Synthesis and Application of 2-Acrylamido-2-Methyl Propane Sulfonic Acid/Acrylamide/N,N-Dimethyl Acrylamide/Maleic Anhydride as a Fluid Loss Control Additive in Oil Well Cementing

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ABSTRACT: Using 2-acrylamido-2-methyl propane sulfonic acid (AMPS), acrylamide (AM), N,N-dimethyl acrylamide (NNDMA), and maleic anhydride (MA), a new dispersive type fluid loss control additive (FLCA) AMPS/AM/NNDMA/MA (PANM) was synthesized by free radical aqueous solution copolymerization, and the new FLCA could be used without dispersant existing in the cement. The optimal PANM (OPANM) was obtained under the optimum reaction conditions: mole ratio of AMPS/AM/NNDMA/MA = 4/2.5/2.5/1, monomer concentration = 32.5%, amount of (by weight of monomer) ammonium persulfate/sodium bisulfate = 1.0%, pH value = 4, and temperature = 40°C. The synthesized copolymer OPANM was identified by FTIR analysis. The evaluation results show the OPANM has excellent dispersing power, fluid loss control ability, thermal resistant, and salt tolerant ability. The OPANM was even stable when the temperature was below 300°C proved by TG analysis. The thickening time of the slurry containing the synthesized additive reduces as the temperature increases. The copolymer OPANM is expected to be an excellent FLCA. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

INTRODUCTION

Oil well cementing is one of the most important operations in the construction of a well bore.[1] It is clearly acknowledged that a lack of fluid loss control may be responsible for primary cementing failures, due to excessive density increase or annulus bridging and that formation invasion by cement filtrate may be deleterious to the production.[2] To control the fluid loss of the cement slurry, FLCA is added to oil well cement to reduce the uncontrolled water loss, which plays a key role in ensuring the safety of oil well cementing operation and improving the cementing quality.[3] Therefore, FLCA is always the hotspot in cementing additives research. Fluid loss control additives (FLCAs) mainly contain granular materials, water soluble polymers, and other organic materials. The most typical granular material is rubber latex. At present, most of FLCAs are water soluble polymers.[4] In the early stage, cellulose derivatives as water-soluble fluid loss polymers were introduced. Cellulose-based FLCAs are highly retardive and can produce excessive mixing viscosities.[5] Thereafter, they have been replaced by synthetic polymers such as polyethylene imine and polyvinyl alcohol, but they are limited in the use of field due to all kinds of reasons.[6] Relying on excellent fluid loss control ability, acrylamide-based FLCA was investigated a lot, but this kind of FLCA is over-retardation, caused when a portion of the acrylamide hydrolyzes to acrylic acid.[7,8] Nowadays the most promising water-soluble fluid loss polymers might be the AMPS-based polymers which show outstanding thermal resistant and salt tolerant ability. AMPS has become the hotspot in polymer synthesis relying on the excellent thermal-resistant and salt-tolerant properties.[9] Copolymers of AMPS and NNDMA were found to be effective FLCAs for well cementing.[10–12] Nevertheless, these AMPS-based polymers as well as other water-soluble fluid loss polymers have the feature of excessively increasing the consistence of the slurry, so numerous dispersant should be introduced to improve the rheological behavior of the slurry and to ensure the safety of cementing operation. However, the dispersant introduced in the cement slurry could impair the fluid loss control ability of the FLCA containing ionic groups due to competitive adsorption.[13] Besides, it is very troublesome to add many kinds of additives in the cement slurry during the preparation process. This problem could be solved by introducing the groups with dispersive ability and adjusting the molecular weight of the FLCA.
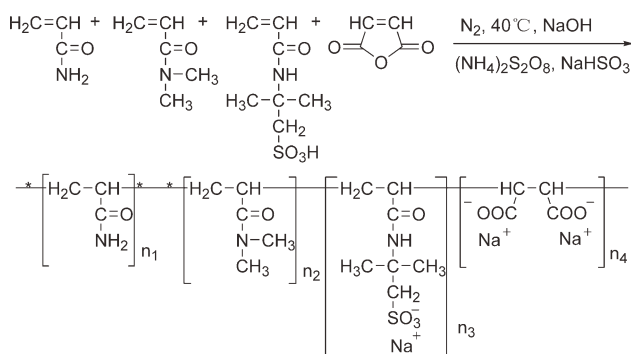
Figure 1. Schematic diagram of copolymerization.

