

The Effect of Synthesis Conditions on the Performance of Large Temperature Difference Retarder

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Abstract: In view of the ability of present oil well cement retarder is poor to adapt to the large temperature difference, using 2-acrylamido-2-methyl propane sulfonic acid (AMPS) and Itaconic Acid (IA), a new retarder AMPS/IA (AI) which could solve the problem of the large temperature difference was synthesized by free radical aqueous solution copolymerization. The effect of reaction conditions on the properties of retarder was studied. The results indicate that the monomer ratio and the pH value are more important than the monomer concentration, temperature and initiator dosage as impact factor for the performance of AI. The optimal AI (OAI) was obtained under the optimum reaction conditions: mole ratio of AMPS/IA = 73/27, monomer concentration = 40%, amount of (by weight of monomer) initiator = 2%, pH value = 2 and temperature = 60°C. Compared with the ordinary retarder, the OAI has merits as follows: excellent thermal resistant ability, less dosage, suitable for large temperature difference. The thickening time of the slurry with 1.5% OAI is 313 min at 180°C; the setting time is 72 h at 50°C, which indicates the OAI could meet the temperature range from 50°C to 180°C. In short, the OAI has excellent ability to adapt to the large temperature difference.

Keywords: Cement, cementing, oil well, retarder, synthesis conditions

INTRODUCTION

Oil well cementing operation is one of very important link in the process of oil well construction (Garnier *et al.*, 2007). As the society demand to oil and gas resources constantly increases, shallow oil and gas resources reduces day by day and the drilling technology improves gradually, the deep or super deep well increases sharply. The increase of the well depth will increase the sealing interval length with cement slurry, which will bring huge challenges for cementing operation (Zhang and Zou, 2005). Well cementing is non-recurring engineering and the 2010 Gulf of Mexico oil well blowout fully illustrates the important position of the safety and quality of cementing operation in the whole drilling process, which also caused the oil industry paying attention to cementing security (Qi *et al.*, 2011). The thickening time of cement slurry is the key parameter to decide the safety of cementing construction, if thickening time is too short, the cement slurry may set in the casing, even resulting in the scrapping of the well; if the thickening time is too long, it will greatly extend the setting time of the cement slurry, increase the drilling cost and be adverse to anti-gas channeling (Wang *et al.*, 1999; Huang *et al.*, 2011). The thickening time of cement slurry could be adjusted by the retarder that is very important as one kind of cement additive. The conventional retarder including

tartaric acid, sodium gluconate, phosphonate (Nelson and Guillot, 2006) has some disadvantages, the most important of which is the poor ability to adapt the large temperature difference of the oil well. For the long sealing interval of the well, the temperature difference between the top and the bottom of the cement slurry column is very big. In order to meet the need of cementing construction safety, the test temperature of the thickening time is based on the bottom hole circulating temperature in cementing engineering design (American Petroleum Institute, 2002), which easily causes the long setting time of the cement slurry in the temperature of top of the cement slurry column. The long setting time of the cement slurry will greatly increase the drilling cost and also be adverse to the safety of the cementing construction. Therefore, the retarder satisfying the large temperature difference needs to be developed.

For large temperature difference retarder, the retarder which adapted to the temperature range 165°C~200°C was developed by Al-Yami (2007) using glycol and calcium lignosulphonate, the retarders were synthesized by Brothers (1991), Eoff (1995), Bu *et al.* (2009), Wang *et al.* (2009), Su and Li (2005) and Jin and Zhuang (2009) respectively using AMPS, acrylic, or itaconic acid and the most excellent of them was synthesized using AMPS and itaconic acid as monomers. Although the performance of the synthetic

retarder is excellent, the effect of the synthetic conditions on the performance of the retarder was studied poorly. In order to provide some reference for the improvement of the performance of this kind of retarder, the effect of the synthetic conditions on the performance of the retarder synthesized using AMPS and IA was studied in this study.

