



Preparation and performance of a lignosulfonate–AMPS–itaconic acid graft copolymer as retarder for modified phosphoaluminate cement



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HIGHLIGHTS

- A new type of lignosulfonate-based graft copolymer OLSAI was studied by single factor analysis and orthogonal experiment.
- The OLSAI has good retarding ability, thermal-resistant properties and little impact on the strength of MPC.
- The comprehensive evaluation indicates that the OLSAI will enable MPC to be applied to high-CO₂ oil-well cementing.

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ABSTRACT

In this article, a new type of graft copolymer retarder as modified phosphoaluminate cement (MPC) has been discussed. Graft copolymerization of 2-acrylamido-2-methyl propane sulfonic acid (AMPS) and itaconic acid (IA) onto sodium lignosulfonate (LS) was performed using potassium persulfate as an initiator by free radical aqueous solution copolymerization. The optimal LS/AMPS/IA copolymer (OLSAI) was obtained under the optimum reaction conditions: mass ratio of LS/(AMPS + IA) = 1:1, temperature = 60 °C, mole ratio of AMPS/IA = 9:1, initiator concentration = 1%, pH value = 5. The synthesized copolymer OLSAI was characterized by FTIR analysis. The experimental results demonstrate that the OLSAI has excellent retarding ability, dispersing power, thermal-resistant and pressure-tolerant properties, and little negative impact on the compressive strength of MPC. The graft copolymer OLSAI is expected to be an excellent retarder for MPC.

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1. Introduction

Geological storage of CO₂ in exploited oil and gas reservoirs has a huge potential for reduction of greenhouse gases emitted into the atmosphere. One of the problems associated with this method of carbon capture and storage is the degradation of conventional Portland cement under the attack of CO₂ which ultimately can lead to CO₂ leakage from the well [1–3]. The degradation of Portland cement by CO₂ involves CaCO₃ formation from calcium hydroxide formed during the hydration of the calcium silicate phases C₃S and C₂S. Once all Ca(OH)₂ has been consumed, even calcium silicate hydrates (C–S–H phases), the main component present in hardened cement which is providing its strength, will be attacked and converted into hydrous silica (SiO₂·nH₂O) [4–6]. To resolve these problems, new cementing systems free of CO₂ reactive components have been introduced [7–11].

One of the most promising systems is modified phosphoaluminate cement (MPC) which will not react with CO₂ at all even high temperature and high pressure. MPC consists of three main components: phosphoaluminate cement, sodium hexametaphosphate (SHP) and water. Phosphoaluminate cement which contains its own cementitious phases with high hydration performances is a type of special cement developed by Li et al. [12]. The main phases of phosphoaluminate cement are a new quaternary compound designed according to crystallochemistry principle that is tentatively called “phase LHss”, modified CA and α-C₃P, and a certain amount of amorphous phase [13]. The general hydration products are apatite crystal (Ca₅(PO₄)₃OH), hydrated calcium aluminate crystal (CXAHY), hydrated calcium aluminate–phosphate gel (C–A–P–H). As a kind of admixture, SHP has the ability of improving compressive strength of the phosphoaluminate cement [11]. MPC possess the characteristics of rapid solidification, rapid hardening and good CO₂-resistant properties.

Oil-well cementing is one of the most important operations in the construction of a well bore. To assure applicability of MPC in

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the field it is crucial to provide a suitable retarder for this new cement system. However, due to the specific mineral composition of MPC, the majority of conventional cement retarders do not work well here [14]. In fact, extending the hydration time of this cementing system to allow for thickening time of over 4 h at elevated temperatures (above 70 °C) was reported to present a major challenge. Though sodium lignosulfonate can prolong the thickening time of MPC to over 6 h, the compressive strength of cured cement has been reduced significantly [15].

Nowadays, the most promising water-soluble polymer retarders might be the AMPS-based polymers which show outstanding thermal-resistant and salt-tolerant ability [16]. This study aimed to develop an effective graft copolymer retarder which was synthesized using sodium lignosulfonate (LS), 2-acrylamido-2-methyl propane sulfonic acid (AMPS) and itaconic acid (IA). The graft copolymerization reaction was accomplished with a variation of reaction parameters such as the mass ratio of LS/(AMPS + IA), the mole ratio of AMPS/IA, and the initiator concentration. The synthesized copolymer OLSAI in this article not only has excellent retarding effect, but also could be used without dispersant existing in MPC.

2. Experimental details

2.1. Synthesis of OLSAI

Graft copolymers of LS, AMPS and IA were prepared by aqueous solution polymerization technique in the laboratory. LS was obtained from Xin Long Hai Company Limited (Jinan, China), and the degree of sulfonation is 1.62 mmol/g. AMPS, IA, sodium

hydroxide, hydrochloric acid and potassium persulfate were all chemically pure substances. The synthesis of OLSAI was conducted in the following specific sequence: (i) sodium hydroxide or diluted hydrochloric acid was added to regulate pH value of aqueous solution, (ii) the monomers (LS/AMPS/IA) were dissolved in the deionized water by stirring it in nitrogen atmosphere and the total monomer concentration was 20% of aqueous solution, (iii) the reaction mixture was heated for 30 min to the desired temperature, (iv) added, dropwise, the initiator solution into the reaction mixture, and (v) the reaction was allowed to proceed for 3.5 h. A possible reaction equation of grafting the AMPS and IA onto the LS structure is shown in Fig. 1.