To explain the working mechanisms, Plank studied different kinds of FLCA in detail.[14–17] For non-crosslinked polyvinyl alcohol, the main mechanism is plugging of the cement filter cake by film formation. The cationic polyethylene imine when used alone does not perform well as a fluid loss additive. Combination with dispersant, however, greatly improves fluid loss control ability. The complex of polyethylene imine and dispersant could form based on ionic interaction between the cationic imine present in polyethylene imine and the anionic sulfonate groups existing in dispersant. The complex could reduce filter cake permeability by a plugging mechanism. The AMPS-based FLCA achieves fluid loss control by reducing filter cake permeability through adsorption on cement.[14] The filter cake structure (pore size) is not affected by the hydroxyethyl cellulose (HEC). HEC swells to a multiple of its initial particle size, which blocks the pores of the cement filter cake and reduces the fluid loss.[18]

A new FLCA (OPANM) was synthesized using AMPS, AM, NNDMA, and MA by investigating the impact of AMPS amount, MA amount, and other synthesis conditions on the properties of PANM through orthogonal experiment. The OPANM copolymer synthesized in this article has excellent thermal-resistant and salt-tolerant properties and could be used without dispersant existing in the cement.

EXPERIMENTAL

Synthesis of PANM

Copolymers of AMPS, AM, NNDMA, and MA were prepared by aqueous solution polymerization technique in the laboratory. AMPS and NNDMA were obtained from RBL Chemicals (Beijing, China). AM, MA, sodium hydroxide, hydrochloric acid, ammonium persulfate, and sodium bisulphite were obtained from Sinopharm Chemical Reagent (Shanghai, China). Before the synthesis, NNDMA was exposed over calcium hydride for about 24 h and then distilled under vacuum to remove the stabilizers. 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 solu-

tion ordinally after the solution with sodium hydroxide was cooled. The pH value was adjusted to the desired value by adding sodium hydroxide solution or diluted hydrochloric acid. 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 heating to the desired temperature, the initiator (ammonium persulfate and sodium bisulphite with weight ratio 1 : 1 in aqueous solution) was added drop-wise to the reaction mixture and the reaction was allowed to proceed for about 4 h. The reaction equation is shown in Figure 1. The reaction product mixture was converted into the powdery size through drying and crush break.

Copolymer Characterization

The copolymer PANM was precipitated in a large amount of acetone and dried in vacuum at room temperature. FTIR spectra of PANM were recorded using a NEXUS Fourier Transform Interferometer. Perkin Elmer thermogravimetric analyzer of type PETA7 was used to check the performance under high temperature. The temperature range was from 40 to 500°C at heating rate of 10°C/min. Molecular weights (M_w, M_n) of PANM were determined by gel permeation chromatography (Waters 2695, US) using $0.2M$ $NaNO_3$ aqueous solution (adjusted with NaOH to pH = 9) as an eluent at a flow rate of 1.0 mL/min. The PANM solution was filtered through a 5-μm filter. Calibration was carried out with glucan (provided by Shodex) standards. An ubbelohde viscometer was used to measure intrinsic viscosity number [η] of PANM with 1 mol/L sodium chloride solution at 30°C.

Cement Slurry Preparation

Cement slurries were prepared in accordance with the test procedure set forth in API Recommended Practice for Testing Well Cements 10B, 23nd, April 2002.[19] Retarder BCR-300L, dispersant CF40S and anti-foaming agent were obtained from Bo-Xing Engineering Science and Technology of CNPC (Tianjin, China). Sodium chloride was obtained from Sinopharm Chemical Reagent (Shanghai, China). API class "G" cement was obtained from Sichuan Jiahua Enterprise (Leshan, China), and the phase composition and physical properties of class "G" cement are presented in Table I. Cement slurry compositions having densities of 1.89 g/cm^3 were prepared comprising a class "G" cement, water (44% by weight of cement, BWOC), different quantities of PANM (BWOC), anti-foaming agent(0.5% BWOC), different amounts of NaCl (by weight on water, BWOW), and different amounts of retarder (BWOC). The cement slurry was mixed with the Waring blender.

