

# Study on Assessment Method for Heat Resistance of Heat-Resistant Polymer Admixture for well Cementation Based on Ubbelohde Viscosity Meter

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**Abstract.** One important reason why it is hard to ensure quality of high-temperature deep well cementation is that it is hard to ensure the comprehensive performances of cement paste at a high temperature. So far, in most cases, admixtures are used to adjust the high-temperature performances of cement paste, and such admixtures are dominated by polymers. At a relatively high temperature, the settlement stability and fluid loss of cement paste are regulated by fluid loss agent, and thickening time is regulated by retarder. The deeper the well is, the higher the temperature and pressure at the well bottom will be. It is hard to regulate the settlement stability, fluid loss and thickening time of cement paste at a high temperature. Therefore, it is hereby to recommend an assessment method for heat resistance of polymer admixture, to facilitate the optimal selection of polymer admixture.

**Keywords:** Polymer at High Temperature, Methods, Assessment Results and Analysis.

## 1. Introduction

The settlement stability of cement paste at a high temperature is not only related to the construction safety for deep well cementation, but also is an important factor[1-3] affecting the quality of well cementation and inducing fluid channeling. Moreover, cement paste will rapidly thicken at a high temperature and pressure, which will greatly shorten the pumping time of cement paste, to further affect the quality of well cementation and subsequent drilling and cementing works, or, in serious cases, to further result in scrapping of the entire well, bringing about huge economic loss. Therefore, it needs to optimally select the polymer admixture to reduce the failure at a high temperature. By now, there has been no unified understanding on assessment for heat resistance of cement polymer admixture for oil well. In most cases, cement paste's performance at a high temperature is assessed to reflect its heat resistance. In this paper, based on the high polymer experimental technique and through analysis of change in "viscosity-molecular weight" of polymer admixture, the heat resistance of polymer admixture is assessed as an assessment method for optimal selection of heat-resistant polymer admixture.

## 2. Analysis of Functions and Failure Mechanism of Polymer

### 2.1 A Subsection Settlement Stability and Retarding Mechanism at High Temperature

Currently, in view of the settlement stability<sup>[4]</sup> of cement paste at a high temperature, the studies are focusing on the stress and mutual coalescence of cement particles, and repulsive potential in space added at high polymer adsorption layer, and have formed the understanding on 3 aspects being kinetic stability, coalescence stability and flocculation stability. in view of retarding mechanism<sup>[6]</sup> of cement paste at a high temperature, the studies are focusing on the inhibitions of retarder to cement particles, C-S-H gel and Ca<sup>2+</sup>, Al<sup>3+</sup> etc. in liquid phase, and have formed an adsorption theory, a complexing theory, a settling theory and a nucleation theory.

## 2.2 Analysis of Failure Mechanism of Polymer at High Temperature

At a high temperature, organic polymer fluid loss agent can go through a degradation reaction<sup>[5]</sup> in 3 ways including depolymerization, random degradation and group desorption, so that its molecular weight will drop, which will reduce the system viscosity and lower the settlement stability. In addition, the hydrogen bonds connecting polymer molecules in fluid loss agent will be ruptured, so such molecules become free. The flexibility of molecular chain will also be increased, so such molecular chain will gradually become extended, which will aggravate the drop in system viscosity.

Retarder is adsorbed on the surfaces of cement particles, and, as temperature rises, the adsorption quantity of organic matters on surfaces of particles will be gradually dropping. Moreover, the higher the temperature is, the poorer the chelation of retarder is, which will result in a degradation in retarding effect of retarder. The higher the temperature is, the lower the molecular weight of the retarder is. As the molecular weight of retarder drops, the retarder's form of adsorption will be changing, which will result in inhibition to hydration of cement, or even no coagulation of cement paste for a long time.

## 3. Assessment Method on Heat Resistance of Polymer

### 3.1 Materials and Apparatuses for Experiment

Main reagents: polymer fluid loss agents (1, 2 and 3), polymer retarders (1, 2 and 3).

