

Studying the physico-chemical properties of commercially available oil-well cement additives using calorimetry

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Abstract Cement additives are typically used to modify the behavior of oil-well cement and to control its fluidity under well conditions. In this study, the retardation effect on cement hydration is investigated for a commercially available lignosulfonate and an NSF condensate at seven different concentrations. Additive solutions at 0.1% and 0.2% each by weight of cement (bwoc) with a ratio of (1:1) are also studied. The retardation of cement hydration process is monitored via isothermal calorimetry. Rheological studies are conducted to study the plasticizing effect induced by these additives. The mechanisms accompanying this process are better understood by studying the morphology of cement/additives systems using environmental scanning electron microscopy. The results show clearly that NSF has a retardation effect on cement hydration reflected on crystal growth. In addition, rheological measurements show that sodium lignosulfonate is more effective than NSF. The rheological effect alters with different cement/additive systems. This article provides recommendations for applying the most effective additive dosages in drilling and well-completion operations as well as enhancing the well-cementing quality.

Keywords Oil-well cement · Retarders · Dispersants · Mechanism · Calorimetry

List of symbols

C ₃ S	Tricalcium silicate (Ca ₃ SiO ₅)
C ₂ S	Dicalcium silicate (Ca ₂ SiO ₄)
C ₃ A	Tricalcium aluminate (Ca ₃ Al ₂ O ₆)
C ₄ AF	Tetracalcium aluminoferrite (Ca ₄ Al _n Fe _(2-n) O ₇)
AF _t	Ettringite ([Ca ₆ Al ₂ (OH) ₁₂](SO ₄) ₃ ·26H ₂ O)
AF _m	Monosulfate ([Ca ₄ Al ₂ (OH) ₁₂](SO ₄)·16H ₂ O)
Portlandite	Calcium hydroxide (Ca(OH) ₂)
C–S–H	Calcium silicate hydrates [(CaO) ₃ (SiO ₂) ₂ ·4(H ₂ O) + 3Ca(OH) ₂] + [(CaO) ₃ (SiO ₂) ₂ ·4(H ₂ O) + Ca(OH) ₂]
Syngenite	(K ₂ SO ₄ ·CaSO ₄ ·H ₂ O)
ESEM	Environmental scanning electron microscope
WOC	Wait on cement
bwoc	By weight of cement

Introduction

Oil-well cementing is considered as one of the most critical operations in petroleum and gas industry. Therefore, it has been an interesting area of research for the last years. The quality of this operation has a huge impact on well production. Nevertheless, unpleasant accidents could happen due to its failure. The main objectives of well cementing are to support vertical and radial loads applied to casing which is placed in the drilled borehole, protecting it from corrosion, sealing of abnormal pressure formations, and providing zonal isolation which means to prevent producible fluids and gas from escaping [1, 2]. Complete zonal isolation provides accurate well testing, maximum recovery, and more effective well simulation at the least cost [3, 4].

Manufacturing Portland cement needs two types of raw materials: “calcareous” which contain lime (calcium

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carbonate) and “argillaceous” which contain alumina, silica, and iron oxide [5]. At 1450 °C, calcium carbonate is converted to calcium oxide, or lime, and the clay minerals yield dicalcium silicate (Ca_2SiO_4) and other inorganic oxides such as aluminates and ferrites. Further heating melts the aluminate and ferrite phases. Tricalcium silicate (C_3S) is responsible for early strength, while dicalcium silicate (C_2S) is very important for cement final strength [1, 6]. Tricalcium aluminate (C_3A) hydrates rapidly and contributes most to heat of hydration. Also, C_3A plays an important role in early strength while tetracalcium aluminoferrite (C_4AF) has little effect on physical properties of the cement [1]. Hydration of cement is defined as combination of all chemical and physical processes taking place after contact of anhydrous solid with water [7]. The kinetics mechanisms of cement hydration are complex and have been an interesting area of research compared to hydration of individual clinker phases [8, 9]. In addition, many theories are proposed to explain the mechanisms of hydration stages [7, 9–11]. The C_3S and C_2S phases are reacting with water to form calcium hydroxide $\text{Ca}(\text{OH})_2$ and calcium silicate hydrates (C–S–H) [7, 8]. Hydration of C_3A and C_4AF in the presence of gypsum initially produces ettringite (AF_t phase). The ettringite phase is unstable under these conditions and gradually converts into more stable monosulfate (AF_m phase) [7, 8, 12, 13].

Retarders are defined as the chemical additives that prolong setting time of cement and prevent premature hardening [14]. Typically, the perfect retarder for oil-well cement is the one which extends the cement setting for adequate time and then suddenly allows the cement hydration to proceed at a rapid rate [15]. There are different kinds of oil-well cement retarders such as calcium and sodium lignosulfonates, saccharide compound, cellulose derivative, organic phosphate, and some inorganic salts (such as borate, phosphate, and chromate) [5, 16]. These retarders are classified according to their chemical nature and the cement phase (C_3S or C_3A) they act on. The type of cement and the operating well conditions play an important role in the retarder selection, too [5, 6].

