HBRC Journal (2016) **xxx**, xxx–xxx

Housing and Building National Research Center

HBRC Journal

http://ees.elsevier.com/hbrcj

FULL LENGTH ARTICLE

Early hydration characteristics of oil well cement pastes admixed with newly prepared organic admixture

S.A. Abo-El-Enein [a], S.M.A. El-Gamal [a,*], I.A. Aiad [b], M.M. Azab [c], O.A. Mohamed [c]

[a] *Faculty of Science, Chemistry Department, Ain Shams University, Egypt*
[b] *Egyptian Petroleum Research Institute, EPRI, Cairo, Egypt*
[c] *Faculty of Science, Chemistry Department, Benha University, Benha, Egypt*

Received 4 May 2016; revised 24 August 2016; accepted 6 September 2016

KEYWORDS

Admixtures;
Oil well cement;
Setting times;
Compressive strength

Abstract A number of chemical and mineral admixtures are usually used in the oil-well cement (OWC) pastes to modify and control their fluidity to resist the higher temperatures and pressures during the drilling process of the well. In this study a newly prepared aliphatic organic compound namely cyclohexanone glyoxylate condensate (CG) was synthesized. The prepared compound (CG) was characterized using Fourier Transition Infrared spectroscopy (FT-IR) and microanalysis of carbon, hydrogen, oxygen and sulfur elemental analysis techniques. The effect of additions of 0.25, 0.50, 0.75 and 1 (mass%) of this admixture on the mechanical and early hydration characteristics of OWC pastes was studied. The phase composition for some selected hardened specimens was investigated using X-ray diffraction (XRD) and thermogravemtic analysis (DTGA) techniques. The results indicated that, addition of cyclohexanone glyoxylate condensate (CG) admixture to OWC pastes causes a slight retardation for the early rate of hydration of OWC. Addition of 0.25% of CG to OWC causes a slight improvement in the compressive strength values during nearly all ages of hydrations. XRD and DTGA results for the neat and CG admixed OWC pastes, indicate that the main hydration products are nearly amorphous calcium silicate hydrates (mainly as CSH-I and CSH-II), calcium sulfoaluminate hydrates (ettringite and monosulfate hydrate) as well as portlandite (CH).

Introduction

Oil-well cementing can certainly be known as one of the most critical operations in petroleum and gas industry among all operations performed during oil or gas well drilling. So, it becomes one of the most interesting areas of research for the last years. The productivity of an oil well was quite affected

* Corresponding author.
E-mail address: safagamal@hotmail.com (S.M.A. El-Gamal).
Peer review under responsibility of Housing and Building National Research Center.

Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.hbrcj.2016.09.001
1687-4048 © 2016 Housing and Building National Research Center. Production and hosting by Elsevier B.V.

by the cementing quality, and therefore well cementing is devoted to restrict movement of fluids between formations at different levels, support vertical and radial loads applied to casing which is placed in the drilled borehole, and protect the casing from corrosion and sealing of abnormal pressure formations [1–3]. Fully understanding of interfacial phenomena behind oil well cement procedures has scientific, economic and environmental importance [1,4]. The rheological behavior, setting and thickening times of the cement slurries must be optimized with appropriate retarders and accelerators to achieve an effective wellbore cementing operation [5]. According to the American Petroleum Institute Specification for materials and testing for well cements (API Specification 10A, 2002) nine special classes of cements were established (Class A–Class J) [6]. Also OWCs are classified into three grades based upon their C_3A contents [7]. A wide variety of cement admixtures are currently available to enhance oil well cement slurry properties, to achieve successful placement and rapid compressive strength development for adequate zonal isolation during the lifetime of the well [8]. In general, several admixtures such as retarder, dispersant, and fluid loss additive were added to oil well cement slurries [9]. The properties of fresh and hardened cement based systems are highly depended on the type and dosage of chemical admixtures used [5,10]. Recently, organic materials admixtures such as polymers are added in small quantities to modify and enhance the properties of cement products [9,11]. The effect of addition of different admixtures on the physicochemical and rheological properties of cement pastes was investigated in various studies [12–17]. Also it was found that, using several additives in cement can cause undesirable interactions [18]. Although very good recipes have been developed, problems of compatibility between cement and additives have arisen [19]. Therefore, intensive research in this area is a must [18,19]. The aim of this study was to prepare a new type of aliphatic organic admixture and investigate their effect on the early hydration characteristics and rheological properties of oil well cement pastes.

