Construction and Building Materials 153 (2017) 327-336

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Influence of mud cake solidification agents on thickening time of oil well cement and its solution



MIS

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HIGHLIGHTS

• MCSAs accelerated the oil well cement hydration remarkably by cutting down induction period.

MCSAs promoted the hydration reaction of tricalcium silicate and dicalcium silicate.

• The MCSAs addition affects the transformation of calcium hydroxide to tobermorite.

• HN-6 is an effective additive to solve the incompatibility of MCSAs.

ARTICLE INFO

Article history: Received 8 October 2016 Received in revised form 6 May 2017 Accepted 13 July 2017

Keywords: Oil well cement Mud cake solidification agents Hydration reaction Hydration product Calcium gluconate

ABSTRACT

The research objectives were to investigate the influence of mud cake solidification agents (MCSAs) on thickening properties of oil well cement and its solution. The results of thickening time tests showed that MCSAs accelerated the oil well cement hydration remarkably by cutting down induction period. X-ray diffraction (XRD), gas chromatography and mass spectrometry (GC–MS), thermogravimetry and differential scanning calorimetry (TG–DSC) were applied in order to study the hydrates in the mixture of MCSAs and oil well cement slurry. The results showed that MCSAs promoted the hydration reaction of tricalcium silicate and dicalcium silicate, resulting rapid and massive calcium hydroxide formation at early hydration. Besides, the addition of MCSAs affects the transformation of calcium hydroxide to tobermorite under high temperature. Quantitative and qualitative analysis of silicate anions indicated that MCSAs promoted the formation of mcSAs on thickening properties of oil well cement particles promoted by MCSAs is responsible for MCSAs on thickening properties of oil well cement. This paper also suggested that adding 1% calcium gluconate in MCSAs can solve this problem without weakening its performance.

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1. Introduction

Cementing is an essential part of well construction in the oil and gas industry [1]. And cementing operations are also one of the

most important and expensive activities done in petroleum wells [2]. The main objective of cementing operation is to provide zonal isolation of the formations which have been penetrated by the wellbore [3]. The service life of oil well mostly depends on the cementing quality and isolation ability of cement-formation interface (CFI) [4–6], because the fluid channeling occurs at CFI [7–9]. Hence, as for the oil industry, there exists significant importance of zonal isolation, service life and economy [1,3].

Today, the proposed solutions to this problem are mud to cement (MTC) [10–16] and mud cake to agglomerated cake (MTA) [17]. However, many concerns about the MTC method have risen since it was turned out that the MTC solidified body exhibited poor physical properties and had big risks of collapse after fracturing [15]. The MTA method has been proved to be effective to enhance the zonal isolation quality in oil wells in Daqing, Shengli,

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Abbreviations: MCSAs, mud cake solidification agents; MTC, mud to cement; MTA, mud cake to agglomerated cake; XRD, X-ray diffraction; GC-MS, gas chromatography (GC) and mass spectrometry (MS); TG-DSC, thermogravimetry (TG) and differential scanning calorimetry (DSC); GJE-I, first component of MCSAs; GJE-II, second component of MCSAs; API, American Petroleum Institute; C-S-H, calcium silicate hydrates; C₃S, tricalcium silicate; C₂S, dicalcium silicate; C₃A, tricalcium aluminate; C₄AF, four calcium ferrite; HN-1, hexametaphosphate; HN-2, lignin sulfonate; HN-3, sodium citrate; HN-4, sodium tartrate; HN-5, sucrose; HN-6, calcium gluconate.

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Nomenclature					
	Wb	Amount of chemically bound water	$W_{b,\infty}$	Maximum value of $w_{\rm b}$	

Henan, Qinghai and Yumen oilfields in China [18], but extremely difficult to be used in deep wells [19].

For this reason, the aqueous mud cake solidification agents (MCSAs) were developed [20], which can be used as prepad fluid in cementing operation thereby it can realize mud cake solidification without affecting the properties of drilling fluid. The results of experiments and applications show that MCSAs improves significantly the shear strength at CFI and zonal isolation of oil and gas wells by solidifying the mud cake [19,21,22]. However, the field applications encounter a failure since MCSAs is found to be incompatible with oil well cement. The feedback data on the spot shows that MCSAs accelerate cement hydration process prominently, resulting in a great risk of well cementing failure. Therefore, some efforts should be made to improve the MCSAs formula and solve the incompatibility with oil well cement without weakening its solidification performance.

The effects of additives on cement and concrete hydration have been published, but most of them deal with the influence of certain kind of additive or little amount (0–10%) of additives [23]. However, MCSAs contain several kinds of chemical agents and the addition of MCSAs in compatibility experiment is from 10% to 50%, which increase the difficulty in problem analysis. Besides, many literature sources pay much attention to crystalline hydration products using X-ray diffraction (XRD) analysis and gas chromatography and mass spectrometry (GC-MS), but little published literature discussed hydrated calcium silicate qualitatively and quantitatively due to its complex constitution and amorphous structure, resulting in obscure insight to real hydration process. Some works discuss the kinetics of cement hydration process using thermogravimetry and differential scanning calorimetry (TG-DSC) at low temperature [24,25], but in fact, the reactions could be totally different at higher temperature. The bottom temperature of oil wells is usually over 90 °C. Thus, if there are no pressurized systems, using TG-DSC to study the cement hydration seems impossible since such high temperature would lead to great water vaporization under atmospheric pressure.