In general, four theories are proposed to explain the hydration inhibition mechanism. The adsorption theory suggests that the retardation occurs due to the adsorption of the retarder onto the surface of the hydration products thereby inhibiting contact with water [6, 14, 15]. The precipitation theory suggests that the retarder reacts with calcium and/or hydroxyl ions in the aqueous phase and forms an insoluble and impermeable layer on the cement grains [6, 8, 15]. The nucleation theory suggests that the retarder adsorbs on the nuclei of the hydration products, poisoning their future growth [14]. Finally, the complexation theory states that the calcium ions are chelated, preventing the formation of nuclei [6, 14]. The complexation

and precipitation theory generally considers to be unlikely because of the chelate–retarder relationship [17]. Recently, a fifth mechanism (“dissolution–precipitation”) is proposed which is exemplified by the dissolution of calcium via extraction with nitrilotris(methylene)phosphonic acid that exposes the aluminum-rich surface to enhance (catalyze) hydration, followed by precipitation of a layered calcium phosphonate that binds to the surface of the cement grains, inhibiting further hydration by acting as a diffusion barrier to water as well as a nucleation inhibitor [8, 15]. This mechanism is considered a special case of surface adsorption [17].

Dispersants (referred to plasticizers and superplasticizers) have been critical in the achievement of higher concrete strengths [18]. Adding dispersants to cement systems have two objectives: first, controlling the flow properties and second, reducing the water to cement ratio while maintaining workability to reach high strength and durability [18, 19]. Old class of cement SPs are based on lignosulfonate or sulfonated melamine or naphthalene formaldehyde condensates, while polycarboxylate (PC) is considered relatively as new class generation [18, 20, 21]. In general, certain amounts of anionic SPs are adsorbed on the surface of the cement grain or its hydrated phases to obtain dispersing effect [20]. Their dispersing effect is due to the adsorption of polymers on particle surfaces and presence of electrostatic and/or steric repulsive forces [19].

In a previous study, the adsorption phenomena is investigated with two different polycondensates superplasticizers (classical) on different cement systems as well as their effects on cement hydration peak [20]. It is found that the adsorption of SPs on cement system is affected by the anionic charge of polymer in water. Also the presence of sulfate in the cement system negatively affects the adsorption of SPs due to compatibility issues. Al-Wahedi et al. [22] studied the chemical and rheological behavior of oil-well cement in the presence of polycondensates SPs. It is stated that SPs enhance the workability as well as the rheology of oil-well cement. Bassioni [12] studied the adsorption of SPs on hydration products via zeta potential measurements. It is found that the zeta potential must be positive for better SPs adsorption. Also it is declared that ettringite and monosulfate show positive zeta potentials while syngenite, protlandite, and gypsum show zero or negative zeta potentials and do not adsorb SPs. This necessitates the optimization of superplasticizers dosages to control retardation since some SP are found to show some retardation effect [23].

Using several additives in cement can cause undesirable interactions [24]. Although, very good recipes have been developed, problems of compatibility between cement and additives have arisen [20]. Therefore, intensive research in this area is a must [20, 24].

The goal of this investigation is to evaluate a commercially available retarder in the presence of a dispersant, given the code R2 and D2. The study starts with analyzing the retardation effect on oil-well cement using thermal analysis measurement of both additives individually. In addition, relating to their synergistic effect solutions of additives at 0.1% and 0.2% (by weight of cement, bwoc) each with a ratio (1:1) are compared with individual solutions at the same concentration.

The influence of those additives on rheological behavior of cement slurry is examined for four different systems and compared to the neat cement slurry. The morphology of cement/additives systems is observed to explain the mechanisms induced by the additives on oil-well cement using ESEM.

Experimental

Sample preparation

All experiments are conducted in the Center for Applied and Environmental Chemistry at the Petroleum Institute in Abu Dhabi. Oil-well cement Class G is used. The commercial sodium lignosulfonate (retarder) and sodium naphthalene sulfonic acid–formaldehyde condensate (dispersant) which are used in this study are obtained from international service companies. The retarder and dispersant are given the names R2 and D2, respectively, and their identified structures are shown in Fig. 1a, b.