Main apparatuses: Ubbelohde viscosity meter (model 1836), provided by Shanghai Liangjing Glassware Plant; electro-thermostatic blast drying oven (model 101), provided by Beijing Ever Bright Instrument Co., Ltd.; thermostat water bath, provided by Applied Technology Research Institute of Shenyang Aerospace University.

### 3.2 Principle of Experiment

The heat resistance of polymer is related to its molecular structure. It can be seen from failure analysis at a high temperature of polymer that degradation, chain scission and group desorption of polymer admixture at a high temperature can all result in drop in molecular weight and viscosity. Under this principle, assessment for heat resistance of polymer admixture is carried out with high polymer experimental technique.

One of the basic characteristics of linear high polymer solution is that the viscosity is related to average molecular weight, and is used to determine the molecular weight<sup>[7]</sup>. Viscosity is not only closely related to molecular weight, but also greatly relies on the concentration of solution. Therefore, influence of concentration on viscosity needs to be primarily removed in experiment. Generally, the dependence of viscosity on concentration is expressed in the following two empirical equations:

$$\frac{\eta_{sp}}{c} = [\eta] + k'[\eta]^2c \quad (1)$$

$$\frac{\ln \eta_r}{c} = [\eta] - \beta[\eta]^2c \quad (2)$$

Where  $\eta_{sp}$  is specific viscosity;  $\eta_r$  is relative viscosity. Assuming that  $\eta_0$  represents the viscosity of solvent and  $\eta$  represents the viscosity of solution,

$$\eta_r = \frac{\eta}{\eta_0} \quad (3)$$

$$\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} = \eta_r - 1 \quad (4)$$

In (1) and (2),  $c$  represents concentration of solution;  $k'$  and  $\beta$  are both constants. Evidently,

$$\lim_{c \rightarrow 0} \frac{\eta_{sp}}{c} = \lim_{c \rightarrow 0} \frac{\ln \eta_r}{c} = [\eta] \quad (5)$$

$[\eta]$  is the intrinsic viscosity of polymer solution, and unrelated to concentration. It can thus be seen that if curve of  $c$  is drawn respectively from  $\eta_{sp}/c$  and  $\ln \eta_r/c$ , their intercepts extrapolated to  $c \rightarrow 0$  shall be overlapped at some point at which the value is equal to  $[\eta]$ . Upon determination of chemical compositions, solvent and temperature of polymer, the value of  $[\eta]$  is only related to molecular weight of polymer, which is generally expressed in the equation below:

$$[\eta] = KM^\alpha \quad (6)$$

The flow of liquid under gravity in capillary viscosity meter can be expressed in the equation below:

$$\eta = Apt - \frac{B\rho}{t} \tag{7}$$

Where A and B are constants of apparatus; with (7) put into (3):

$$\eta_r = \frac{\rho}{\rho_0} \cdot \frac{At - B/t}{At_0 - B/t_0} \tag{8}$$

In case that apparatus used is properly designed and the right solvent is selected, (8) can be simplified to:

$$\eta_r = \frac{\rho}{\rho_0} \cdot \frac{At}{At_0} = \frac{\rho t}{\rho_0 t_0} \tag{9}$$

Also because the determination of viscosity of polymer solution is generally carried out at a very low concentration ( $c < 0.01 \text{ g}\cdot\text{mL}^{-1}$ ), solution and solvent have the similar densities,  $\rho \approx \rho_0$ . Therefore, equations (3) and (4) can be modified to:

$$\eta_r = \frac{t}{t_0} \tag{10}$$

$$\eta_{sp} = \eta_r - 1 = \frac{t - t_0}{t_0} \tag{11}$$

Where  $t, t_0$  respectively represent the efflux times of solution and pure solvent.

