



# The ability of sodium metasilicate pentahydrate to adjust the compatibility between synthetic Fluid Loss Additives and Retarders applying in oil well cement



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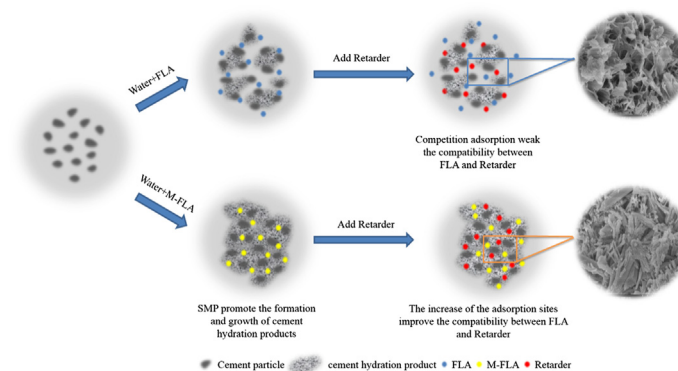
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## HIGHLIGHTS

- The increase of adsorption sites of cement hydration products is the key factor to SMP adjust the compatibility between the admixtures.
- Through the modification of pore size distribution of cement hydrated particles, SMP could effectively alleviate the super-retardation of cement slurry caused by the carboxyl group in the admixtures.
- The effects of SMP on the compatibility and the early strength of cement are achieved by promote the formation and growth of early cement hydration products.

## GRAPHICAL ABSTRACT

Schematic of sodium metasilicate pentahydrate adjusting the compatibility between synthetic Fluid Loss Additives and Retarders.



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## ABSTRACT

Sodium metasilicate pentahydrate (SMP), a type of hardening-accelerator, was widely used in cement slurry to enhance its early strength. In this study, we found that SMP had a novel ability to adjust the compatibility between 2-Acrylamido-2-Methyl Propane Sulfonic Acid (AMPS)-based Fluid Loss Additive (FLA) and Retarder in oil well cement. Reasons for this significance were proposed, that SMP promoted the formation and growth of hydration products by accelerating the hydration velocity of cement slurry, which increased the adsorption sites of cement hydration products. As that happens, the more AMPS-based polymers were adsorbed simultaneously on the surface of the hydration product, thereby effectively improving the compatibility problem caused by the competitive adsorption between the admixtures. Meanwhile, while SMP speeded up the hydration reaction, the pores space of cement hydrated particles were filled with more hydrates and consequently altered its pore size distribution, whose action was also beneficial to the development of early strength. Overall, those findings will provide further interpretation for the effect of admixture on cement hydration products and offer some useful references for practical application of oil well cementing in the future.

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**Abbreviations:** FLAs, Fluid Loss Additives; SMP, Sodium Metasilicate Pentahydrate; AMPS, 2-Acrylamido-2-Methyl Propane Sulfonic Acid; NVP, N-Vinyl-2-Pyrrolidone; IA, itaconic Acid; MA, Maleic Anhydride; NaOH, sodium hydroxide; APS, ammonium persulfate; NNDMA, N,N-dimethyl acrylamide; API, American Petroleum Institute; BWOW, by weight of water; BWOF, by weight of fluid loss additive; HTHP, high temperature, high pressure.

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## 1. Introduction

Synthetic polymer Fluid Loss Additive (FLA) and Retarder constitute one of the most popular admixtures used in cementitious systems. In cement they are applied for two different purposes: FLA is added to prevent water mud filtrate entering the formation, which results in the deterioration of cement slurry's fluidity and the destruction of the reservoir [1], and the Retarder is utilized to achieve sufficient pumpability time over long distances by delaying the setting time of the slurry [2]. Due to these superior properties, considerable work has been conducted to study the synthesis method, reaction monomer and performance of synthetic admixtures. Especially, 2-Acrylamido-2-Methyl Propane Sulfonic Acid (AMPS) based polymer has become the most widely researched and applied admixtures, owing to its excellent heat resistance, salt tolerance and stable performance characteristics [3–6].