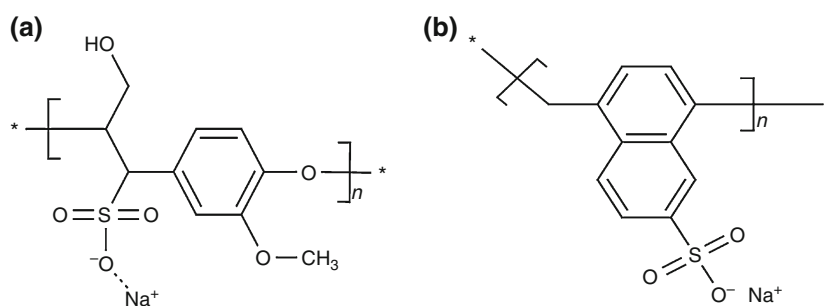
For each of the studied additives R2 and D2 seven different concentrations (0.1–0.7%) (bwoc) are prepared. Also solutions of 0.1% and 0.2% each (bwoc) of R2 + D2 with a ratio of (1:1) are prepared. The water-to-cement ratio (w/c) is set at 0.4 for all experiments. Sonication is used for 1 h for each solution to homogenize the mixtures. Samples and experiments are prepared and conducted at 25 °C temperature.

Thermal analysis measurements

The thermal analysis measurements are conducted by isothermal calorimetry (TAM air, TA, USA) as shown in Fig. 2

at constant temperature 25 °C. In the beginning, the eight channels are balanced using the general performance test (GPT). The system is calibrated using the gain calibration criteria for almost 1 h. The system is stabilized for 24 h. During this time eight reference ampoules are prepared with distilled water. The amount of distilled water for reference ampoules is calculated by summation of heat capacity fractions for cement and water over the heat capacity of water, which gives a value of 2.3656 g [25]. The reference ampoules are loaded into calorimetry chambers and the system is stabilized for another 24 h to make sure that the signals are measured in a range between $\pm 10 \mu\text{W}$ from the reference point (0 μW). The results are normalized to the mass of cement (4 g). The initial baseline duration is defined for 15 min with stability criteria of absolute slope value and standard deviation $< 4 \mu\text{W/h}$ and 4 μW , respectively, while the maximum waiting time for stability is 1 h. The following procedures are done in series arrangement with a gap of 2 min between two samples. During the initial baseline stability, eight samples with 4 g of cement are weighted accurately with electronic balance and transferred to disposable glass ampoule used specially for isothermal calorimetry measurements. A 1.6 ml of additives solution is poured into the cement ampoule using a micropipette. An aluminum cap is placed onto the ampoule and crimped to produce a perfect sealing for cement/additives system as shown in Fig. 3a and b, respectively. The samples ampoules were mixed for 1 min homogeneously by a test tube shaker (Vortex-Genie 2, Scientific Industries, USA). A centering tool is used to make an indication in the cap because it will provide guidance for the lifting eyelet as shown in Fig. 3c and d, respectively. The lifting eyelet is threaded into the cap as shown in Fig. 3e. All glass ampoules are wiped from outside using acetone to clean any sample traces and fingerprints before loading the sample ampoules. The black cover of the sample chamber and then the aluminum heat sink lid are removed using the lifting tool as shown in Fig. 3f. The sample ampoules are loaded into the calorimeter sample chamber. The heat sink lid and then the black cap are replaced back into the calorimeter sample chamber. The hydration process of cement is monitored until it

Fig. 1 Typical molecular structure of studied additives
a sodium lignosulfonate
b sodium naphthalene sulfonate formaldehyde