Polymer solution is diluted to determine the efflux times corresponding to different concentrations of solution. Based on equations (1), (2), (10) and (11), value of  $[\eta]$  is obtained through extrapolation of concentration, which is referred to as the “extrapolation method”. Change  $\Delta M$  in value  $[\eta]$  of polymer before and after the thermal treatment is calculated. The closer the absolute value of  $\Delta M$  to 0 is, the smaller the change in molecular weight (structure) of solvent before and after thermal treatment is, and the better the heat resistance is.

$$\Delta M = \frac{M_{\text{after}} - M_{\text{before}}}{M_{\text{before}}} = \frac{[\eta]_{\text{after}}^{\frac{1}{\alpha}} - [\eta]_{\text{before}}^{\frac{1}{\alpha}}}{[\eta]_{\text{after}}^{\frac{1}{\alpha}}} \times 100\% \tag{12}$$

### 3.3 Experiment Methods

1. Washing of Glassware Viscosity meter is washed with deionized water first and then dried for use.
2. Preparation of Sample Fluid loss agents (1, 2 and 3) and retarders (1, 2 and 3) are placed into a roller oven and thermally treated at  $180^\circ \text{ C}$  for 3 hours, then cooled and taken out for use.
3. Measurement of Efflux Times of Solution Thermostatic bath is adjusted to  $30 \pm 0.1^\circ \text{ C}$ . Rubber tubes are connected to tubes B and C of viscosity meter. Viscosity meter is clipped by an iron clamp and then put into the thermostatic bath, where capillary tubes are kept vertical to water surface, and ball above line a is immersed under such water surface. 10mL of solvent are filled from tube A by use of a pipette. After temperature has become stable for 10 min, rubber tube connected with tube C is clipped with a clamp for stuffiness, while rubber tube connected with tube B is exhausted by a syringe, until the solvent surface drops to the level half of the height of the ball above the line a. Syringe is removed and clamp is released from tube C so that air can enter ball D, and solvent in capillary tube can be separated from the ball at the bottom of tube A. Time for level liquid surface to flow through line a and line b is recorded, i.e.  $t_0$ . This operation is repeated for more than three times, with an error not more than 0.2s. Average value is taken as  $t_0$ .

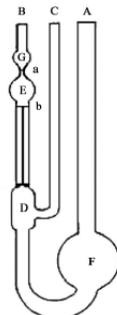


Fig. 1 Ubbelohde viscosity meter

.4 Preparation of Sample Preparation of Solution 1mL of sample is weighed and filled into 100mL of volumetric flask. Deionized water is filled to required scale, such flask is shaken up for dissolution, and then placed into a thermostatic bath.

.5 Measurement of Efflux Times of Solution 10mL of solution are filled into the viscosity meter by a pipette, and efflux time  $t_1$  of solution is measured. After this, 5mL of deionized water is filled, in this case, concentration of solution in viscosity meter is 2/3 of the former one. Viscosity meter is shaken up and  $t_2$  is measured in the same way. In the same way, 5mL, 10mL and 10mL of deionized water are respectively filled to measure  $t_3$ ,  $t_4$  and  $t_5$ .

#### 4. Assessment Results and Analysis of Heat Resistance

The initial concentration of solution is set to  $c_0$ , the actual concentration is  $c = c'c_0$ , and the relative concentrations after 5mL, 5mL, 10mL and 10mL of deionized water are respectively filled in sequence are 2/3, 1/2, 1/3 and 1/4 (expressed by  $c'$ ), then  $\eta_r, \ln\eta_r, \ln\eta_r/c', \eta_{sp}$  and  $\eta_{sp}/c'$  are calculated. Curve of  $\ln\eta_r/c'$  to  $c'$  is drawn and intercept A is obtained via extrapolation, and then: intrinsic viscosity is  $[\eta] = A/c_0$ .