MATERIALS AND METHODS

Synthesis of AI: Copolymers of AMPS and IA were prepared by aqueous solution polymerization technique in the laboratory. AMPS were obtained from RBL CHEMICALS CO. (Beijing, China). IA, sodium hydroxide and potassium per sulfate were obtained from SINOPHARM CHEMICAL REAGENT CO. (Shanghai, China). IA and AMPS were all used without further purification. To avoid the premature polymerization induced by excessive heat released by the dissolution of sodium hydroxide, a certain sodium hydroxide was first added to the deionized water before the monomers. The monomers were added into the solution ordinarily after the solution with sodium hydroxide was cooled. The pH value was adjusted to the desired value by adding sodium hydroxide solution. The solution was added into the three-necked flask, after it had been prepared. Nitrogen gas was bubbled through the reaction mixture for 30 min with slow stirring. After heated to the desired temperature, potassium per sulfate was added drop-wise to the reaction mixture and the reaction was allowed to proceed for about 4 h. The reaction product mixture was converted into the powdery size through drying and crush break.

Cement slurry preparation: Cement slurries were prepared in accordance with the test procedure set forth in API Recommended Practice for Testing Well Cements 10B (American Petroleum Institute, 2002). Fluid loss control additive BXF-200L and anti-foaming agent were obtained from BO-XING ENGINEERING SCIENCE AND TECHNOLOGY CO. OF CNPC (Tianjin, China). Amino Trimethylene Phosphonic Acid (ATMPA), tartaric acid (TA), Sodium Gluconate (SG) was obtained from SINOPHARM CHEMICAL REAGENT CO. (Shanghai, China). API class "G" cement was obtained from SICHUAN JIAHUA ENTERPRISE CO. (Leshan, China). Cement slurry compositions were prepared comprising a class "G" cement, water (44% By Weight of Cement, BWOC), different quantities of retarder (BWOC), anti-foaming agent (0.5% BWOC), BXF-200L (4% by weight of cement, BWOC). The cement slurry was mixed with the Waring blender.

Test procedure: Thickening time of the cement slurry was determined following API RP 10B using the HPHT (high pressure-high temperature) consist meter (American Petroleum Institute, 2002). The main

apparatus is a rotating cylindrical slurry container equipped with a stationary paddle assembly, all enclosed in a pressure vessel capable of withstanding the well-simulation pressures and temperatures. The slurry container is rotated at a speed of 150 ± 15 rpm (2.5 ± 0.25 rev/s). The consistency of the slurry which is expressed in Bearden units of consistency (Bc) should be measured. After the slurry container installed in the HPHT consist meter, the temperature and pressure should be increased in accordance with the appropriate well-simulation test schedule. The thickening time is the elapsed time from the initial application of pressure and temperature to the time at which the slurry reaches a consistency deemed sufficient to make it un-pumpable (e.g., 70 or 100 Bc).

The measurement of setting time was performed using vicat apparatus according to European Norm (EN 196-3, 2005). The method is based on measuring a depth of penetration of a steel right cylinder needle into the cement paste. The time between starting time and the time at which the distance between the needle and the top of the slurry is $d = 6 \pm 3$ mm, measured to the nearest minute, is defined as the Final Setting Time (FST) of the cement. Within this study, penetration tests were performed at regular time intervals until the cement paste was completely set.

RESULTS AND DISCUSSION

Effect of monomer ratio: The copolymers were conducted with a 20% total monomer concentration in deionized water, 2% (by weight of monomer) potassium persulfate, different mole ratio of AMPS/IA and a pH value 2 at 60°C for 4h. Table 1 shows the effect of monomer mole fractions of IA on the performance of the cement slurry containing 0.5% AI. The thickening time of the slurry increases from 80 min to 295 min at 120°C, from 209 min to more than 600 min at 50°C and the setting time of the slurry increases from 8 h to 30 h as the mole fraction of IA increases from 0% to 25%. The thickening time of the slurry decreases from 295 min to 13 min at 120°C, from more than 600 min to 30 min at 50°C and the setting time of the slurry decreases from 30 h to 3 h as the mole fraction of IA increases from 25% to 100%. The variation direction of the thickening time and setting time is consistent and the optimum mole ratio of AMPS and IA is 75:25.

