicates that the agglomeration effect of core-shell microsphere is stronger than that of the polymeric microsphere.

The initial average diameter is 8.13 μm (Figure 5(a)) and then expands 4.00 times to 32.48 μm after hydration for 3 days (Figure 5(b)). It increases to 53.00 μm after 7 days (Figure 5(c)), expanding by 6.50 times. Before hydration time of 7 days, the core-shell microsphere has constant expansion velocity, which is higher than that during 7–21 days. On hydration time of 21 days, the average

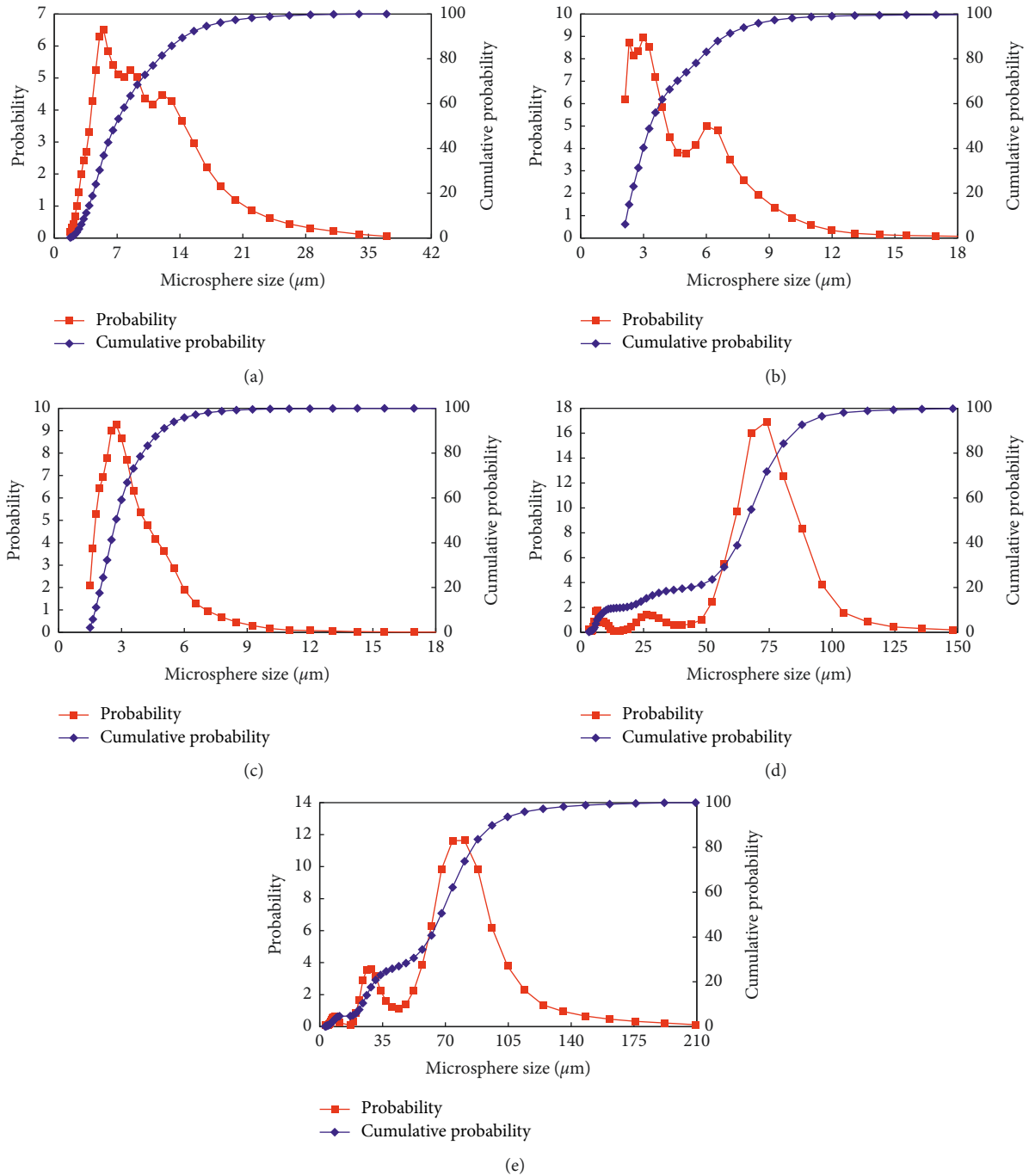


FIGURE 5: Core-shell microsphere size distribution at different periods of hydration: (a) 0 days, (b) 3 days, (c) 7 days, (d) 14 days, and (e) 21 days.

diameter continues to increase to 63.81 μm by 7.8 times, compared with the initial size (Figure 5(e)).

The average diameter of the core-shell microsphere increases monotonically (Figure 6). As shown in Figures 5 and 6, the core-shell microsphere has different swelling velocities and average diameter from the polymeric microsphere. First, the core-shell microsphere has higher expansion velocity than the polymeric microsphere in the early stage, that is, 0–3 days. Second, the core-shell microsphere has relatively larger average diameter than the polymeric microsphere. These two key features indicate