API Static Fluid Loss

Static fluid loss was determined following API RP 10B using a stirred fluid loss cell.[19] After the slurry was poured into the filter cell, the paddle was agitated to make the slurry keep flowing when the slurry was heated to objective temperature. Once the slurry has reached the specified temperature, stop stirring, invert the fluid loss cell, and apply 1000 psig differential pressure to the

Table I. Phase Composition and Physical Properties of Class G Oil Well Cement

C$_3$S (wt %)	C$_2$S (wt %)	C$_3$AC (wt %)	C$_3$AF (wt %)	Specific density (kg/L)	Specific surface area (m^2/kg)
53.7	30.46	2.8	8.0	3.17	332

Table II. Effect of MA Amount on the Properties of PANM

| No. | MA (mol %) | PANM (BWOC%) | Rheological behavior | | APIf (mL) |
			K (Pa s^n)	n	
1	0	1	2.3	0.42	294
2	5	1	0.31	0.87	29
3	10	1	0.37	0.86	26
4	20	1	0.39	0.81	28
5	30	1	0.21	0.89	38

fluid loss cell. Filtration proceeded through a 3.5-in.[2] mesh metal sieve placed at the bottom of the cell. The filtrate produced by the differential pressure was collected for 30 min. As described by API RP 10B, the collected filtrate volume was doubled and designated as API fluid loss (APIf) of the corresponding cement slurry. APIf was tested at 90°C in Tables II–IV, VI, and VII.

Rheology

Rheology of the cement slurry was determined at 52°C following API RP 10B using the FANN 35SA viscometer equipped with R1 rotor sleeve (rotor diameter: 36.83 mm), B1 bob (bob diameter: 34.49 mm, bob height: 38.00 mm), and F1 torsion spring was used.[19] The cement slurry obtained after stirring in the atmospheric consistometer was poured into the 300-mL cup of the FANN rheometer. Shear stress was measured at shear rates of 511, 340, 170, 10.2, 5.11 s^{-1}. Power Law model is usually used to describe the rheological behavior of the cement slurry. The Power Law model is shown as equation (1) in which n is referred to as flow behavior index and K is referred to as the consistency index. K and n in Tables II, III, VI, and VII were determined by the formula (2) and (3) according to Power Law model.[19] For shear thinning fluids (pseudo-plastic), n is a positive number between zero and one. Cement slurries normally exhibit pseudo-plastic behavior. For shear thickening fluids (dilatant), n is a positive number greater than one. Cement slurries normally do not exhibit dilating behavior. If n is equal to one, the fluid conforms to the Newtonian fluid model. The unit of K is Pa s^n, and the flow behavior index n is dimensionless. For the conventional cementing operation, low friction pressure drop is needed to avoid the formation fractured and lost circulation during cementing operation. The cement slurry should be a low viscosity and weak thixotropic fluid. Therefore, a low K value and a n value close to one are preferred.

$$\tau = K \times r^n \tag{1}$$

$$n = 2.092 \lg\left(\frac{\tau_{511}}{\tau_{170}}\right) \tag{2}$$

$$K = \frac{\tau_{511}}{511^n} \tag{3}$$

τ_{511} and τ_{170} were the shear stress measured for shear rates of 511, 170 s^{-1} using FANN 35SA viscometer.

Thickening Time

Thickening time of the cement slurry was determined following API RP 10B using the HPHT (high pressure–high temperature) consistometer.[19] 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 consistometer, the temperature and pressure should be increased in accordance with the appropriate well-simulation test schedule.[19] 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 unpumpable (e.g. 70 or 100 Bc).