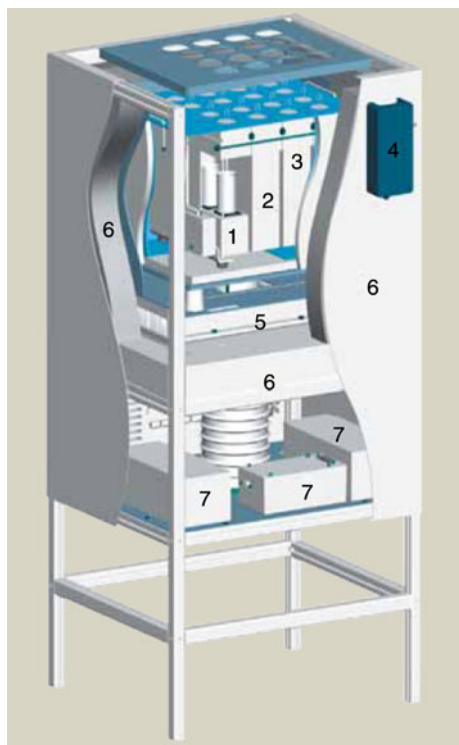


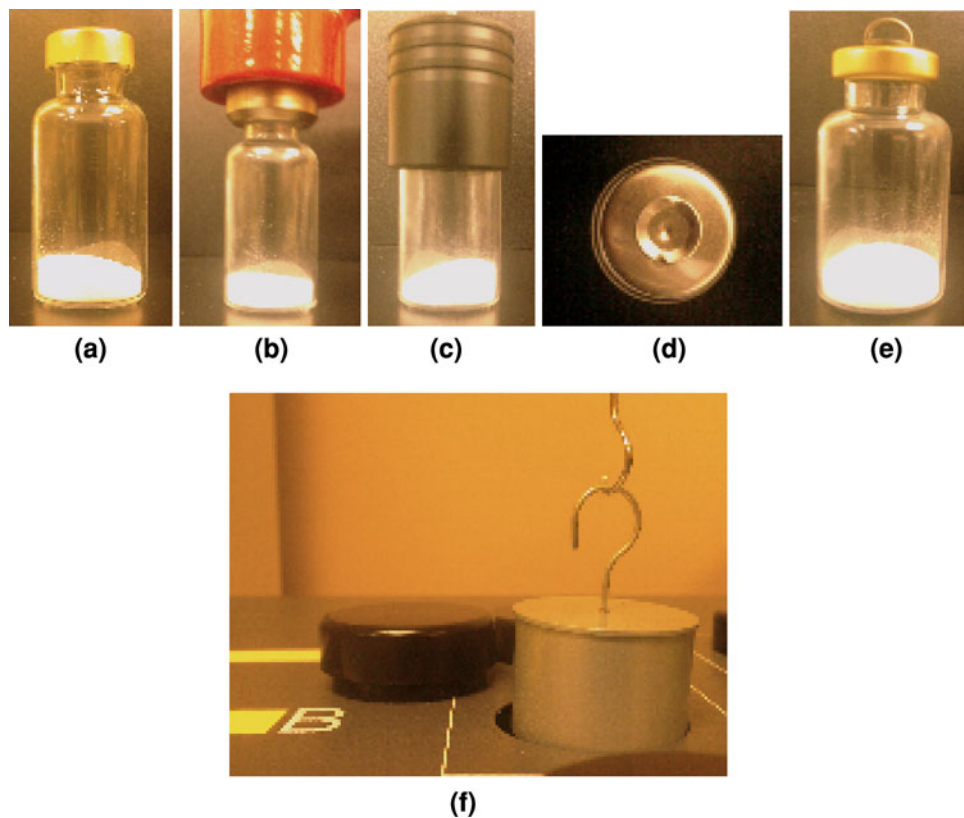
Fig. 2 A cut-away drawing of isothermal calorimetry with the top insulated lid removed [30]

reaches a minimum. The final baseline is established with the same criteria of initial baseline. The resulting data are exported to spreadsheets for further analysis. The heat evolved during the hydration process is measured in mW/g. The time elapsed for the cement to hydrate is measured in h.

Rheology measurements

Rheology of oil-well cement slurry is concentrated on four different additive systems and compared to the blank one. These systems are 0.2% bowc R2 and D2 alone and mixtures with a ratio (1:1) of R2 + D2 at 0.1% and 0.2% each bowc. A 650 g of oil-well cement, 260 g of distilled water, and solid additives are weighed using an electronic balance. The cement slurries are prepared and mixed according to ASTM C-305 using cement blinder (Toni-MIX, Toni Technik Baustoffprüfssysteme GmbH, Germany) [26]. The water is subsequently poured into the mixing bowl then the required amounts of solid admixtures are added. The mixing is started at low speed to ensure that the additives are fully dispersed in water for 15 s. The cement is added gently to the solution and is allowed to settle for almost 30 s. The cement is mixed at low speed (145 ± 5 r/min) for 30 s. The mixer is stopped for 15 s.

Fig. 3 Sample preparation for isothermal calorimetry



During this time the cement on the side of the bowl is scraped down into the batch. The cement is mixed again at medium speed (285 ± 10 r/min) for 1 min. According to API RP 10-B2, the cement slurry is stirred for 20 min at low speed (145 ± 5 r/min) [27].

The rheology test is conducted according to API RP 10-B2 specification using (OFITE 800 Viscometer, OFI testing equipments, USA) [27]. The test is conducted at constant temperature 25°C . The shear stress is measured at shear rates of 3, 6, 30, 60, 100, 200, 300 r/m. The shear rate is measured in s^{-1} . The viscosity is measured in mP s.