2.2. Copolymer characterization

The graft copolymer OLSAI was rinsed in a large amount of acetone and dried in vacuum at room temperature. FTIR spectra of OLSAI were recorded using a NEXUS Fourier Transform Interferometer.

2.3. Cement slurry preparation

Cement slurries were prepared in accordance with the test procedure set forth in API recommended practice for testing well cements 10B, 23rd, April 2002 [17]. An organic silicon anti-foaming agent used in conventional Portland cement was chosen. Sodium hexametaphosphate containing 68.7% of P_2O_5 was used as admixture to enhance compressive strength. Phosphoaluminate cement was obtained from Zibo, China, and the phase composition and physical properties of this cement are presented in Table 1. Cement slurry compositions with densities of 1.87 g/cm³ were prepared comprising phosphoaluminate cement, SHP (15% by weight of cement, BWOC), water (50% BWOC), a certain amount of OLSAI, anti-foaming agent (0.7% BWOC). The cement slurry was mixed with the Waring blender.

2.4. Thickening time

Thickening time of the cement slurry was determined following API RP 10B using the HPHT (high pressure-high temperature) consistometer [17]. The consistency of the slurry expressed in Bearden units of consistency (Bc) was measured.

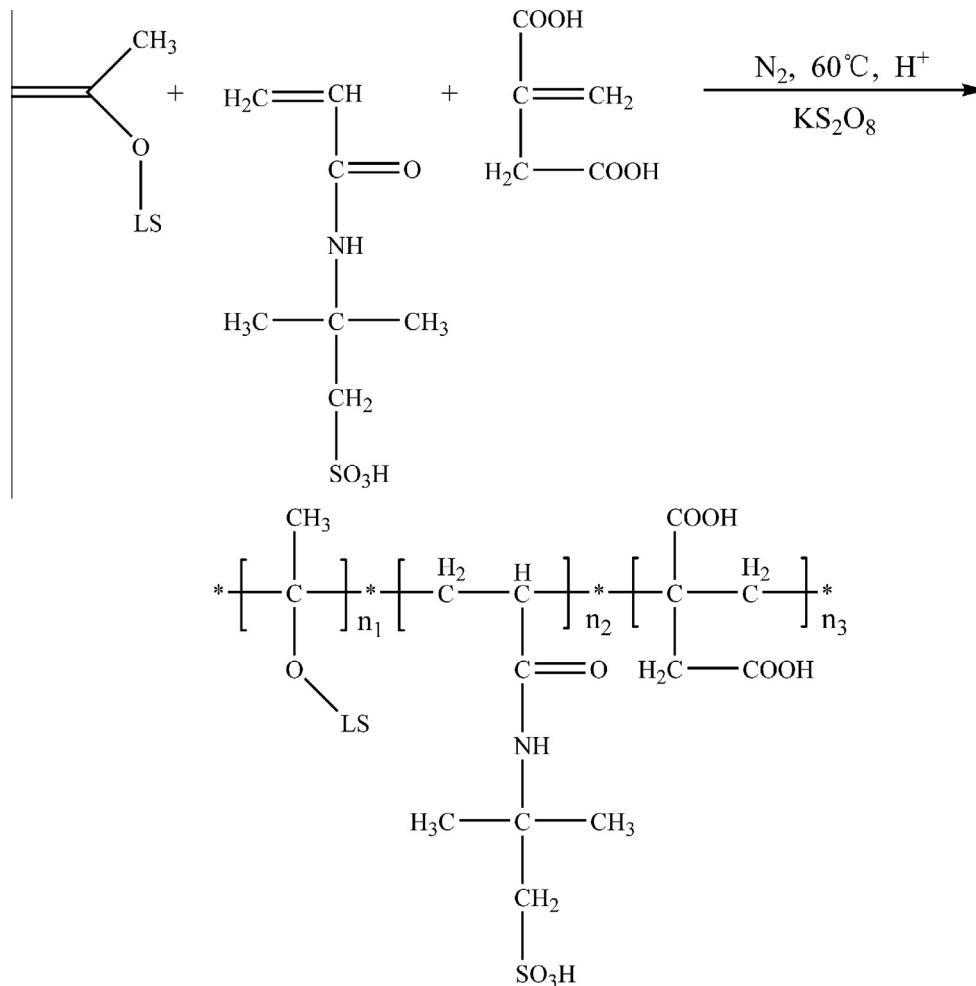


Fig. 1. Schematic diagram of graft copolymerization.

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