

A Method for Rapid Determination of Polymer Dissolution Time

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Abstract

In view of the limited space of offshore platform and the large amount of polymer injection, the dissolution time method, solution viscosity method, conductivity method, undissolved filtration method and falling ball method are used as the rapid detection methods of polymer dissolution. The results show that the error of conductivity method is the largest, and that of dissolution time method is the second, and both methods are time-consuming. Solution viscosity method and insolubles filtration method have small error, but they are time-consuming, especially for offshore platforms, which makes detection inconvenient. The drop ball method is simple in operation and has a small error, so it is a promising method for rapid determination of polymer dissolution time.

Keywords

Rapid Determination, Dissolution Time, Viscosity Method, Falling-Ball Method

1. Introduction

Bohai oilfield belongs to offshore heavy oil field, and its conventional heavy oil reserves account for about 70% of the total offshore reserves [1]. With the need of stable production and increasing production of offshore oil fields in China, the research and application of tertiary oil recovery technology in Bohai oilfield has been accelerated. The mechanism of polymer flooding [2] [3] [4] [5] is to improve the oil-water mobility ratio, and effectively improve the sweep efficiency of displacement phase, which reduces oil saturation, to achieve the improvement of oil recovery. Since 2003, the polymer flooding pilot test has been carried out in Bohai oilfield [6] [7], which has achieved good effect of precipitation and

oil increase. It has become one of the important means for stable production and production increase of SZ36-1, LD10-1, JZ9-3 and other offshore heavy oil fields. With the expansion of polymer flooding scale, the demand for platform space increases. In order to reduce the demand for platform space, it is necessary to study the efficient polymer injection technology, improve and break through the bottleneck of chemical flooding supporting technology [8] [9]. The dissolution degree of polymer solution can be defined as the ratio percentage of the amount of polymer dissolved in the solution to the total amount of polymer at a certain time. The detection of this parameter is the prerequisite for polymer instant dissolution experiment. Conventional experimental methods need viscometers and other instruments, and the aging is poor. Especially for offshore oil fields, field detection is easier to operate than land oil fields with higher timeliness [10]. In this paper, through the study of polymer dissolution time method, polymer solution viscosity method, undissolved filter method, conductivity method, falling ball method, etc., a feasible and simple method for rapid detection of polymer dissolution degree is established.

2. Experiments

2.1. Experimental Chemicals

The hydrophobical associating polymer: marked as AP-P4 molecular weight 1250×10^4 , solid content 90%.

For the experimental water, we prepared the synthetic brine according to the current polymer flooding situation of the oilfield, with the composition is shown in **Table 1**.

2.2. Experimental Instruments

Brookfield DV-III viscometer, DDS-307 conductivity meter (Shanghai Leici), QNQ falling ball viscometer (Shanghai Meiyu Instrument Equipment Co., Ltd.), digital suspension arm mixer (Jiangyin poly scientific research equipment Co., Ltd.), timer, 20 ml pipette, syringe (10 ml, needle removal head).

2.3. Experimental Methods

1) Dissolution time method

5000 mg/L polymer mother liquor was prepared at 45°C, and the stirring speed was 200 r/min. In the preparation process, determine the viscosity of the preparation solution at different times (once every 5 min), record the viscosity value until the viscosity changes little in the three sampling tests, stop sampling, draw the curve of polymer dissolution degree with the dissolution time, and calculate the dissolution degree according to the dissolution time discrimination method.

Table 1. The composition of synthetic brine.

Composition	K ⁺ + Na ⁺	Mg ²⁺	Ca ²⁺	Cl ⁻	SO ₄ ²⁻	HCO ₃ ⁻	CO ₃ ²⁻	Total salinity
content/(mg/L)	3091.96	158.68	276.17	5436.34	85.29	311.48	14.21	9374.12

a) Absolute time discriminant expression

$$R_1 = \frac{t_j}{t_2} \times 100\% \quad (1)$$

R_1 —Dissolution degree of relative time discrimination, %;

t_2 —The basic dissolution time, min;

t_j —The time point of need to determine dissolution degree, min, $t_1 \leq t_j \leq t_2$.

b) Relative time discriminant expression

$$R_2 = \frac{t_j - t_1}{t_2 - t_1} \times 100\% \quad (2)$$

R_2 —Dissolution degree of relative time discrimination, %;

t_1 —Complete swelling time, min;

t_2 —The basic dissolution time, min;

t_j —The time point of need to determine dissolution degree, min, $t_1 \leq t_j \leq t_2$.