This work focuses on the effects of MCSAs on cement hydration including the thickening time, hydrates and hydration degree in mixed slurries (MCSAs and pure cement slurry are mixed with certain mixing ratios) under simulated oil-gas well conditions. This paper also comes up with a solution to solve this problem without weakening its solidification performance.

2. Experimental

2.1. Materials

2.1.1. Oil well cement slurry

Class G oil well cement in our experiments was obtained from Jiahua Enterprise Company (Chongqing, China). The typical mineral composition and physical properties of class G oil well cement are given in Table 1. The typical chemical composition is given in Table 2. This cement was used in preparing all samples. The formula of pure cement slurry from well VB107 (Sichuan, China) is 600 g class G oil well cement, 210 g quartz sand, 12 g stabilizer for quartz sand, 25 g fluid loss

control additive, 5 g retarder A, 6 g retarder B, 18 g anti-gas migration additive, 4 g Dispersing agent, 4 g defoamer and 300 g water. These additives were obtained from well YB107. The density of slurry was 1.90 g/cm³.

2.1.2. Chemicals

Ethanol was purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China), which was used to stop hydration process and remove free water. Trimethylchlorosilane, hexamethyldisiloxane and dimethylformamide were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The n-tetradecane purchased from Dr. Ehrenstorfer Co., Ltd. (Germany) was used as internal standard for GC–MS analysis. Anhydrous calcium chloride from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) was used to dry organic layer. All chemicals were utilized without further purification. Hexametaphosphate (HN-1), lignin sulfonate (HN-2), sodium citrate (HN-3), sodium tartrate (HN-4), sucrose (HN-5) and calcium gluconate (HN-6) from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) were used as inhibitors to depress the influence of MCSAs on cement hydration.

2.1.3. MCSAs

MCSAs were composed of GJE-I and GJE-II [20]. GJE-I solution was a kind of latex diluted using silicate solution, and GJE-II was a kind of strong base solution. The density of GJE-I and GJE-II were 1.11 g/cm³ and 1.05 g/cm³, respectively. The pH value of GJE-I and GJE-II were 10 and 13, respectively. In the well cementing operation, MCSAs were acted as prepad fluid and 2 m³ GJE-I and 4 m³ GJE-II were pumped into well in sequence before pumping the cement slurry.

2.1.4. Sample preparation for interactions between MCSAs and cement slurry

In order to investigate the interactions between MCSAs and cement slurry, the powder samples of pure cement slurry and mixed slurries were prepared. The slurries were prepared according to the API method [29]. The mixing ratios of GJE-I, GJE-II and cement slurry were set as 1:1:8, 1:2:7 and 2:3:5 by volume. All slurries were poured into square molds ($50mm \times 50mm \times 50mm$) and then were cured in a pressurized curing chamber for 5 h and 3 days in water, respectively. Then samples were crushed and ground with carnelian mortar to pass 80-mesh. Hydration reaction was stopped with ethanol and the powders were dried for approximately 2 h.

2.2. Methods

2.2.1. Thickening time test

The thickening time of cement slurry which is related to pumpability time needs to be considered [26–28]. The pumpability or consistency of the slurry is measured in Bearden units (BC). The end of a thickening time test is defined when the cement slurry reaches a consistency of 100 Bc. However, considering the safety of cementing job in deep wells, the thickening time test is ended when the slurry achieves a consistency of 70 Bc. The slurry container which is equipped with a stationary paddle assembly is rotated at a speed of 150 r/min. Considering the temperature conditions of well YB107 in downhole, in our research, the consistency was tested at 131 °C and 140 MPa. The time it takes for cement slurry to reach bottom from surface, which was 85 min. The experimental procedure of thickening time is the API method [29].

In order to evaluate the influences of MCSAs on cement slurry hydration, MCSAs and pure cement slurry were mixed with certain mixing ratios and both thickening time of pure cement slurry and mixed slurries were measured. The mixing ratios of MCSAs and pure cement slurry are listed in Table 3, which were designed by the injection sequence and usage of GJE-I, GJE-II and pure cement slurry. All mixing ratios in this paper refer to the volume ratio of GJE-I: GJE-II: gJE-II pure cement slurry.

2.2.2. Test of shear strength at CFI

The experimental samples for shear strength measurement at CFI were prepared and tested as the procedures described in the literature [7].

Table 1

Phase composition and physical properties of class G oil well cement.

C ₃ S (wt%)	C ₂ S (wt%)	C ₃ A (wt%)	C ₄ AF (wt%)	Specific density (kg/L)	Surface area (m ² /kg)
64.1	13.56	1.38	16.62	3.18	333

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