Table 1: Effect of mole fractions of IA on AI

| IA/% | Thickening time/min | | Setting time/h |
|------|---------------------|------|----------------|
| | 120°C | 50°C | |
| 0 | 86 | 209 | 8 |
| 20 | 163 | >600 | 18 |
| 25 | 295 | >600 | 30 |
| 30 | 250 | >600 | 30 |
| 40 | 143 | 362 | 10 |
| 60 | 49 | 90 | 7.5 |
| 80 | 14 | 35 | 3 |
| 100 | 13 | 30 | 3 |

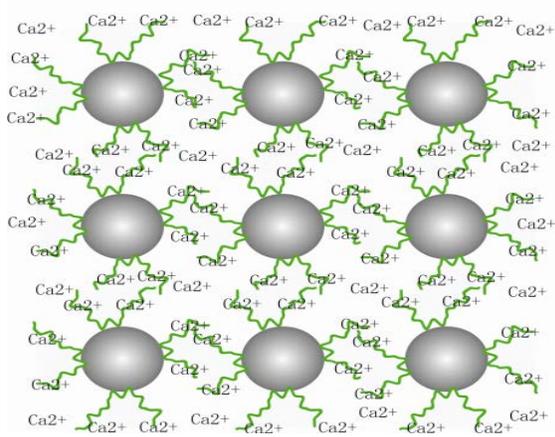


Fig. 1: The shape of molecular chain of AI with good alkali-resisting ability in the cement slurry

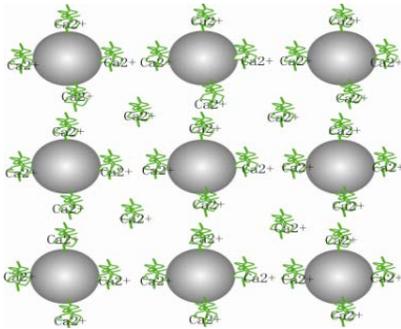


Fig. 2: The shape of molecular chain of AI with poor alkali-resisting ability in the cement slurry

The monomer AMPS could provide sulfonic acid group for the ability of temperature resistance, salt resistance and alkali resistance of the AI and the monomer IA could provide double carboxyl groups for the chelating ability and the adsorption ability of the AI. On the one hand, the dissolution rate of the cement mineral could be delayed by the adsorption of the AI in the active point of the cement particle surface, therefore the increasing speed of calcium ion in the solution is delayed from the source; On the other hand, the formation of calcium hydroxide crystal is delayed by the concentration decrease of calcium ion induced by effective chelation of double carboxyl. The ability of salt and alkali resistance of AI is strong due to the high content of sulfonic acid group when the mole fraction of IA is below 25%. Therefore, the stretch of the AI is excellent as shown in Fig. 1, which is benefit for the adsorption of AI on the surface of cement particles. The chelating and adsorption capacity increases gradually as the mole fraction of IA increases from 0% to 25%, therefore the thickening time of the slurry increases gradually. The ability of salt and alkali resistance of AI decreases gradually as the mole ratio of IA increases from 25% to 100%. Therefore, the stretch of the AI is poor; especially when the mole fraction of IA is more than 60%, the precipitation of the AI (Fig. 2) will

Table 2: Effect of pH value on AI

| pH | Thickening time/min | | Setting time/h |
|----|---------------------|------|----------------|
| | 120°C | 50°C | |
| 1 | 294 | >600 | 30 |
| 2 | 295 | >600 | 30 |
| 3 | 294 | >600 | 30 |
| 5 | 134 | 367 | 9 |
| 8 | 133 | 353 | 9 |
| 11 | 125 | 351 | 9 |
| 14 | 116 | 309 | 8.5 |

Table 3: Effect of initiator dosage on AI

| Initiator/% | Thickening time/min | | Setting time/h |
|-------------|---------------------|------|----------------|
| | 120°C | 50°C | |
| 0.5 | 261 | >600 | 30 |
| 1.5 | 273 | >600 | 30 |
| 2 | 295 | >600 | 30 |
| 2.5 | 279 | >600 | 30 |
| 5 | 274 | >600 | 30 |
| 10 | 179 | >600 | 19 |

happen in the solution with pH above 12, which is disadvantage of the adsorption of AI on the surface of cement particles. After the AI is introduced in the slurry, the AI will chelate calcium ion and then precipitate in the solution, which reduces the content of calcium ion in the solution and accelerates the dissolution of the cement mineral. Therefore, the hydration of the cement with the AI which the mole fraction of IA is above 60% was accelerated.