that we can use the core-shell microsphere for near-wellbore zone blockage, whereas the polymeric microsphere for deep formation blockage. Based on the X-ray computed tomography (CT), 3D digital core structure models [41] can be developed to evaluate the applicability of different types of microspheres.

4.3. Comparison between Two Types of Microspheres. Both the polymeric and core-shell microspheres have micron-scale diameters. With the increasing hydration time, the

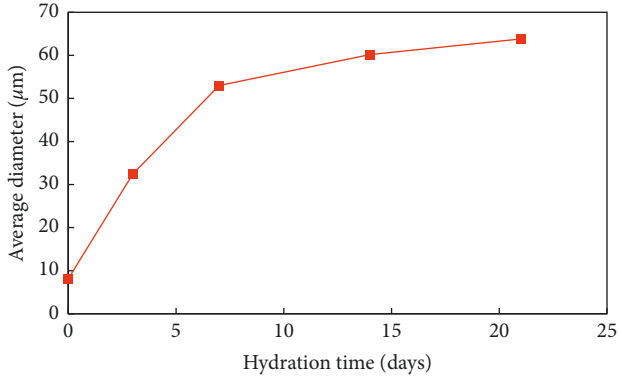


FIGURE 6: Average diameters of core-shell microspheres versus hydration time.

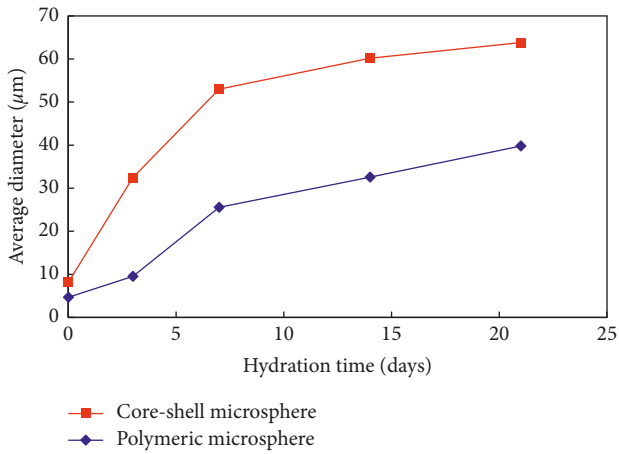


FIGURE 7: Comparison of average diameter evolution between polymeric and core-shell microspheres.

sizes of two types of microspheres increase gradually. However, there are two main differences between them.

Compared with the size distribution of polymeric microsphere (Figure 3), the size distribution of core-shell microsphere is narrower (Figure 5) due to positive and negative electrostatic interactions between different microspheres. In synthesis of core-shell microspheres, the shell part is mainly made from acrylamide and anion monomer, whereas the core part is mainly made from acrylamide and cation monomer [42]. The 3D microscopic graphs show that the core-shell microspheres swell to different sizes, and the smaller ones will be attached around the bigger ones [43], which indicate that there is an electrostatic interaction between different microspheres.