##### 4.1 Analysis of Heat Resistance of Fluid Loss Agent at High Temperature

Efflux times of water solution corresponding to five concentrations (relative concentrations: 1, 2/3, 1/2, 1/3 and 1/4) of fluid loss agents (1, 2 and 3) before and after the thermal treatment are measured, to calculate  $\ln\eta_r/c'$  corresponding to different concentrations, and then the relative concentration  $c'$  of solution is taken as X-coordinate and  $\ln\eta_r/c'$  is taken as Y-coordinate to draw the relational diagram of  $c', \ln\eta_r/c'$ . The test results of efflux times of fluid loss agent 1 (before and after thermal treatment) are shown in Table 1 and 2:

Table 1 Test results of efflux times of fluid loss agent 1 solution (before thermal treatment at 180°C)

Fluid Loss Agent 1	Efflux Time (s)				$\eta_r$	$\ln\eta_r$	$\ln\eta_r/c'$	$\eta_{sp}$	$\eta_{sp}/c'$
	1	2	3	Average					
t0	85	86	86	85.66667	1	0		0	
t1(c=c0)	810	807	808	808.3333	9.435794	2.24451	2.24451	8.435794	8.435794
t2(c=2/3c0)	635	629	627	630.3333	7.357974	1.995785	2.993677	6.357974	9.536961
t3(c=1/2c0)	527	525	524	525.3333	6.132293	1.813569	3.627138	5.132293	10.26459
t4(c=1/3c0)	449	449	448	448.6667	5.237352	1.655816	4.967448	4.237352	12.71206
t5(c=1/4c0)	343	341	338	340.6667	3.976652	1.38044	5.521761	2.976652	11.90661

Table 2 Test results of efflux times of fluid loss agent 1 solution (after thermal treatment at 180°C)

Fluid Loss Agent 1	Efflux Time (s)				$\eta_r$	$\ln\eta_r$	$\ln\eta_r/c'$	$\eta_{sp}$	$\eta_{sp}/c'$
	1	2	3	Average					
t0	85	86	86	85.66667	1	0		0	
t1(c=c0)	232	233	233	232.6667	2.715952	0.999143	0.999143	1.715952	1.715952
t2(c=2/3c0)	204	206	205	205	2.392995	0.872546	1.308819	1.392995	2.089493
t3(c=1/2c0)	196	197	197	196.6667	2.295719	0.831046	1.662092	1.295719	2.591438
t4(c=1/3c0)	182	183	182	182.3333	2.128404	0.755372	2.266117	1.128404	3.385212
t5(c=1/4c0)	158	157	158	157.6667	1.840466	0.610019	2.440076	0.840466	3.361865

With the relational diagram of  $c', \ln\eta_r/c'$  drawn with relative concentration  $c'$  of solution as X-coordinate and  $\ln\eta_r/c'$  as Y-coordinate and via extrapolation, index of specialty  $[\eta]$  before and after thermal treatment of fluid loss agent is obtained. The fitting equation of fluid loss agent 1 before thermal treatment is  $y = -4.3729x + 6.2762$ ,  $[\eta]_{\text{before}} = 6.2762$ ; the fitting equation of fluid loss agent 1 after thermal treatment is  $y = -1.9794x + 2.824$ ,  $[\eta]_{\text{after}} = 2.824$ , as shown in Fig. 2. The fitting equation of fluid loss agent 2 before thermal treatment is  $y = -3.5096x + 5.3814$ ,  $[\eta]_{\text{before}} = 5.3814$ ; the fitting equation of fluid loss agent 2 after thermal treatment is  $y = -1.679x + 2.9775$ ,  $[\eta]_{\text{after}} = 2.9755$ , as shown in Fig. 3. The fitting equation of fluid loss agent 3 before thermal treatment is  $y = -1.3887x +$

2.1242,  $[\eta]_{\text{before}} = 2.1242$ ; the fitting equation of fluid loss agent 3 after thermal treatment is  $y = -1.1925x + 1.776$ ,  $[\eta]_{\text{after}} = 1.776$ , as shown in Fig. 4.