Effect of pH: The copolymers were conducted with a 20% total monomer concentration in deionized water, 2% (by weight of monomer) potassium per sulfate, different pH value and mole ratio of AMPS/IA = 75/25 at 60°C for 4 h. Table 2 shows the effect of pH of the synthesized solution on the performance of the cement slurry containing 0.5% AI.

The thickening time of the slurry decreases from 294 min to 116 min at 120°C, from more than 600 min to 309 min at 50°C and the setting time of the slurry increases from 30 h to 8.5 h as the pH value of the synthesized solution increases from 1 to 14. The variation direction of the thickening time and setting time is consistent. The performance of AI synthesized on the pH = 1, 2 and 3 all are excellent. But the optimum pH value was fixed at 2, for the pH of the solution with the AMPS and IA is naturally 2 after the AMPS and IA were introduced in the solution. AMPS and IA gradually change from the acid to the salt as the pH value increases, which will lead to the change of reactivity ratio of the monomer (Pan, 2007). The performance of AI changes due to the reason mentioned above in the end.

Effect of initiator dosage: The copolymers were conducted with a 20% total monomer concentration in deionized water, different amount of potassium per sulfate (by weight of monomer), mole ratio of AMPS/IA = 75/25 and a pH value 2 at 60°C for 4h.

Table 4: Effect of monomer concentration on AI

| Monomer concentration/% | Thickening time/min | | Setting time/h |
|-------------------------|---------------------|------|----------------|
| | 120°C | 50°C | |
| 10 | 193 | >600 | 20 |
| 15 | 286 | >600 | 30 |
| 20 | 295 | >600 | 30 |
| 30 | 300 | >600 | 30 |

Table 5: Effect of temperature on AI

| Temperature/°C | Thickening time/min | | Setting time/h |
|----------------|---------------------|------|----------------|
| | 120°C | 50°C | |
| 40 | 278 | >600 | 30 |
| 50 | 286 | >600 | 30 |
| 60 | 295 | >600 | 30 |
| 70 | 274 | >600 | 30 |
| 80 | 231 | >600 | 29 |
| 90 | 156 | >600 | 26 |

Table 3 shows the effect of initiator dosage on the performance of the cement slurry containing 0.5% AI.

The thickening time and setting time change little as the dosage of the initiator increases from 0.5% to 5%, but the thickening time and setting time reduce obviously as the dosage of the initiator increases to 10%. The variation direction of the thickening time and setting time is consistent and the optimum dosage of the initiator is 2%.

The molecular weight of the polymer is inversely proportional to the square root of the concentration of the initiator (Pan, 2007), the molecular weight of the polymer decreases as the dosage of the initiator increases. The thickening time of the slurry changes little as the dosage of the initiator increases from 0.5% to 5%, which indicates the performance of the AI changing little over the range of the molecular weight. The thickening time reduces obviously as the dosage of the initiator increases to 10%, which indicates the decrease of the molecular weight impairs the performance of the AI.

Effect of monomer concentration: The copolymers were conducted with 2% (by weight of monomer) potassium per sulfate, different monomer concentration, mole ratio of AMPS/IA = 75/25 and a pH value 2 at 60°C for 4 h. Table 4 shows the effect of monomer concentration on the performance of the cement slurry containing 0.5% AI. The thickening time of slurry at 120°C increases from 193 min to 300 min, the thickening time at 50°C is all more than 600 min and the setting time at 50°C increases from 20 h to 30 h, as the monomer concentration increases from 10% to 30%. The variation direction of the thickening time and setting time is consistent.

The molecular weight of the polymer is proportional to the monomer concentration, the molecular weight increases as the increase of the monomer concentration. The molecular weight of the polymer increases as the monomer concentration increases from 10% to 15%. The increase of the molecular weight is beneficial to the adsorption of the

AI on the surface of cement particles, which weakens desorption of the AI from the surface of cement particles induced by the rise of temperature and enhances the performance of the AI. The thickening time changes little as the monomer concentration increases from 15% to 30%, which may be due to the adsorption force of AI synthesized at the monomer concentration of 15% is strong enough to balance the desorption induced by the rise of the temperature.

Effect of synthesized temperature: The copolymers were conducted with a 20% total monomer concentration in deionized water, 2% (by weight of monomer) potassium per sulfate, mole ratio of AMPS/IA = 75/25 and a pH value 2 at different synthesized temperature for 4 h.