2) Solution viscosity method

5000 mg/L polymer mother liquor was prepared at 45°C, and the stirring speed was 200 r/min. In the preparation process, determine the viscosity of the prepared solution at different times (once every 5 min), record the viscosity value until the viscosity of the three sampling tests changes little, stop sampling, and draw the curve of the viscosity of the polymer solution with the dissolution time. According to the characteristics of the curve, the viscosity μ_{t_1} at the time of complete swelling and μ_{t_2} at the time of basic dissolution are determined. It is necessary to determine the viscosity μ_{t_j} at the time point of dissolution.

a) The expression of absolute viscosity discriminant

$$R_3 = \frac{\mu_{t_j}}{\mu_{t_2}} \times 100\% \quad (3)$$

R_3 —Solubility of relative viscosity, %;

μ_{t_2} —Viscosity at basic dissolution, mPa·s;

μ_{t_j} —Viscosity at the time point where the degree of dissolution needs to be determined, mPa·s.

b) The expression of relative viscosity discriminant

$$R_4 = \frac{\mu_{t_j} - \mu_{t_1}}{\mu_{t_2} - \mu_{t_1}} \times 100\% \quad (4)$$

R_4 —Solubility of relative viscosity, %;

μ_{t_1} —Viscosity at full swelling, mPa·s;

μ_{t_2} —Viscosity at basic dissolution, mPa·s;

μ_{t_j} —Viscosity at the time point where the degree of dissolution needs to be determined, mPa·s.

3) Conductivity method

At 45°C, prepare 5000 mg/L polymer mother liquor, and test the viscosity and conductivity changes of the mother liquor at different dissolution times until the viscosity of the polymer mother liquor tends to be stable. The viscosity and conductivity of polymer solution are tested every five minutes to ensure that the

viscosity and conductivity data can correspond at the same time. According to the change curve of viscosity conductivity, the change trend is analyzed to judge the change of solubility. The following formula shall be used to calculate the dissolution degree of solution at different dissolution times:

$$R_5 = \frac{k_t - k_0}{k_f - k_0} \times 100\% \quad (5)$$

R_5 —Solubility determined by conductivity method, %;

k_t —Conductivity of solution at a certain time, $\Omega^{-1}\cdot\text{cm}^{-1}$ or $\text{mS}\cdot\text{cm}^{-1}$;

k_0 —Conductivity of solvent, $\Omega^{-1}\cdot\text{cm}^{-1}$ or $\text{mS}\cdot\text{cm}^{-1}$;

k_f —Conductivity of solution when viscosity is stable, $\Omega^{-1}\cdot\text{cm}^{-1}$ or $\text{mS}\cdot\text{cm}^{-1}$.

4) Insolubles filtration

The experimental steps are as follows: a) prepare 5000 mg/L polymer mother liquor at 45°C; b) weigh the mass of empty syringe (10 ml) m_1 ; c) use 10 ml syringe to take 10 ml of prepared and stirred polymer solution, and call its mass m_2 ; d) inject M2 polymer solution onto 120 mesh screen, stand for about 10 min, and then extract the remaining polymer solution on the screen, and call its mass m_3 ; e) calculate the content of undissolved micelles; f) take samples every 10 minutes to ensure that they are synchronized with the viscosity measurement; g) when the content of undissolved substances changes little, end the sampling.

5) Drop ball method

Based on Stokes formula, the method can determine the degree of polymer dissolution by measuring the falling time of small ball in solution.

$$R_6 = \frac{t_s}{t_0} \times 100\%$$

R_6 —Dissolution degree determined by falling ball method, %;

t_0 —Falling time of steel ball when polymer dissolves completely, min;

t_s —Falling time of steel balls at different dissolution time points, min, $t_s \leq t_0$.

3. Results and Analysis

3.1. Dissolution Time Method

According to the experimental steps of dissolution time method in 1.3, the change curve of polymer dissolution degree with dissolution time is drawn, as shown in **Figure 1**. It can be seen from **Figure 1** that with the increase of dissolution time, the dissolution degree of polymer dry powder increases gradually, and the change rate of dissolution degree with the dissolution time is certain. The dissolution degree measured by absolute time method is larger than that measured by relative time method, but the difference of dissolution degree calculated by the two methods is gradually reduced.

3.2. Solution Viscosity Method

According to the experimental procedure of solution viscosity method in 1.3, the change curve of polymer solution viscosity with solution time is drawn, as shown in **Figure 2**. It can be seen from **Figure 2** that the longer the dissolution

time is, the higher the dissolution degree of polymer dry powder particles is. The dissolution degree measured by absolute viscosity method is greater than that measured by relative viscosity method, and the difference between the dissolution degrees calculated by the two methods gradually decreases with the increase of dissolution time.