The average diameter of the core-shell microsphere increases faster than that of the polymeric microsphere in the hydration time of 0–3 days (Figure 7). The expansion velocity of the polymeric microsphere mainly shows the original swelling velocity which is caused by polymer gel swelling. The higher expansion velocity of the core-shell microsphere in 0–3 days results from the electrostatic attraction between core-shell microspheres with different

TABLE 1: Coefficients by matching average diameters.

Particles	d_{i0} (μm)	d_{imax} (μm)	a_{sh}	c_0	c_1	c_2	n
Polymeric microspheres	4.67	56	0.10	18	8	5	3
Core-shell microspheres	8.13	81	0.18	-19	46	-0.1	5

hydration degrees. Therefore, we conclude that the electrostatic interaction mainly acts in the early stage, that is, 0–3 days for the core-shell microsphere.

Apparently, the average diameter calculation plays an important role on microsphere optimization and field application design. In reservoir-scale numerical simulation, a mathematical model is necessary to describe the evolution of the microsphere size. In the next section, we apply a simple model to match the curves in Figure 7 and evaluate the model feasibility on two types of microspheres.

5. Mathematical Models for Average Diameter Evolution

Based on the experimental results, we apply the traditional mathematical model (1) to describe average diameter variation versus hydration time. If the hydration time does not exceed the critical time t_{wc} , the average diameter increases with hydration time. The average diameter is calculated as follows:

$$d_i = d_{i0} + (d_{imax} - d_{i0}) \frac{a_{sh} t_w}{1 + a_{sh} t_w}, \quad (1)$$

where d_i is the microsphere average diameter at time t_w , d_{i0} is the initial average diameter, d_{imax} is the maximal average diameter, t_w is the hydration time, and a_{sh} is the coefficient based on experimental data.

Apparently, there is accelerated size evolution in the hydration process of polymeric microspheres (Figure 7). Accordingly, we present a new mathematical model (2) to describe this feature:

$$d_i = c_0 - c_1 (c_2 - t_w)^{1/n}, \quad (2)$$

where c_0 , c_1 , and c_2 are coefficients based on experimental data.

By matching the curves in Figure 7 with (1) and (2), respectively, we obtain the corresponding coefficients, which are shown in Table 1.

Figure 8(a) indicates that the traditional mathematical model (1) yields a good fitting with measured data for the core-shell microsphere throughout the expansion process. The slow-expansion feature of the polymeric microsphere in the early stage (0–3 days) cannot be captured by the model. Therefore, in the average diameter calculation, the traditional model (1) is applicable to the core-shell microsphere but not to the polymeric microsphere.

In comparison, Figure 8(b) illustrates that the proposed model (2) can capture the size evolution behavior not only for core-shell microspheres but also for polymeric microspheres.