Environmental scanning electron microscopy (ESEM)

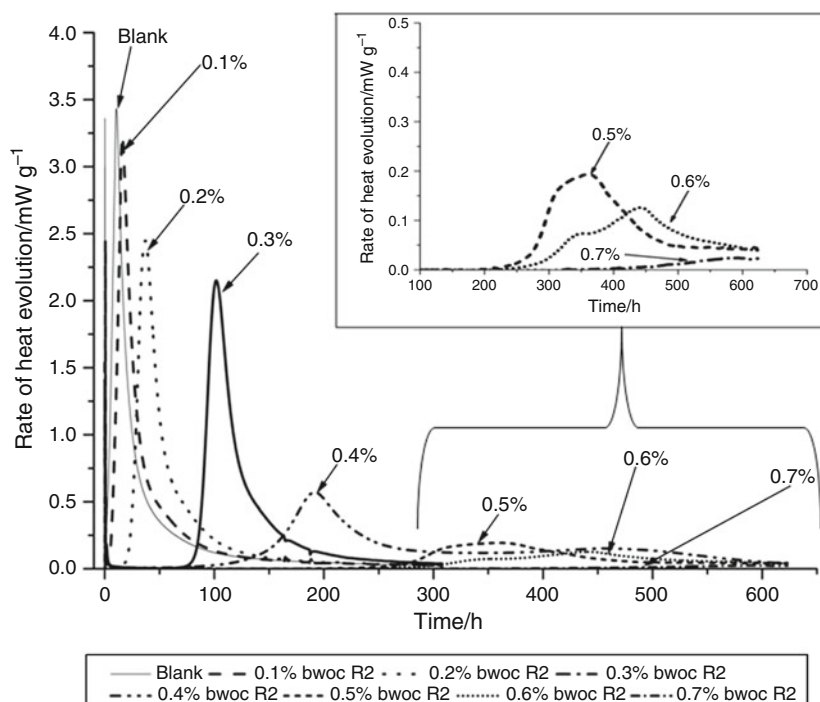
The microstructure and morphology of cement/additives systems are analyzed using an ESEM (FEI Quanta, FEG250, USA). These analyses are providing better understanding of cement–additives interaction and mechanisms. The hydrated cement/additives mixtures obtained from the isothermal calorimetry are fractured into small pieces and coated with gold. The ESEM measurements for cement samples are conducted at high vacuum and 5–10 kV. In addition, neat cement powder and hydrated blank cement are observed for comparison. Solid sample of R2 at high vacuum and 10 kV are monitored by ESEM. The samples are attached to the sample holders by copper tapes.

Results and discussion

Thermal analysis

Cement hydrates upon addition of water evolving heat due to the exothermic reaction accompanying this process. Therefore, isothermal calorimetry is a useful technique to monitor that heat. The effect of different additives on cement hydration compared to the plain ones is observed using heat flow calorimetry as well. The hydration peak gained from the heat calorimetry is divided into five different stages [10]. The impact of different cement/additives systems is illustrated in Figs. 4, 5, and 6 with respect to the plain cement. The hydration peaks shown in the figures are the sum of simultaneous ongoing hydration processes of different clinker phases in the presence of different additives [19]. Figure 4 represents the effect of R2 on the hydration process which has been fully investigated by the authors in a previous study. It is shown that R2 prolongs the dormant period (stage 2) with decreasing the peak maximum of heat released during the hydration process. In other words, the hydration peak is shifted by increasing R2 concentration compared to the blank cement at constant water-to-cement ratio. As can be seen in Fig. 4, the peak maximum for the blank cement sample is found at 10.7 h while at 0.7% bowc of R2 the hydration peak maximum starts to rise after 25 days which is considered as an overdose of R2. It is stated that increasing the retarder's concentration continuously increases the time of hydration

Fig. 4 The heat flow during oil-well cement hydration as function of retarder R2 dosage



process but not in a linear way. The exact retardation mechanism of lignosulfonate on oil-well cement is not fully determined yet. It has been postulated that the mechanism associated with lignosulfonate/cement interaction is a combination of adsorption and nucleation [15]. It suggests that lignosulfonate molecules (sulfonate and hydroxyl groups) are adsorbed on anhydrous cement compounds and thus, create a barrier that slows down the cement hydration [28]. The inhibition of crystal growth (nucleation theory) suggests that adsorption occurs on the hydration products and not the anhydrous ones [28]. In details, it has been stated that lignosulfonate effective functional groups are adsorbed onto C–S–H gel layer causing a change on morphology of the C–S–H phases [15]. In addition, lignosulfonate is adsorbed strongly onto hydrated C_3A compared to C–S–H [15]. Figure 5 demonstrates the effect of D2 on the hydration peak with respect to the blank cement. As can be seen, D2 has a retardation effect on cement hydration. Also, the cement setting time is increasing by changing D2 concentrations. The retardation efficiency of D2 is weak compared to R2; therefore, it is not recommended to use it as a retarder. In addition, the hydration peak maximum does not reduce that much as in R2. From Fig. 5, the hydration peak at 0.1% bwoc reaches the maximum after 1 h with respect to the plain cement. Another important example, the peak maximum at 0.7% bwoc of D2 has shifted by 6 h with respect to the blank sample. This kind of behavior is not desirable especially when it affects waiting on cement time (WOC) and thus, increasing the cost of well operations. Therefore, precautions should be taken during designing the cement slurry. The retardation behavior of D2 is mainly attributed to the