3.3. Conductivity Method

According to the experimental steps of conductivity method in 1.3, the viscosity and conductivity value change during polymer dissolution are measured, and the results are shown in **Figure 3**. It can be seen from **Figure 2** that the viscosity of the polymer increases in the dissolution process. The viscosity rises slowly before 25 min, and rapidly between 25 min and 55 min. After 55 min, the viscosity tends to be stable and fluctuates slightly. The conductivity of the polymer increases with time in the dissolution process, and increases rapidly before 30 min, and then tends to be stable after 30 min.

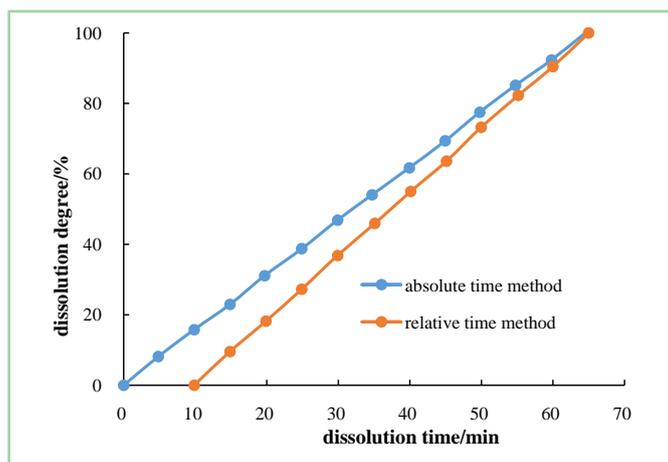


Figure 1. Curve of polymer dissolution degree with time measured by dissolution time method.

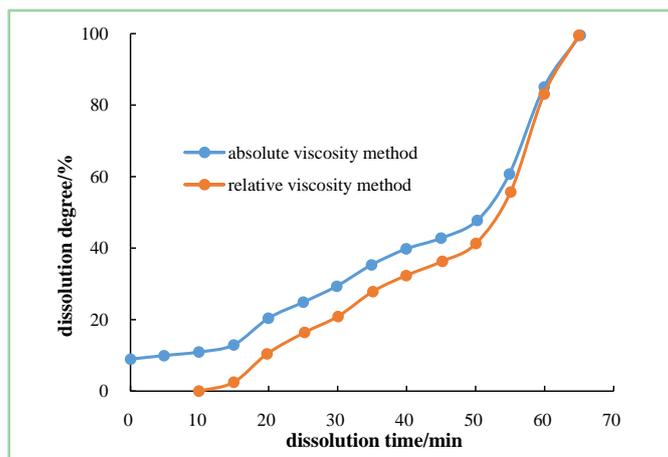


Figure 2. Change curve of polymer solubility with time measured by solution viscosity method.

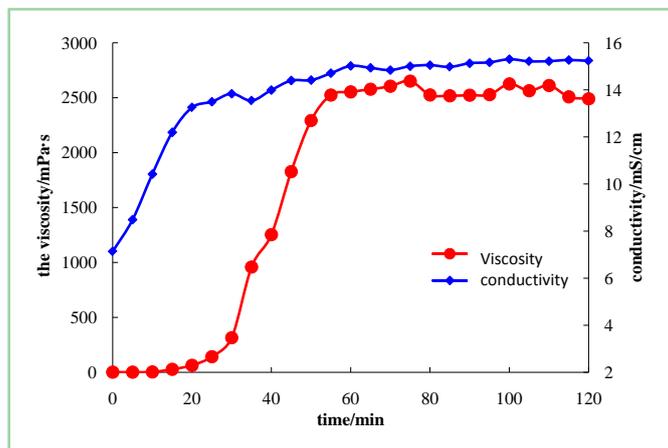


Figure 3. Change curve of polymer solubility viscosity and conductivity.

3.4. Insolubles Filtration

According to the experimental steps of conductivity method in 1.3, determine the change curve of viscosity and insoluble content in the dissolution process, as shown in **Figure 4**. It can be seen from **Figure 4** that the content of no-swelling micelle increases with the increase of viscosity of polymer solution. When the viscosity of polymer solution tends to be stable, the content of insoluble substance also tends to be stable, basically unchanged, and the change trend basically conforms to the change trend of viscosity of polymer solution.