Considering the complexity and variability of oil well cement system during the hydration process, which incorporates several admixtures such as FLA, Retarder and dispersant etc. Increasingly more researches are focused on the compatibility and interaction mechanism among admixtures at present [7–12]. For example, Plank J illustrates the presence of a dispersant negatively impact the performance of AMPS-based copolymers. As a consequence of competitive adsorption between FLA and other admixtures, the amount of FLA adsorption on cement decrease, and poor performance on water retention ability is observed for the AMPS-based copolymers, whose result is also proved by other reports [3,13,14]. Such examples are indicative of the complexity of multi system compatibility adjustment in practical application of oil-cement slurries.

However, to date, the compatibility between synthetic admixtures in oil well cement has been researched for many years, the exact working mechanism is not yet fully understood. Related theories are confined primarily to the competitive adsorption between admixtures, there are few reports about the effect of admixtures on the formation and growth of cement hydration products.

Thus, the aim of this investigation is to start with the interaction between the admixtures and cement hydration process, through the introduction of SMP to solve the incompatibility problem between AMPS-based FLA and Retarder in oil well cement slurry, and relevant mechanism is characterized by means of conduction calorimetry, X-ray powder diffraction, Raman Spectroscopy, Solid state NMR, total organic carbon and Zeta potentials. Meanwhile, an adsorption-desorption isotherms and Barrett-Joyner-Halenda test are employed to evaluate the variation of the surface area and pore size distribution of cement hydrated particles, thereby illustrating the influence of SMP on early compressive strength development of cement slurries. Finally, the effect of SMP on morphologies of the hydration process of cement is analyzed with Scanning Electronic Microscope observations at different curing time.

## 2. Material and methods

### 2.1. Materials

2-Acrylamido-2-Methyl Propane Sulfonic Acid (AMPS) and N,N-dimethyl acrylamide (NNDMA) were supplied by RBL Chemicals (Beijing, China) and used as received. N-vinyl-2-pyrrolidone (NVP, distilled before use), ammonium persulfate (APS), itaconic acid (IA) and sodium metasilicate pentahydrate (SMP) were purchased from Aladdin Industrial Corporation (Shanghai, China). Maleic anhydride (MA, purified by recrystallization from anhydrous benzene) and sodium hydroxide (NaOH) were obtained from KeWei Chemical Reagent (Tianjin, China). Distilled water was used throughout the work, which was prepared by a water purification system (Ulupure, Chengdu, China).

### 2.2. Syntheses of polymers

#### 2.2.1. Syntheses of Fluid Loss Additives

FLA (200 g, solid content: 20 wt%) was synthesized via free radical aqueous solution copolymerization using APS as initiator. The FLA was synthesized following the method described by Lummer et al. [15] with some modifications. AMPS, NNDMA and MA (molar ratio of 1:0.63:0.105) were added into the beaker with distilled water, after adjust the pH value of the solution mentioned above to 6 using 0.5 mol/L NaOH solution. Then the solution was removed into a three neck flask with mechanical stirring, and heated to 60 °C. The initiator (ammonium persulfate, 0.8% of solid quality) was dissolved in the distilled water, and then dropwise added into the flask slowly under N<sub>2</sub> protection. The Fluid Loss Additives were obtained after conducting the reaction under the desired temperature for 4 h. Modified FLA (M-FLA) was realized by introducing SMP, which was synthesized in similar with the method of FLA. The mass ratio of SMP to FLA ranged from 0.5% to 2.5%. Details were shown in Table 1.

#### 2.2.2. Syntheses of Retarder

AMPS, NVP, and IA at the molar ratios of 1: 0.175: 0.410 were added into a beaker filled with distilled water, and then make it was fully dissolved by magnetic stirring [3,16]. Then the solution was removed into a three neck flask with stirrer, and heated to 45 °C. The initiator (ammonium persulfate and ethidene diamine with molar ratio 1:1 in aqueous solution) was dropped into the flask slowly under N<sub>2</sub> protection. The Retarder was obtained after the reaction proceeding under the desired temperature for 4 h.