RESULTS AND DISCUSSION

Effect of MA Amount

The copolymers were conducted with a 25% total monomer concentration in deionized water, 0.4% (by weight of monomer) ammonium persulfate/sodium bisulfite, and a pH value 7 at 60°C for 4 h. Figure 2 shows the effect of MA amount on the shear-stress of cement slurry with the monomer mole ratio of AMPS/AM/NNDMA fixed at 4/2.5/2.5. K and n in Figure 2 was obtained by linear fit. Owing to the slurry containing the PANM with 0% MA was too thick to prepare, therefore, 0.5% dispersant (BWOC) was introduced into the slurry. It is not reasonable to compare it with the other PANM. In the condition of taking no account of the hydrolysis of the AM, the anion

Table III. Effect of AMPS Amount on the Properties of PANM

| NO. | AMPS (mol %) | PANM (BWOC%) | Rheological behavior | | APIf (mL) |
			K (Pa s^n)	N	
1	20	1	1.67	0.63	30
2	30	1	1.01	0.78	26
3	40	1	0.37	0.86	26
4	50	1	0.13	0.96	29
5	60	1	0.09	0.97	34

Table IV. Effect of Intrinsic Viscosity Number on the APIf of PANM

Initiator concentration (wt %)	Intrinsic viscosity number (mL/g)	APIf (mL)
0.5% Ammonium persulfate	428.14	40
0.2% Ammonium persulfate:sodium bisulfite = 1:1	283.30	22
0.4% Ammonium persulfate:sodium bisulfite = 1:1	226.87	26
1.0% Ammonium persulfate:sodium bisulfite = 1:1	171.20	27
1.5% Ammonium persulfate:sodium bisulfite = 1:1	156.67	40

Figure 3. Effect of AMPS amount on the rheological behavior of slurry. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

constitutional units' mole fractions of the PANM were 47.22, 50, 55.56, 61.11%, respectively, when the MA mole fractions were 5, 10, 20, 30%, respectively. Generally, the dispersing power of the PANM and the rheological behavior of the slurry improve gradually as the amount of anion constitutional units increases. However, the AMPS mole fraction decreases as the MA mole fraction increases, which affects the salt tolerant property of the PANM. Therefore, the rheological behavior of slurry containing PANM with different MA amount does not show much consistency with the change of the MA amount. Table II shows the effect of MA amount on the properties of cement slurry with the monomer mole ratio of AMPS/AM/NNDMA fixed at 4/2.5/2.5. The number of MA affects the properties of the PANM, and both K and APIf are relatively excellent when the mole fraction of the MA is 10% MA. The difference of n and K between Table II and Figure 2 is mainly due to the Power law model which does not apply to the slurry at low shear rate.

Effect of AMPS Amount
The copolymers were conducted with a 25% total monomer concentration in deionized water, 0.4% (by weight of monomer)
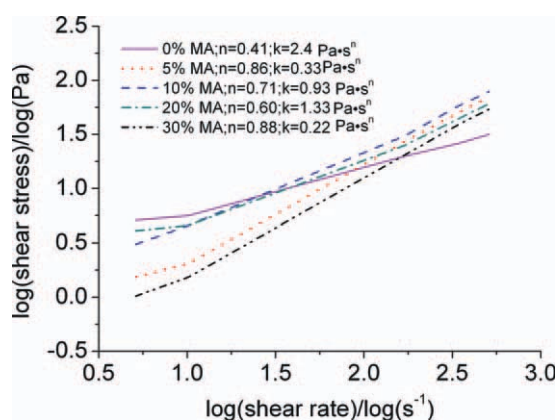