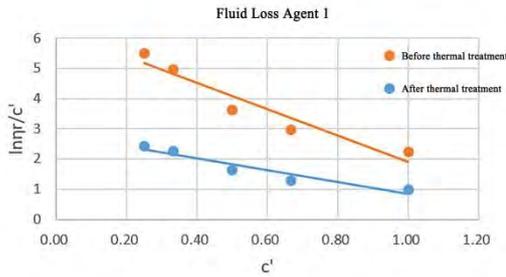


Fig. 2 Relation curve of  $c', \ln \eta_r / c'$

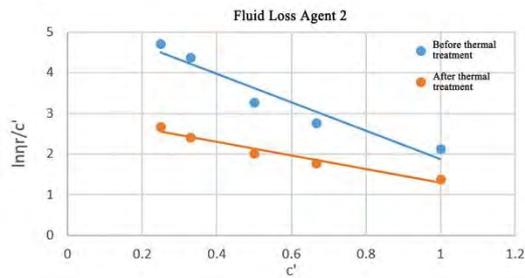


Fig. 3 Relation curve of  $c', \ln \eta_r / c'$

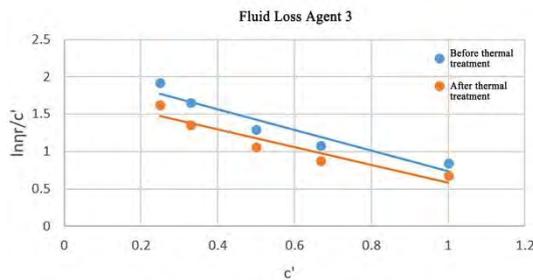


Fig. 4 Relation curve of  $c', \ln \eta_r / c'$

Experimental results are put into equation (11), to respectively calculate the change  $\Delta M$  in value  $[\eta]$  of polymer before and after the thermal treatment (at 180°C). The closer the absolute value of  $\Delta M$  to 0 is, the smaller the change in molecular weight (structure) of solvent before and after thermal treatment is, and the better the heat resistance is. Refer to Table 3 for calculated results.

Table 3 Rate of change in molecular weight of fluid loss agent after thermal treatment (at 180°C)

	Before thermal treatment $[\eta]$	After thermal treatment $[\eta]$	$ \Delta M $
Fluid Loss Agent 1	5.381	2.978	62.70%
Fluid Loss Agent 2	6.276	2.824	73.58%
Fluid Loss Agent 3	2.124	1.776	25.79%

It can be seen from the experimental results that the absolute value  $|\Delta M|$  of rate of change in molecular weight of fluid loss agent 1 before and after thermal treatment (at 180°C) is 62.70%, the absolute value  $|\Delta M|$  of rate of change in molecular weight of fluid loss agent 2 before and after thermal treatment (at 180°C) is 73.58% and the absolute value  $|\Delta M|$  of rate of change in molecular weight of fluid loss agent 3 before and after thermal treatment (at 180°C) is 25.79%. The experimental results indicate that the change in molecular weight of fluid loss agent 2 before and after the thermal treatment (at 180°C) is slight, and accordingly, a relatively good heat resistance can be realized.

#### 4.2 Analysis of Heat Resistance of Retarder at High Temperature

Efflux times of water solution corresponding to five concentrations (relative concentrations: 1, 2/3, 1/2, 1/3 and 1/4) of retarders (1, 2 and 3) before and after the thermal treatment are measured, to calculate  $\ln \eta_r / c'$  corresponding to different concentrations, and then the relative concentration  $c'$  of solution is taken as X-coordinate and  $\ln \eta_r / c'$  is taken as Y-coordinate to draw the relational diagram of  $c', \ln \eta_r / c'$ . The test results of efflux times of retarder 1 (before and after thermal treatment) are shown in Tables 4 and 5:

Table 4 Test results of efflux times of retarder 1 solution (before thermal treatment at 180°C)