The thickening time of the slurry at 120°C first increases and then decreases as the increase of the temperature. But the thickening time and setting time change little as the temperature increases from 40°C to 60°C and the thickening time at 120°C and the setting time at 50°C reduces obviously as the temperature increases from 70°C to 90°C. The variation direction of the thickening time and setting time is consistent. The optimum temperature is 60°C.

The synthesis temperature mainly influences the decomposition rate of the initiator and the chain termination way of the polymer. The monomer percent conversion increases and the molecular weight of polymer decreases as the temperature increases. Though the molecular weight of the AI and the adsorption force of the AI on the surface of the cement particles reduce as the temperature increases from 40°C to 60°C, the adsorption force of the AI within the temperature between 40°C and 60°C is strong enough to balance the desorption induced by the rise of the temperature, which leads to the thickening time of the slurry changing a little as the synthesis temperature increases from 40°C to 60°C. Nevertheless, the monomer percent conversion increases as the synthesis temperature increases, which leads to the performance of the AI improving a little but not so much. The molecular weight of the AI and the adsorption force of the AI on the surface of the cement particles reduce as the temperature increases from 60°C to 90°C, the adsorption force of the AI is not strong enough to balance the desorption induced by the rise of the temperature gradually, which leads to the performance of the AI impaired (Table 5).

Orthogonal experiment: The effect of the synthesis conditions on AI shows that the monomer ratio, pH value, initiator concentration, monomer concentration and temperature all have some influence on the performance of the AI. For the performance of AI synthesized with the pH value between 1 and 3 changes little and 2 is the natural pH value of the synthesis

Table 6: Synthesized parameters and levels for orthogonal experiment

| Level | T (°C) [A] | Monomer concentration (%) [B] | Initiator concentration (%) [C] | Mole ratio of AMPS/IA [D] |
|-------|------------|-------------------------------|---------------------------------|---------------------------|
| 1 | 50 | 20 | 1.5 | 73:27 |
| 2 | 60 | 30 | 2.0 | 75:25 |
| 3 | 70 | 40 | 2.5 | 77:23 |

Table 7: Analysis of test results

| Test no. | A | B | C | D | Thickening time/min |
|---------------|------------------|-----|-----|-----|---------------------|
| 1 | A1 | B1 | C1 | D1 | 290 |
| 2 | A1 | B2 | C2 | D2 | 292 |
| 3 | A1 | B3 | C3 | D3 | 271 |
| 4 | A2 | B1 | C2 | D3 | 283 |
| 5 | A2 | B2 | C3 | D1 | 310 |
| 6 | A2 | B3 | C1 | D2 | 290 |
| 7 | A3 | B1 | C3 | D2 | 270 |
| 8 | A3 | B2 | C1 | D3 | 260 |
| 9 | A3 | B3 | C2 | D1 | 309 |
| K1 | 284 | 281 | 280 | 303 | |
| K2 | 294 ^a | 287 | 295 | 284 | |
| K3 | 280 | 290 | 283 | 271 | |
| R | 14 ^b | 9 | 15 | 32 | |
| Optimal level | A2 | B3 | C2 | D1 | |

a: $K_i^A = (\sum \text{the thickening time of synthesized AI at } A_i)/3$; b: $R_i^A = \text{Max}\{K_i^A\} - \text{min}\{K_i^A\}$

Table 8: The optimal experiment condition and the performance of OAI

| T(°C) | Monomer concentration (%) | Initiator concentration (%) | Mole ratio Of AMPS/IA | Thickening time at 120°C/min | Thickening time at 50°C/min | Setting time at 50°C/h |
|-------|---------------------------|-----------------------------|-----------------------|------------------------------|-----------------------------|------------------------|
| 60 | 40 | 2 | 73:27 | 325 | >600 | 30 |