Figure 2. Effect of AM amount on the rheological behavior of slurry. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
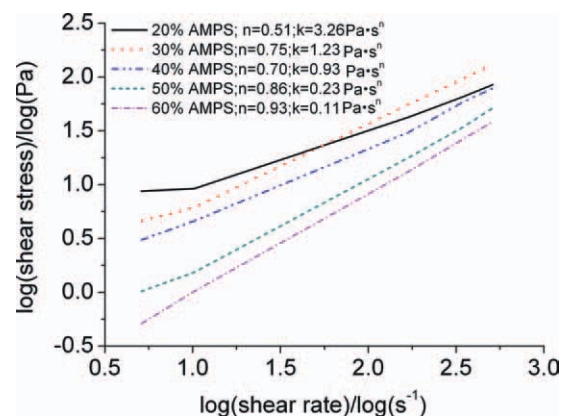
ammonium persulfate/sodium bisulfite, and a pH value 7 at 60°C for 4 h. Figure 3 shows the effect of AMPS amount on the shear-stress of cement slurry with mole ratio of AM/NNDMA fixed at 1/1 and 10% MA (by mole of total monomer). K and n in Figure 3 was obtained by linear fit. In the condition of taking no account of the hydrolysis of the AM, the anion constitutional units' mole fractions of the PANM were 30, 40, 50, 60, 70%, respectively, when the AMPS mole fractions were 20, 30, 40, 50, 60%, respectively. The shear stress of slurry decreases as the amount of anion constitutional units or the AMPS mole fraction increases except for 20% AMPS (by mole of total monomer) as shown in Figure 3, which demonstrates the dispersing power of the PANM increases as the AMPS mole fraction increases. The shear stress increase from AMPS mole fraction = 20% to AMPS mole fraction = 30% may be due to the molecular chain becomes more stretched as the amount of the anion constitutional units increases. Table III shows the effect of AMPS amount on the properties of cement slurry with mole ratio of AM/NNDMA fixed at 1/1 and 10% MA (by mole of total monomer). The K value decreases and the n value increases gradually as the AMPS mole fraction increases as shown in Table III, which indicates the rheological behavior of the slurry becomes better and is close to Newtonian fluid gradually. The difference of n and K between Table II and Figure 2 is mainly due to the Power law model which does not apply to the slurry at low shear rate. However, the APIf decreased to the minimum and then increased with the increasing of AMPS. Due to the AMPS has a strong anionic group, the dispersing power of PANM increases gradually with the increasing of AMPS. The filter cake permeability decreased when the diameter of cement particles decreased with the increasing of dispersing power of PANM, so the APIf decreased at the beginning. Due to the complicated charges characteristic of cement particle, the optimal adsorption property of PANM could be achieved only when the anionic group and nonionic group had a proper ratio. Too much anionic group of FLCA led to the increasing of APIf after it reached to the minimum. Because both the fluid loss control ability and the dispersing power of PANM with 40%AMPS (by

Table V. Synthesized Parameters and Levels for Orthogonal Experiment

Level	Parameters				
	Mole ratio of AMPS/AM/NNDMA/MA [A]	Monomer concentration (%) [B]	Initiator concentration (%) [C]	pH [D]	T (°C) [E]
1	4/2.5/2.5/1	10	0.4	4	40
2	4/3.25/1.75/1	17.5	0.6	7	50
3	4/4/1/1	25	0.8	10	60
4	4/4.75/0.25/1	32.5	1.0	13	70

mole of total monomer) behave well, the PANM with 40% AMPS and 10% MA (by mole of total monomer) was chosen to study further.

Effect of Molecular Weight

The copolymers were conducted with a 25% total monomer concentration in deionized water, different amount of initiator, AMPS:AM:NNDMA:MA=4 : 2.5 : 2.5 : 1, and a pH value 7 at 60°C for 4 h. Table IV shows the effect of $[\eta]$ on the APIf of cement slurry. The APIf increased as $[\eta]$ increases except for 0.5% ammonium persulfate as the initiator. When the ammonium persulfate and sodium bisulfite were used as initiator, the APIf changed a little until the amount of initiator exceeded 1.0%. Owing to the slurry containing the PANM with 0.5% ammonium persulfate as the initiator was too thick to prepare,

therefore, 1.0% dispersant (BWOC) was introduced into the slurry, which may affect the APIf of the cement slurry containing PANM according to Plank[13] and cause the APIf greater than PANM with 0.2% ammonium persulfate and sodium bisulfite as initiator. Generally, water-soluble polymers adsorbed onto the cement particle surfaces can adopt different conformations. These are "train" type (all functional groups positioned along the trunk chain of the polymer are bound to the surface), "loop," or "tail" type (only partial binding of the polymer to the surface). For FLCA, "loop" or "tail" type is more effectively than "train" type to control fluid loss.[17] High molecular weight polymer may be more inclined to adsorb onto the cement particle surfaces with "loop" or "tail" type; however, low molecular weight polymer may be more inclined to adsorb onto the cement particle surfaces with "train" type. In other words, only