Retarder 1	Efflux Time (s)				$\eta_r$	$\ln\eta_r$	$\ln\eta_r/c'$	$\eta_{sp}$	$\eta_{sp}/c'$
	1	2	3	Average					
t0	85	86	86	85.66667	1	0		0	
t1(c=c0)	95	94	94	94.33333	1.101167	0.09637	0.09637	0.101167	0.101167
t2(c=2/3c0)	94	93	93	93.33333	1.089494	0.085713	0.12857	0.089494	0.134241
t3(c=1/2c0)	92	93	92	92.33333	1.077821	0.074941	0.149882	0.077821	0.155641
t4(c=1/3c0)	92	92	92	92	1.07393	0.071324	0.213973	0.07393	0.221789
t5(c=1/4c0)	92	91	91	91.33333	1.066147	0.064052	0.256207	0.066147	0.26459

Table 5 Test results of efflux times of retarder 1 solution (before thermal treatment at 180°C)

Retarder 1	Efflux Time (s)				$\eta_r$	$\ln\eta_r$	$\ln\eta_r/c'$	$\eta_{sp}$	$\eta_{sp}/c'$
	1	2	3	Average					
t0	85	86	86	85.66667	1	0		0	
t1(c=c0)	91	91	91	91	1.062256	0.060395	0.060395	0.062256	0.062256
t2(c=2/3c0)	91	90	91	90.66667	1.058365	0.056726	0.085088	0.058365	0.087548
t3(c=1/2c0)	90	90	90	90	1.050583	0.049345	0.098691	0.050583	0.101166
t4(c=1/3c0)	97	97	96	96.66667	1.128404	0.120804	0.362413	0.128404	0.385213
t5(c=1/4c0)	90	90	90	90	1.050583	0.049345	0.197382	0.050583	0.202333

With the relational diagram of  $c', \ln\eta_r/c'$  drawn with relative concentration  $c'$  of solution as X-coordinate and  $\ln\eta_r/c'$  as Y-coordinate and via extrapolation, index  $[\eta]$  of specialty before and after thermal treatment of retarder is obtained. The fitting equation of retarder 1 before thermal treatment is  $y=-0.2027x + 0.2802$ ,  $[\eta]_{\text{before}} = 0.2802$ ; the fitting equation of retarder 1 after thermal treatment is  $y=-0.1717x + 0.2141$ ,  $[\eta]_{\text{after}} = 0.2141$ , as shown in Fig. 5. The fitting equation of retarder 2 before thermal treatment is  $y=-0.8031x + 1.3479$ ,  $[\eta]_{\text{before}} = 1.3479$ ; the fitting equation of retarder 2 after thermal treatment is  $y=-0.7671x + 1.1861$ ,  $[\eta]_{\text{after}} = 1.1861$ , as shown in Fig. 6. The fitting equation of retarder 3 before thermal treatment is  $y=-0.435x + 0.7648$ ,  $[\eta]_{\text{before}} = 0.7648$ ; the fitting equation of retarder 3 after thermal treatment is  $y=-0.3751x + 0.5962$ ,  $[\eta]_{\text{after}} = 0.5962$ , as shown in Fig. 7.