adsorption of NFS molecules to cement hydrates particles and intercalation into hydrate phases such as ettringite which inhibit the development of the hydration products [28]. Chen and Struble [29] disagree and report that NFS only intercalate the monosulfate (AF_m) phase and not the ettringite (AF_t). Figure 6 illustrates the effect of mixture additives solutions on the cement hydration peak and compares individual additives pastes with the blank one. At 0.1% each bwoc of R2 + D2 the hydration peak maximum is found after 21 h, while at 0.2% each bwoc of R2 + D2 the peak appeared after 49 h. As can be seen, dispersant addition retards the cement hydration by 13 h with respect to 0.2% bwoc R2. From this result we can conclude that both additives have synergies together to prolong the cement hydration process. As mentioned before, this phenomenon has a bad influence on well cementing operations. Therefore, the dosages of both additives are reduced to the half to reach convenient (WOC) times.

Rheology of the cement slurries

The fluidity of cement slurries is considered as one of the important factors to meet the oil-well cementing operation requirements. Therefore, studying the rheological properties of different cement/additives system is a must. Figure 7 demonstrates the impact of studied additives on cement fluidity and compares it to the neat cement slurry. The tendency observed in all additives systems enhanced the rheology of cement slurry by reducing the viscosity at constant water-to-cement ratio. For example, the blank sample viscosity is found to be 436.51 mP s at shear rate equivalent to 107.23 s^{-1} (100 r/m). In addition, at the same

Fig. 5 The heat flow during oil-well cement hydration as function of dispersant D2 dosage

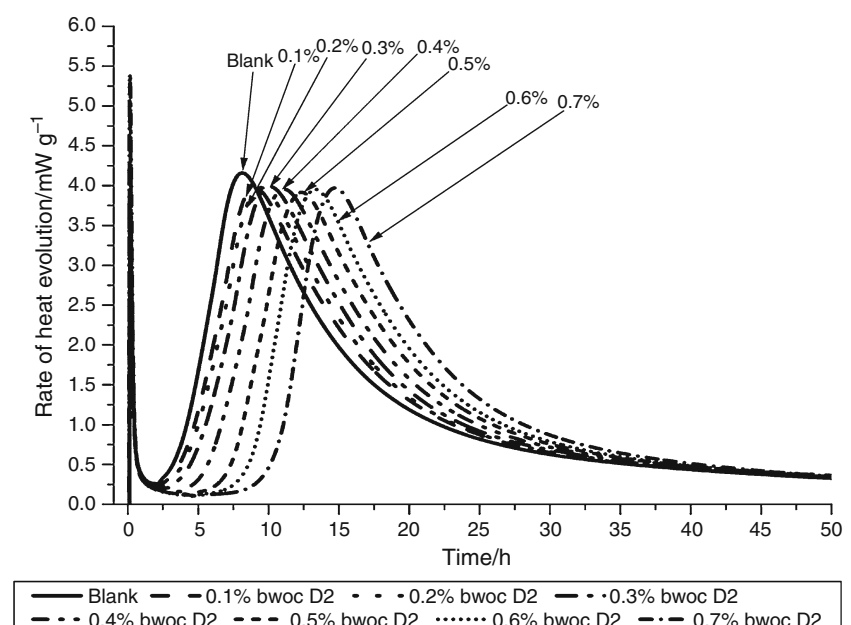


Fig. 6 The heat flow during oil-well cement hydration as function of 0.1% and 0.2% each bwoc mixture of D2 + R2