Table VI. Analysis of L_{16} (4^5) Test Results

Test no.	A	B	C	D	E	K (Pa s^n)	APIf (mL)	H
1	A1	B1	C1	D1	E1	0.028	36.00	0.984
2	A1	B2	C2	D2	E2	0.083	30.00	0.983
3	A1	B3	C3	D3	E3	0.718	85.00	0.796
4	A1	B4	C4	D4	E4	0.536	96.00	0.811
5	A2	B1	C2	D3	E4	2.975	371.60	0.000
6	A2	B2	C1	D4	E3	1.682	200.82	0.466
7	A2	B3	C4	D1	E2	0.009	35.00	0.988
8	A2	B4	C3	D2	E1	0.248	27.00	0.960
9	A3	B1	C3	D4	E2	1.280	285.69	0.410
10	A3	B2	C4	D3	E1	0.783	33.00	0.861
11	A3	B3	C1	D2	E4	0.562	31.40	0.900
12	A3	B4	C2	D1	E3	0.042	31.00	0.989
13	A4	B1	C2	D2	E3	0.037	55.00	0.955
14	A4	B2	C1	D1	E4	0.015	50.00	0.966
15	A4	B3	C4	D4	E1	1.574	94.00	0.639
16	A4	B4	C3	D3	E2	0.846	34.00	0.849
K1	0.894[a]	0.587	0.800	0.982	0.861			
K2	0.604	0.819	0.653	0.950	0.808			
K3	0.790	0.831	0.783	0.627	0.802			
K4	0.852	0.902	0.904	0.582	0.669			
R	0.290[b]	0.315	0.251	0.400	0.192			
Optimal level	A1	B4	C4	D1	E1			

[a] $K_i^A = (\sum \text{the } H \text{ value of synthesized PANM at } A_i)/4$, [b] $R_i^A = \max\{K_i^A\} - \min\{K_i^A\}$.

Table VII. The Optimal Experiment Condition and the Performance of OPANM

Mole ratio of AMPS/AM/NNDMA/MA	Monomer concentration (%)	Initiator concentration (%)	pH	E (°C)	K (Pa s^n)	APIf (mL)	H
4/2.5/2.5/1	32.5	1.0	4	40	0.041	30.00	0.99

a fraction of the FLCA polymer functionalities actually adsorb onto the surface, with the remainder protruding freely into the cement pore solution in the form of loops and tails, and the unadsorbed polymer segments increase as the molecular weight increases. Thus the APIf decreases as the molecular weight of PANM increases. However, if the molecular weight of the PANM is excessively high, one polymer chain may bridge several cement particles and it is hard for the cement particle fully covered by the polymers, which is adverse for the fluid loss control ability of the PANM. Oversized FLCA also induces the slurry with a high viscosity. Thus, proper molecular weight of PANM is needed.

Orthogonal Experiment

An orthogonal $L_{16}(4^5)$ test design was used for optimization of the synthesis conditions.[20] Table V lists the schedule of the orthogonal experiment in which five key parameters including monomer mole ratio (A), monomer concentration (B), initiator concentration (ammonium persulfate and sodium bisulphite with weight ratio 1 : 1) (C), pH value (D), and temperature(E) were selected. Because the study involved multiobjective optimization, so the APIf and K were processed simply. The APIf and K were normalized, and both were given a 50% weight factor. The detailed process was presented in the following:

$$F = 1 - (K - \min(K))/(\max(K) - \min(K))$$
$$= 1 - (K - 0.009)/(2.975 - 0.009) \quad (3)$$

$$G = 1 - (APIf - \min(APIf))/(\max(APIf) - \min(APIf))$$
$$= 1 - (APIf - 27)/(371.6 - 27) \quad (4)$$

$$H = 0.5 \times F + 0.5 \times G \quad (5)$$

The bigger the H value will be, the smaller the APIf and the K will be. According to the orthogonal experiment design princi-

ple, the bigger the R^X (X could be A, B, C, D, or E), the more significant influence of X on the test result. Table VI shows that the influence on the H value of PANM decreases in the order: $D>B>A>C>E$ according to the R values. According to the orthogonal experiment design principle, the bigger the K_i^X (X could be A, B, C, D, or E, and i could be 1, 2, 3, or 4), the better the test result under the Xi condition. For example, according to Table VI, K_1^A is the biggest among K_1^A, K_2^A, K_3^A, and K_4^A, so $A1$ was chosen to be the optimal level for factor A. Table VII lists the optimal experiment condition optimized from the orthogonal test and the performance of OPANM which was obtained under the optimal condition. Under the optimal conditions, the APIf and K were 30 mL and 0.041 Pa s^n, respectively, which were better than the test result under other experiment conditions. An OPANM sample was prepared under the optimal experiment condition for other experiment test and evaluation. The intrinsic viscosity number of OPANM was 167.21 mL/g.