### 2.3. Purification and characterization of polymers

#### 2.3.1. Purification of polymers

The polymers (FLA and Retarder) were isolated through dialysis and lyophilization to yield a white spongy solid. <sup>1</sup>H-NMR spectra (400 MHz instrument using a BRUKER WP400 spectrometer) and Fourier transforms infrared spectroscopy (Nicolet 6700, NEXUS, USA) were employed to confirm the formation of polymers.

#### 2.3.2. <sup>1</sup>H-NMR spectra

The composition of the polymer was determined by <sup>1</sup>H-NMR analysis (using D<sub>2</sub>O as the solvent). The <sup>1</sup>H-NMR spectrum of FLA was shown in Fig. 1a. The two peaks at 1.40 and 2.52 ppm were assigned to the protons of —CH<sub>2</sub>— and —CH— in the main chains, respectively. —NCH<sub>3</sub> could be contributed to the signal at 3.02 ppm. Furthermore, the peak at 2.84 ppm was assigned to the protons of —CH<sub>2</sub>— in MA.

Fig. 1b showed the <sup>1</sup>H-NMR spectra of AMPS-IA-NVP copolymer as a Retarder. The characteristic —CH<sub>3</sub> signal of AMPS appeared at 1.36 ppm. Similarly, the signal at 3.54 ppm appeared due to the characteristic —CH<sub>2</sub>N existing in NVP. Furthermore, the peak at 2.90 ppm was assigned to the protons of —CH<sub>2</sub>— in IA.

#### 2.3.3. Fourier transforms infrared spectroscopy (FT-IR) measurements

After FLA and Retarder were washed, purified, dried and ground, they were prepared by pressing the mixture of the 1 mg sample and 100 mg KBr, then the molecular structure and chemical compositions of the sample were characterized by FT-IR spectroscopy in the range of 4000 cm<sup>-1</sup> and 500 cm<sup>-1</sup>.

Fig. 2a showed the FT-IR spectra of AMPS-NNDMA-MA copolymer as a FLA. The peak at 3438.89 cm<sup>-1</sup> was assigned to the stretching vibration of N—H of AMPS, the peak at 2979.85 cm<sup>-1</sup> was assigned to the —CH<sub>3</sub> of NNDMA; the peak at 1662.55 cm<sup>-1</sup> was assigned to —C=O of MA; the absorption peak of 1542.97 cm<sup>-1</sup> was belonged to the stretching vibration of —C—N, and the peak at 1047.29 cm<sup>-1</sup> was assigned to —S=O group.

Fig. 2b showed the FT-IR spectra of AMPS-IA-NVP copolymer as a Retarder. The peak at 3426.89 cm<sup>-1</sup> was assigned to the stretching vibration of N—H of AMPS; the peak at 1727.43 cm<sup>-1</sup> was assigned to the —CON of NVP; the peak at 1644.99 cm<sup>-1</sup> was assigned to —C=O of IA. The absorption peak of 1552.42 cm<sup>-1</sup> was due to the stretching vibration of —C—N, while the peak at 1040.78 cm<sup>-1</sup> was assigned to —S=O group.

**Table 1**

The constituents of admixtures.

kinds of admixtures	abbreviations	chemical constituents	molar ratio
Retarder	R	AMPS:NVP:IA	1:0.175:0.410
Fluid Loss Additive	FLA M-FLA	AMPS:NNDMA:MA AMPS:NNDMA:MA (containing SMP)	1:0.63:0.105 1:0.63:0.105: (from 0.25% to 2.5%) <sup>a</sup>

Note:

<sup>a</sup> The mass ratios of SMP to FLA range from 0.25% to 2.5%.

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