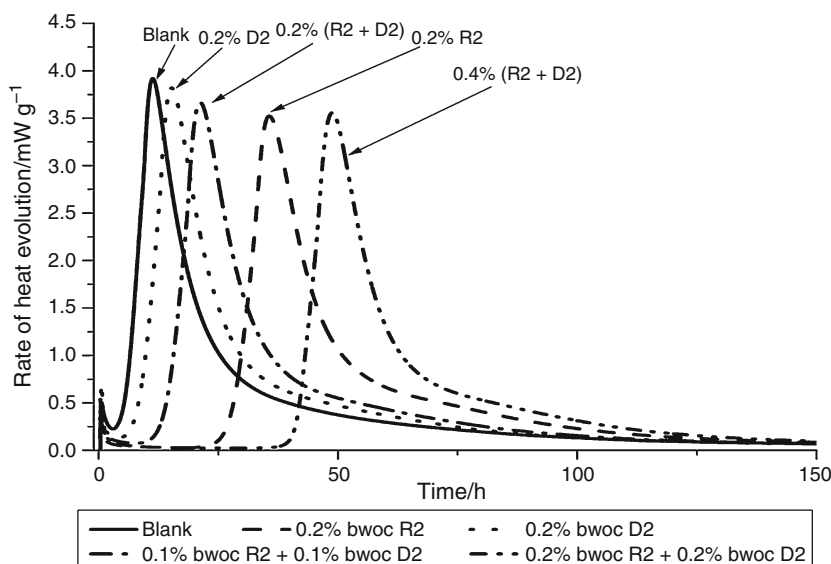
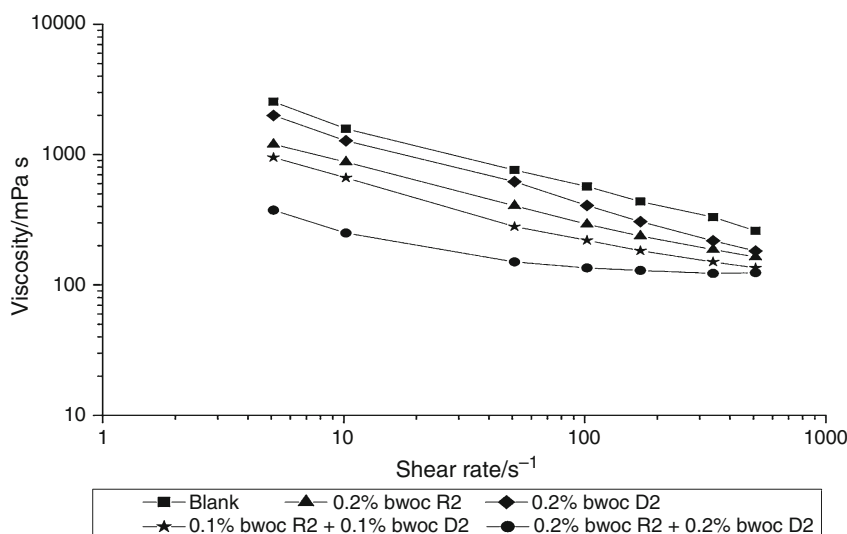


Fig. 7 Logarithmic plot of viscosity versus shear rate for cement/additives systems



shear rate the viscosities for 0.2% bwoc R2 and 0.2% bwoc D2 are measured to be 237 and 306 mPa s, respectively. According to the previous results the viscosity of the cement slurry has dropped by almost 30–45%. Moreover, R2 is found to be more effective than D2 in its dispersivity—not only at this particular shear rate (100 r/m) but also for the whole range of shear rates as shown in the figure. This is because different admixtures have different dispersion mechanisms [28]. The mechanism associated with lignosulfonates is attributed to a combination of electrostatic repulsion and steric hindrance while the dispersion mechanism of NSF is related to steric hindrance only [28]. Moreover, research studies report that the performance of NSF is varying with cement compositions and is related specifically to sulfate or alkali sulfate contents in cement [29]. Additives solution with 0.2% each bwoc of R2 + D2 shows better dispersion performance than R2 and D2

individually at the same concentration. Also, 0.1% each bwoc of R2 + D2 has the same behavior but not as effective as 0.2% bwoc mixture. For example at 60 r/m (102.14 s^{-1}) the viscosity for the blank slurry is evaluated to be 570 mPa s, while for 0.1% and 0.2% each bwoc of R2 + D2 the viscosities are equal to 220 and 135 mPa s, respectively. Moreover, the viscosities for 0.2% bwoc individual additive systems of R2 and D2 are found to be 292 and 407.5 mPa s. According to these results both additives are combined together to reach the best dispersion behavior and thus, increase the cement fluidity.

ESEM measurements

Figure 8a shows an ESEM of anhydrous oil-well cement. As can be seen, different crystals in various shapes and sizes are attributed to cement clinker main phases and other