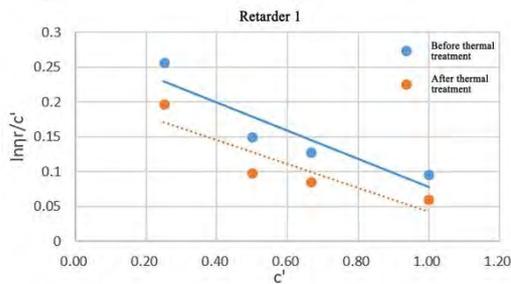


Fig. 5 Relation curve of  $c', \ln\eta_r/c'$

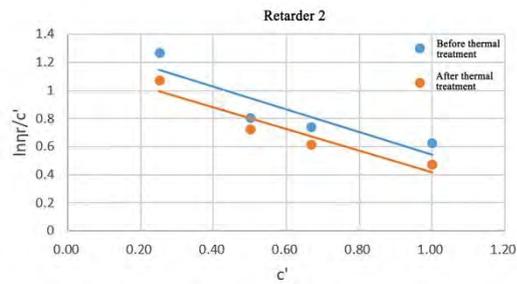


Fig. 6 Relation curve of  $c', \ln\eta_r/c'$

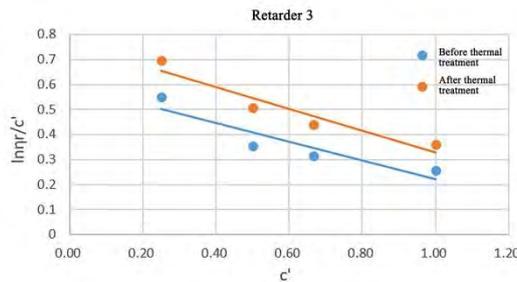


Fig. 7 Relation curve of  $c', \ln\eta_r/c'$

Experimental results are put into equation (11), to respectively calculate the change  $\Delta M$  in value  $[\eta]$  of polymer before and after the thermal treatment (at 180°C). The closer the absolute value of  $\Delta M$

to 0 is, the smaller the change in molecular weight (structure) of solvent before and after thermal treatment is, and the better the heat resistance is. Refer to Table 6 for calculated results.

Table 6 Rate of change in molecular weight of retarder after thermal treatment (at 180°C)

	Before thermal treatment $[\eta]$	After thermal treatment $[\eta]$	$ \Delta M $
Retarder 1	0.2802	0.2141	36.137%
Retarder 2	0.7648	0.5962	33.970%
Retarder 3	1.3479	1.1861	19.195%

It can be seen from the experimental results that the absolute value  $|\Delta M|$  of rate of change in molecular weight of retarder 1 before and after thermal treatment (at 180°C) is 36.137%, the absolute value  $|\Delta M|$  of rate of change in molecular weight of retarder 2 before and after thermal treatment (at 180°C) is 33.970% and the absolute value  $|\Delta M|$  of rate of change in molecular weight of retarder 3 before and after thermal treatment (at 180°C) is 19.195%. The experimental results indicate that the change in molecular weight of retarder 1 before and after the thermal treatment (at 180°C) is slight, and accordingly, a relatively good heat resistance can be realized.

Through assessment of heat resistance of aforesaid polymer admixtures, fluid loss agent 2 and retarder 1 are preferably selected as the heat-resistant admixture for cement paste. Assessment of settlement stability at a high temperature is carried out. With cement pastes respectively in the densities of 1.88, 2.0 and 2.2g/cm<sup>3</sup> and cured at 180 °C, differences of densities are respectively lower than 0.03, 0.04 and 0.04g/cm<sup>3</sup>, which indicates that the settlement stability at a high temperature is well kept. Fluid losses at a high temperature (180°C) are respectively 38mL, 39mL and 47mL. High-temperature thickening experiment is carried out. After thermal treatment at 180°C, the thickening times of the 3 retarders are all shortened, but the biggest drop in thickening time appears at retarder 1, which indicates a better stability after thermal treatment, and this matches the experiment results of assessment for heat resistance.

## 5. Conclusion

(1) The heat resistance of polymer is related to its molecular structure. Degradation, chain scission and group desorption of polymer admixture at a high temperature can all result in drop in molecular weight and viscosity. Under this principle, assessment for heat resistance of polymer admixture is carried out with high polymer experimental technique. This method is simple, feasible and easy to conduct, featuring a large range of application of molecular weight, and can be used as the optimal assessment method for cement polymer admixture for oil well.

(2) The stability of cement paste for well oil at a high temperature and regulation performance in high-temperature thickening experiment can only be improved by polymer admixture with a good heat resistance. This method is conducive to guiding the design of cement paste system for well cementation at a high temperature.

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