3.5. Drop Ball Method

According to the experiment steps of falling ball method in 1.3, determine the viscosity of mother liquor and the curve of time s and dissolution time of falling ball, as shown in **Figure 5**. It can be seen from **Figure 5** that the falling time of the small ball in the initial stage is very small and basically remains unchanged. In this stage, the polymer is in the swelling stage and the viscosity of the solution is low. After the initial stabilization, the falling time of the pellets increased rapidly, and the viscosity of the solution increased rapidly as the polymer entered the dissolution stage. After the rising stage, the falling time of the small ball tends to be stable. At this time, the polymer enters the aging stage, and the polymer gradually dissolves from the basic solution to the complete solution. The viscosity of the polymer solution rises slowly, and the swelling particles of the polymer finally almost completely dissolve, and the viscosity of the solution tends to be stable. It can be seen from the figure that the dissolution time measured by viscosity method and ball drop method is closer.

The results of the above five methods are summarized in **Table 2**. It can be seen from **Table 2** that the falling ball method is simple in operation, with relatively small experimental error. The change trend of the falling time of the small ball is the same as that of the viscosity of the polymer solution. In addition, the falling ball viscometer is easy to use and carry, so the falling ball viscometer can be used to measure the solubility of the polymer quickly and conveniently.

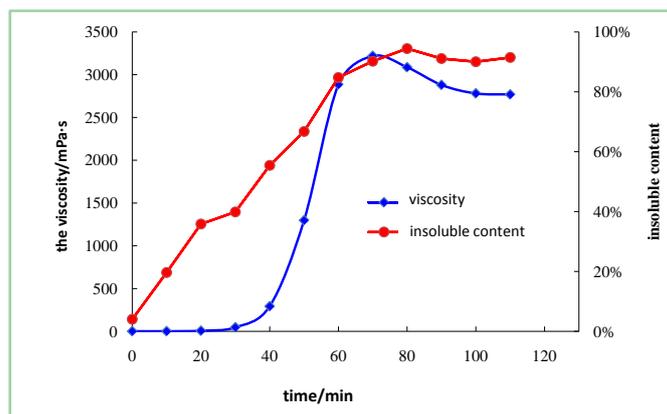


Figure 4. Dissolution curve of polymer by insoluble filtration.

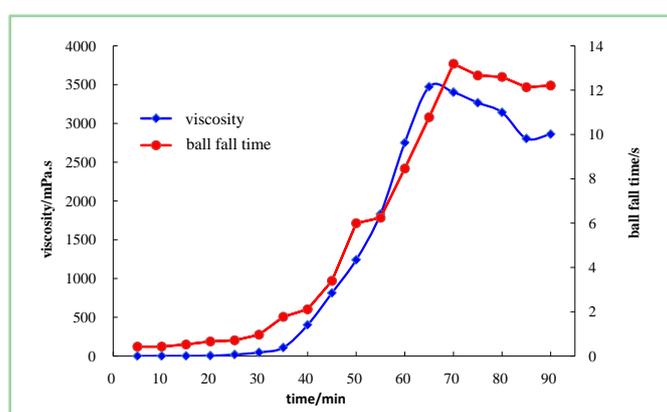


Figure 5. Polymer dissolution curve by falling ball method.

Table 2. Comparison of advantages and disadvantages of five methods.

method	dissolution time/min	determination of dissolution time by viscosity method/min	characteristic
dissolution time method	65	50	Large error and time consuming
solution viscosity method	50	50	It's viscosity method in itself, so it's time-consuming to measure the precise experiment
conductivity method	30	55	Large error and time consuming
insolubles filtration	65	60	Small error and time consuming
drop ball method	70	65	Simple operation and small error

4. Conclusions

1) With the increase of dissolution time, the dissolution degree of polymer powder particles increases gradually, and the change rate of dissolution degree with dissolution time is certain. The dissolution degree measured by absolute time method is larger than that measured by relative time method, but the difference between the two methods is gradually reduced.

2) The longer the dissolving time is, the higher the dissolving degree of polymer powder particles is. The dissolving degree measured by absolute viscosity method is larger than that measured by relative viscosity method, and the difference of dissolving degree calculated by the two methods gradually decreases with the increase of dissolving time.

3) Compared with the result of viscosity method, the result of conductivity method has a large error. This method is not easy to operate and takes a long time, so it is not the best method.

4) The error of the insoluble-filtration method is small, but the operation is time-consuming.

5) The tendency of the falling time of the small ball is the same as that of the viscosity of the polymer solution. The falling ball viscometer is easy to use and carry, so the falling ball method is a more promising method for rapid determination of polymer dissolution time.

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Conflicts of Interest

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

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