Fig. 8 ESEM micrographs of **a** cement powder and **b** retarder R2

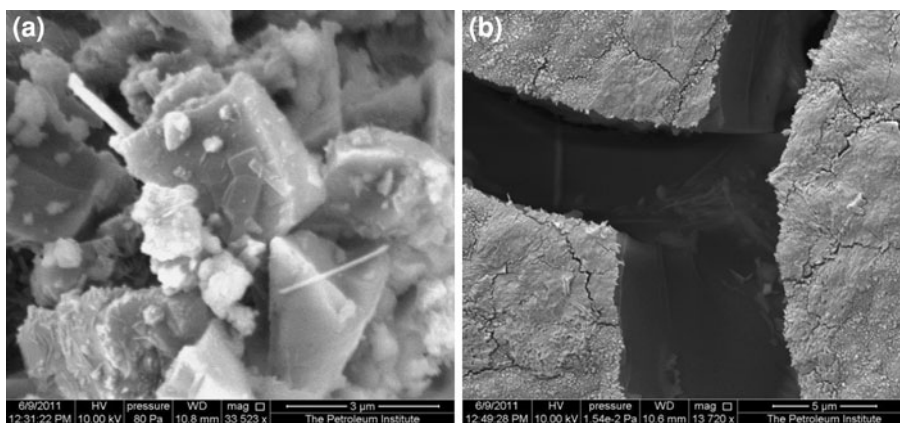
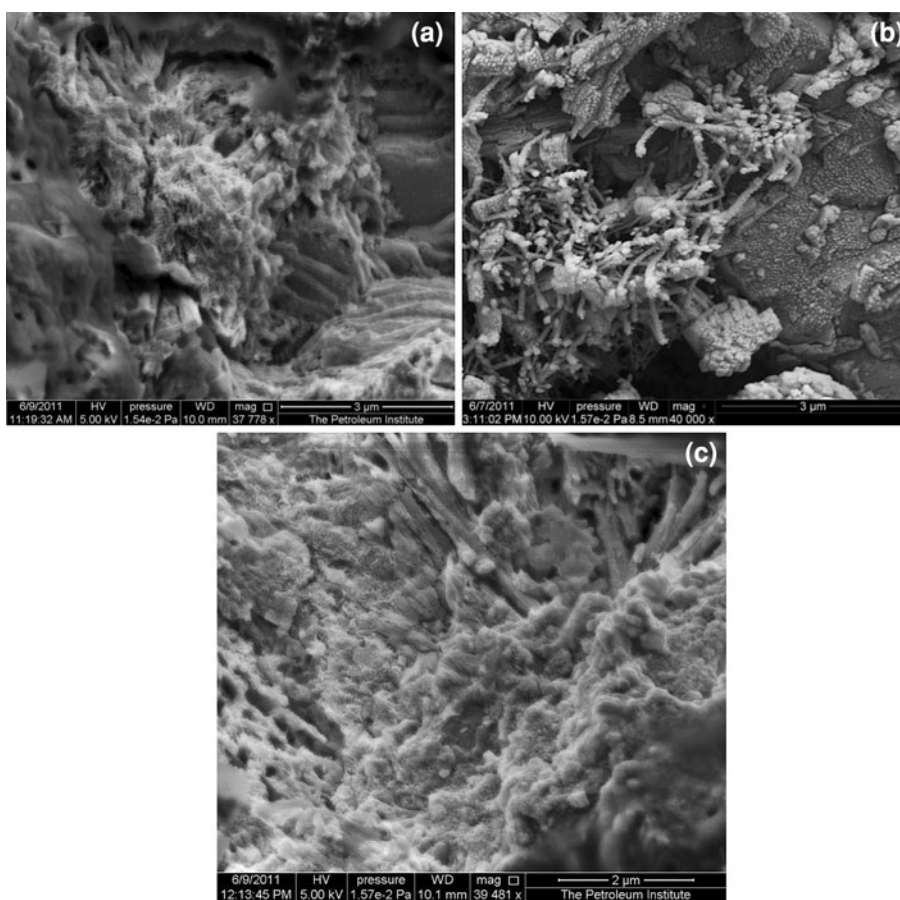


Fig. 9 ESEM micrographs of hydrated oil-well cement. **a** Blank, **b** in the presence of 0.4% bwoc R2, and **c** in the presence of 0.6% bwoc R2



components. Figure 8b demonstrates R2 solid sample. Moreover, it is noticed that the shape of lignosulfonates enables a larger surface area for better performance. Figure 9 shows exemplary an ESEM of three different hydrated oil-well cement samples. The first sample in Fig. 9a shows hydrated oil-well cement, whereas the other two samples in Fig. 9b and c are attributed to hydrated oil-well cement in the presence of 0.4% and 0.6% bwoc of R2, respectively. Compared to the morphology of blank hydrated cement, it can be seen that the crystal growth is

affected upon retarder addition, which lies in good accord to previously reported observations [15].

Conclusions

This study gives better understating of cement additives compatibility and their interactions on oil-well cement. The results show that NSF D2 has retardation effect on oil-well cement hydration which increases (WOC) time of oil-well

cementing operation. The additives solution at 0.2% each bowc of R2 + D2 has prolonged the cement hydration due to the synergistic behavior of both R2 and D2. Rheological properties of different cement additives system are investigated. The results show that lignosulfonate R2 is more effective than NSF D2 due to the difference in dispersion mechanisms of both additives. In addition, the mixtures solutions enhance the fluidity of cement slurries compared to the individual additive system. The recommended additives solution at 0.1% each bowc of R2 + D2 gives the more convenient hydration time with high pumpability performance. Therefore, these additives dosages are advised to be used in oil-well cementing. The morphology and crystal growth of hydrated cement in the presence of R2 are changed compared to the blank one which is attributed to the mechanism of cement hydration retardation.

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