The Infrared Spectrum of OPANM

Figure 4 shows the FTIR spectra for the OPANM. The spectra shows typical absorption bands as follows: a band 3430 cm^{-1} attributed to υ_{-NH_2}; a band 2920 cm^{-1} attributed to υ_{-CH_3}; a band 1650 cm^{-1} attributed to $\upsilon_{-C=O}$; the bands 1650 and 1400 cm^{-1} attributed to carboxylate; as the carboxylic acid anhydride changed into carboxylate, the bands 1800 and 1760 cm^{-1} cannot be seen; a band 1440 cm^{-1} attributed to υ_{-CH_2-}; the bands 1040 and 1200 cm^{-1} attributed to υ_{-SO3}; a band 1220 cm^{-1} attributed to υ_{-C-N-}.

TG Analysis

The TG thermogram of OPANM was shown in Figure 5. As the temperature is below 300°C, a little mass loss which may be
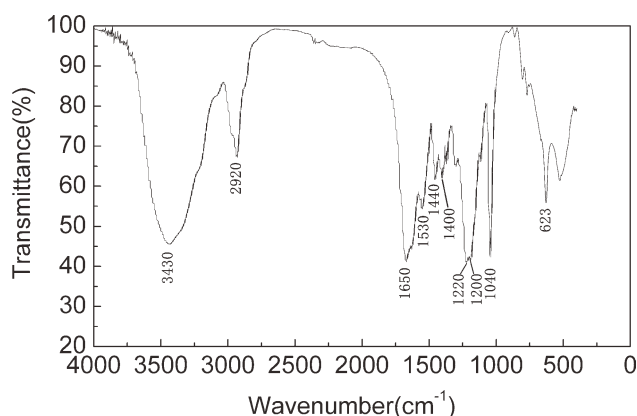
Figure 4. FTIR spectra of synthesized copolymer OPANM.
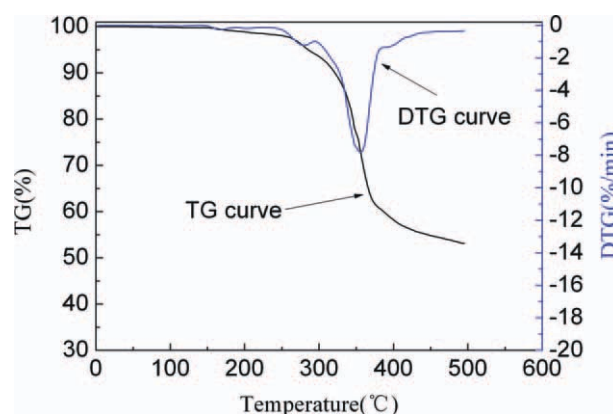
Figure 5. TG-DTG curve of synthesized copolymer OPANM. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
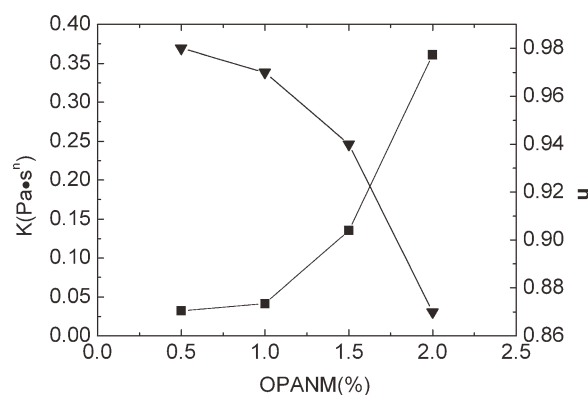
Figure 6. Effect of amount of OPANM on slurry rheological behavior (temperature = 52°C).