Table 9: Comparison between AI and common retarder

| Retarder | Dosage/% | Thickening time | | | Setting time | | |
|----------|----------|-----------------|---------------------|------|---------------------|------|----------------|
| | | T/°C | Thickening time/min | T/°C | Thickening time/min | T/°C | Setting time/h |
| TA | 0.7 | 110 | 245 | 50 | >600 | 50 | 72 |
| TA | 0.8 | 120 | 102 | 50 | >600 | 50 | >240 |
| SG | 0.4 | 110 | 210 | 50 | >600 | 50 | >240 |
| ATMPA | 0.15 | 110 | 298 | 50 | >600 | 50 | >240 |
| AMPS-IA | 0.2 | 110 | 300 | 50 | 540 | 50 | 8 |
| AMPS-IA | 0.5 | 120 | 325 | 50 | >600 | 50 | 30 |
| AMPS-IA | 1.1 | 150 | 347 | 50 | >600 | 50 | 48 |
| AMPS-IA | 1.5 | 180 | 313 | 50 | >600 | 50 | 72 |

solution, therefore pH = 2 is fixed in the design of the orthogonal experiment.

An orthogonal $L_9(4^3)$ test design was used to optimize the other synthesis conditions further (Fang and Ma, 2001). Table 6 lists the schedule of the orthogonal experiment in which four key parameters including:

- Temperature
- Monomer concentration
- Initiator Concentration
- Mole ratio of AMPS/IA

The variation direction of the thickening time and setting time is consistent by the study of the single factor experiment and the thickening time at 120°C was chosen for the optimization goal of the orthogonal test. Table 7 shows the performance of the cement slurry containing 0.5% AI. Table 7 shows that the influence on the thickening time of the slurry containing AI decreases in the order: D>C>A>B according to the R values. According to the orthogonal experiment design principle, the bigger the K_i^X (X could be A, B, C, or D

and i could be 1, 2, or 3), the better the test result under the X_i condition. For example, according to Table 7: K_2^A is the biggest among K_1^A , K_2^A and K_3^A , so A2 was chosen to be the optimal level for factor A. Table 8 lists the optimal experiment condition optimized from the orthogonal test and the performance of AI which was obtained under the optimal condition. Under the optimal conditions, the thickening time at 120°C, the thickening time at 50°C and the setting time at 50°C are 325 min, more than 600 min and 30 h, respectively, which were better than the test result under other experiment conditions. An OAI sample was prepared under the optimal experiment condition for other experiment test and evaluation.

The comparison between AI and common retarder:

The performance of the slurry containing different retarders was tested to compare the performance of the retarders. Table 9 shows the performance of the slurry containing different retarders. The thickening time and the setting time all extends as the dosage of AI increases. The thickening time of the slurry at 110°C containing 0.7%TA, 0.4%SG, 0.15% ATMPA and

0.2% AMPS-IA was 245 min, 210, 298 and 300 min, respectively and the setting time at 50°C was 72 h, more than 240 h, more than 240 and 8 h, respectively. The setting time exceeds 240 h will prolong the construction time tremendously, which indicates the SG and ATMPA being not appropriate for the large temperature difference cementing at all. The setting time of the slurry containing 0.7% TA at 50°C is 72 h, which indicates the TA being appropriate for the temperature range between 50°C and 110°C basically, but the setting time containing 0.7% TA is 9 times longer than 0.2% AI. The thickening time of the slurry at 120°C containing 0.8% TA and 0.5% AI was 102 and 325 min, respectively and the setting time at 50°C is more than 240 and 30 h, respectively, which indicates the TA being not appropriate for the temperature range between 50°C and 120°C and the AI could satisfy the need of the temperature range between 50°C and 120°C easily. The thickening time of the slurry containing 1.5% AI at 180°C is 313 min and the setting time at 50°C is 72 h, which indicates the AI with the ability to satisfy the temperature range between 50°C and 180°C.

CONCLUSION

The monomer ratio and pH value have very important effect on the performance of the AI and the monomer concentration, synthesis temperature and initiator dosage all have some effect on AI but not so much.

The OAI was obtained under the optimum reaction conditions: mole ratio of AMPS/IA = 73/27, monomer concentration = 40%, amount of (by weight of monomer) initiator = 2%, pH value = 2 and temperature = 60°C.

Compared with the ordinary retarder, the OAI has merits as follows: excellent thermal resistant ability, less dosage, suitable for large temperature difference. The thickening time of the slurry with 1.5% OAI is 313 min at 180°C, the setting time is 72 h at 50°C, which indicates the OAI could meet the temperature range from 50°C to 180°C.

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