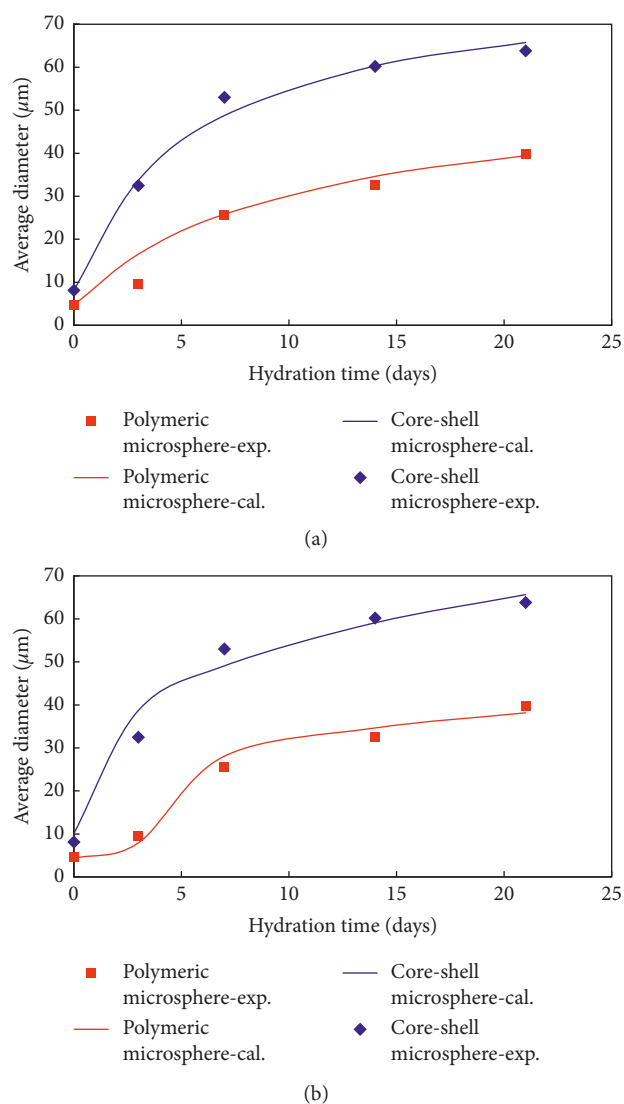


FIGURE 8: Matching curves of microsphere average diameters by (a) traditional model (1) and (b) proposed model (2).

6. Conclusions

Experimental study on expansion characteristics of two types of microspheres leads to the following conclusions:

- (1) The size distribution evolution of the core-shell microsphere is different from that of the polymeric microsphere. The core-shell microsphere exhibits unimodal distribution, dominated by the agglomeration effect. The polymeric microsphere exhibits multimodal distribution, resulting from the swelling effect.
- (2) The expansion process of polymeric microspheres can be divided into two stages. In the early stage, that is, 0–3 days, it swells slowly as it needs time to unfold the polymer structures. In the late stage, that is, 7–21 days, it swells faster because of the agglomeration between different microspheres.

- (3) The traditional mathematical model is applicable to average diameter evolution of the core-shell microsphere but not to the polymeric microsphere in the early stage of hydration. The proposed model can be applied for both core-shell and polymeric microspheres.
- (4) The core-shell microsphere has larger initial size and expands faster than the polymeric microsphere. Therefore, the core-shell microsphere is suitable for injection in the early stage to block the near-wellbore zone, and the polymeric microsphere is suitable for subsequent injection to block the formation away from the well.

Data Availability

The datasets used to support this study are currently under embargo while the research findings are commercialized. Requests for data, 12 months after initial publication, will be considered by the corresponding author.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

Acknowledgments

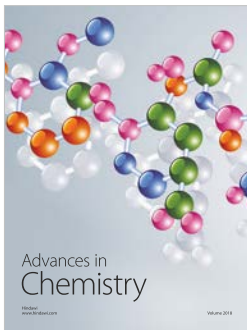
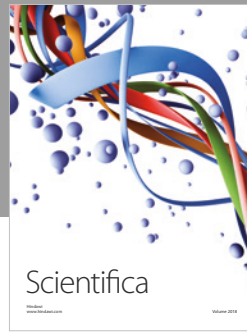
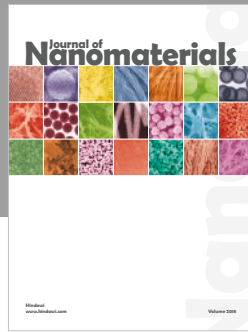
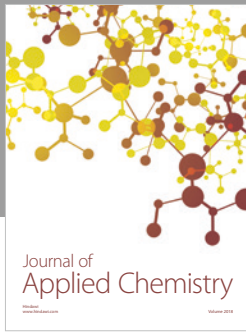
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