Figure 8. Effect of amount of OPANM on APIf (temperature = 120°C).

caused by desorption of the bound water adsorbed in the strong polar groups is observed. The thermal degradation of the copolymer became appreciable at ∼ 300°C. This indicates the OPANM may have an excellent heat resisting property. However, the TG analysis test circumstance is different from the real circumstance, which the OPANM exists in, the alkaline circumstance of the cement slurry may impair the thermostability of the OPANM to some extent.

The Evaluation of the OPANM

The impact of OPANM amount on the rheological behavior of cement slurries containing the OPANM is shown in Figure 6, and the cement slurry shows a good rheological behavior even when the dosage of OPANM is 2% (BWOC). Figure 7 shows the impact of temperature on the cement slurry containing 2% (BWOC) OPANM. When the dosage of OPANM is 2% (BWOC), the APIf is 47 mL at 150°C and 60 mL at 180°C. The impact of OPANM amount on the APIf is shown in Figure 8. The APIf of slurry without salt decreases as the dosage of OPANM increases, and the APIf is 19 mL with 2% OPANM (BWOC) at 120°C. The APIf of slurry with 30% salt (BWOW) is higher than the slurry without salt, which indicates the salt impairs the fluid loss control ability of the OPANM. The APIf is 33 mL with a 2% OPANM (BWOC) and 30% salt (BWOW) at 120°C, which indicates the OPANM with a good salt-resistant
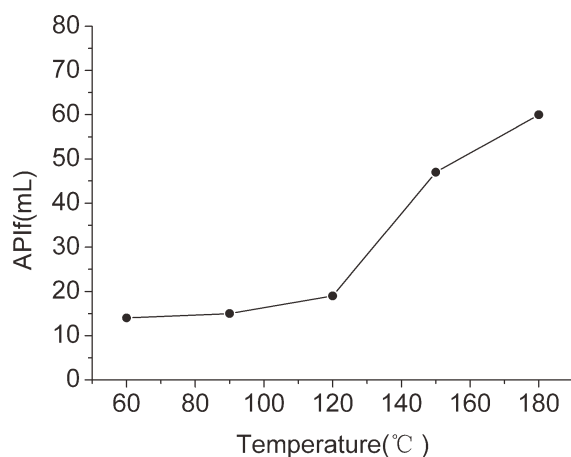
ability. Figure 9 shows the impact of temperature on the thickening time of cement slurry containing 1% (BWOC) OPANM. The thickening time decreases with the increasing of temperature and no over-retardation appears, and the phenomena of the thickening time longer in the higher temperature than in the lower temperature does not appear due to the limited mole fraction of MA.[8]

CONCLUSIONS

Aiming at good fluid loss control ability and excellent dispersibility, a new dispersive type FLCA has been synthesized in aqueous solution.

The optimal PANM (OPANM) was obtained under the optimum reaction conditions: mole ratio of AMPS/AM/NNDMA/MA = 4/2.5/2.5/1, monomer concentration = 32.5%, amount of (by weight of monomer) ammonium persulfate/sodium bisulfite = 1.0%, pH value = 4, and temperature = 40°C.

Through TG analysis, the OPANM has an excellent performance to resist high temperature and its chemical structure is stable below 300°C.

The OPANM has excellent fluid loss control ability, dispersing power, thermal-resistant and salt-tolerant properties. No over-
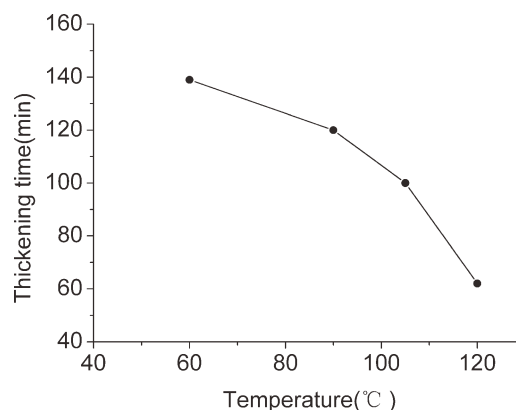

Figure 7. Effect of temperature on APIf (dosage: OPANM 2% BWOC).

Figure 9. Effect of temperature on thickening time (dosage: OPANM 1% BWOC).

retardation appears through testing the thickening time of the cement slurry containing the OPANM.

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