Experimental

Materials

Oil well cement (OWC) used in this investigation is moderate sulfate-resistant (MSR) Class G according to American Petroleum Institute (API) with a specific gravity of 3.15 g/cm^3, and it was supplied from Dyckerhoff AG, Wiesbaden, Germany. Its chemical and mineral compositions are given in Table 1. A new type of aliphatic organic admixture namely cyclohexanone glyoxylate condensate (CG) is synthesized.

Preparation of cyclohexanone glyoxylate

270 g of water and 244.1 g of 50% aqueous glyoxylic acid were introduced into a 1 liter reaction vessel. 123.4 g of 50% aqueous caustic soda was added while the contents of the vessel are stirred, and the pH was adjusted to 4.0. The temperature was raised to 50 °C and 98.15 g of cyclohexanone added with continued stirring. The contents of the vessel were stirred at 50 °C for a further 75 min. The solution was heated to reflux for about 3 h under constant stirring using an oil bath (120 °C) until a final viscosity of 5.52 cSt (20 wt.% solution at 20 °C) was obtained. The pH was adjusted to 10.0 by adding 39.2 g of 50% caustic soda, and the reaction mixture was cooled to 25 °C. The preparation equation of CG compound is given in Fig. 1.

Preparation of admixed OWC pastes

The hardened cement pastes were prepared from OWC using the water/cement (W/C) ratios of standard water of consistency with various CG additions of 0.25%, 0.5%, 0.75% and 1.0 wt.% of cement. The values of standard water of consistency and setting times for each paste are given in Table 2. The pastes were molded in 1 inch cubic molds, cured at 100% relative humidity up to 24 h, and then cured under tap water for of 3, 7 and 28 days. For hydration kinetics, the applied time intervals are 0.5, 1, 2, 6 h and 1, 3, 7 and 28 days.

Techniques

Characterization and experimental testing of the aliphatic organic admixture

The molecular structure of the prepared compound (CG) was identified using FTIR spectra analysis and microanalysis of carbon, hydrogen, nitrogen, oxygen and sulfur was determined.

Characterization of the hardened cement pastes

At each time interval, three cubes of each hardened cement paste were subjected to compressive strength test and the average value was recorded. This was accomplished using a Tonindustrie machine (West Germany) for maximum load of 60 tons. The resulting crushed specimens of the hardened cement pastes were ground and the hydration reaction was stopped [20]. The samples were then dried at 90 °C for 3 h in CO_2-free atmosphere.

Kinetics of hydration was studied by the determination of chemically combined water and free lime contents at different ages of hydration using the ground dried samples.

The chemically combined water content, (Wn,%), was determined from ignition loss at 1000 °C for 1 h. Duplicate measurements were carried out for each sample and the mean value was recorded. $Wn,\% = [(W_o - Wi)/Wi] \times 100$; where W_o = dried sample mass and Wi = ignited sample mass.

The free lime content, CaO (%), was determined by using the glycerol/ethanol extraction method and the mean value of the two independent determinations was recorded [20].

Table 1 Chemical and mineral compositions of oil well cement class G (MSR).

Item	SO_3	MgO	Na_2O	L.O.I	C_3S	C_2S	C_3A
Mass/%	3.0	6.0	3.0	0.75	58–48	–	0.80

L.O.I. loss on ignition.