Construction and Building Materials 144 (2017) 574–585

Contents lists available at ScienceDirect

Construction and Building Materials

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

Can nanosilica sol prevent oil well cement from strength retrogression under high temperature?

Chengwen Wang *, Xin Chen, Xiaotong Wei, Ruihe Wang

College of Petroleum Engineering, China University of Petroleum (East China), Qingdao 266580, China

HIGHLIGHTS

- Nanosilica sol (NSS) cannot improve compressive strength of cement under high temperature.
- NSS can react with $Ca(OH)_2$ and generate honeycomb $C_3S_2H_4$.
- NSS improves compressive strength of cement containing 35% SF through filling effect.

ARTICLE INFO

Article history:
Received 17 December 2016
Received in revised form 5 March 2017
Accepted 28 March 2017

Keywords:
Nanosilica
High temperature
Strength retrogression
Oil well cement

ABSTRACT

Under high temperature, the compressive strength of cement will decrease with curing time, which is called strength retrogression. It is a great challenge to prevent strength retrogression, especially for cementing deep and thermal wells. In the past, cementing crew tend to improve the compressive strength of cement under high temperature by the addition of 35%–40% silica flour (SF). Nanosilica (NS) and silica flour have the same chemical composition, i.e., SiO_2. But NS possesses smaller particle size and larger specific surface area, which can have some special properties that SF does not have. In this paper, we investigated the effect of nanosilica sol (NSS) on the oil well cement under high temperature and revealed its mechanism through experiments. The results indicate that NSS can prevent cement stone from strength retrogression under high temperature, though it is unable to improve the compressive strength. With high addition, NSS can react with $Ca(OH)_2$ (i.e., pozzolanic reaction), but the reaction product $C_3S_2H_4$ has a honeycomb and loose structure. Besides, after adding 6–8% NSS, the compressive strength of cement containing 35% SF will be further improved. This is because the nanosilica particles are able to fill in the pores of the spatial structure formed by the cement hydration products, which makes a more compact and dense structure. This study lays a foundation for the application of nanosilica and other nanomaterials as cement additives, and also provides a new perspective for high-temperature well cementing design.

1. Introduction

Nanotechnology is an emerging research field and has a potential impact on every domain of science and technology [1,2]. Nanotechnology can be defined as the science of controlling the properties at nanometer scale (lesser than 100 nm) which can make revolutionary changes in bulk material properties. The aim of the application of ultra-fine additives like nanosilica in cement and concrete is to improve its over-all properties. The main advances have been in the nanoscience of cement and concrete with an increase in the knowledge and understanding of basic phenomena in cement at the nanoscale [1,3,4].

Extensive research is going on to reveal the effect of nanomaterials on cement and concrete. So far, some nanomaterials have been studied as cement additives, such as nano-SiO_2, nano-Al_2O_3, nano-Fe_2O_3, nano-TiO_2, nano-$CaCO_3$, carbon nanotubes. And nano-SiO_2 is one of the most commonly used and researched nanomaterials [1,5–10].

Many researchers have investigated the effects of nanosilica (NS) on the rheological and mechanical properties of cement. All the reports demonstrated that NS would decrease the workability of cement slurry and thicken the slurry [7,9,11–15]. There are some controversies about the influence of NS on the mechanical property of cement stone. Most reports delivered a good opinion about the improvement of NS on cement strength. And the improvement might be mainly attributed to two reasons: as a filler to improve the microstructure; as an activator to promote the pozzolanic

* Corresponding author.
E-mail address: wangcw@upc.edu.cn (C. Wang).

http://dx.doi.org/10.1016/j.conbuildmat.2017.03.221
0950-0618/

reaction. And a few researchers still disputed the promotion of NS on the pozzolanic reaction [15–23]. Haruehansapong et al. found that the agglomeration and ineffective dispersion of too small NS particles would limit the strength improvement [21]. For the concrete, NS can make a denser interfacial transition zone (ITZ) between aggregates and bulk cement paste, which is responsible for strength enhancement [4,10,11,20,24,25]. Some researchers stated no appreciable improvement in the compressive strength of cement stone after adding nanosilica [7,13]. Based on the experimental results, Zyganitidis et al. revealed a reduction effect of NS on the mechanical property of cement stones. This resulted from the spatial structure of cement hydration destroyed by the existence of NS particles [26]. The differences of these research findings might be caused by the variety of the NS type and size and the curing condition.

Besides, NS can also affect the cement hydration. The accelerating effect of NS on cement is widely reported in the literatures [3,8,27–32]. The mechanism is mainly related to the large specific surface area of nanosilica, because it works as nucleation site for the precipitation of CSH gel. However, according to Kong et al., the acceleration may have nothing to do with the seeding effect and is highly dependent on the rapid depletion of calcium ions in the cement [28]. On contrary, Bagheri et al. found that NS could accelerate cement hydration at early ages, but reduce the cement hydration at later ages [32].

In addition, several papers have reported that NS has particular impacts on cement, which can make cement be used for other purposes. Heikal et al. demonstrated that NS particles combined with blast-furnace slag (GBFS) improved the fire resistance of cement stone up to 650 °C [33,34]. Jittabut stated that NS was able to enrich thermal conductivity and volume heat capacity of the cement significantly which could be used as thermal energy storage materials [35]. Ridha et al. found that NS would endow geopolymer cement with better acid resistance to improve wellbore integrity upon acidizing job [36]. And NS has been proved to mitigate the external sulfate attack of cement and reduce the corrosion rate of steel bars embedded in concrete, resulting in the durability improvement [37,38]. Bahadori et al. found that cement consumption could be reduced by the addition of NS particles [39].

At present, most reports concentrate on the improvement of ordinary Portland cement (OPC) properties after adding NS under normal temperature (20–80 °C). Although several papers evaluated the effect of NS on oil well cement, they focused on the accelerating effect [3,8,15,26]. Until now, there is a lack of study about the effect of nanosilica on cement that is cured under high temperatures.

With the increasing growth of petroleum requirement, the oil and gas companies are searching in new or unexplored areas. This search is getting extreme in terms of depth, temperature and pressure. High-temperature wells face special cement system design challenges. The physical and chemical behavior of oil well cement changes significantly under elevated temperatures and pressures. Under higher temperatures (above 110 °C), CSH gel is subject to metamorphism and converts to a phase called dicalcium silicate hydrate (C_2SH), which usually results in decreased compressive strength and increased permeability of the set cement [40–42]. In the petroleum literature, Swayze described this phenomenon as “strength retrogression” [42].

The strength retrogression problem can be prevented by reducing the bulk lime-to-silica ratio (C/S ratio) in the cement. To accomplish this, the oil well cement is partially replaced by ground quartz, usually as fine silica sand or silica flour. CSH gel has a variable C/S ratio, averaging about 1.5. The conversion to C_2SH can be prevented by the addition of 35% to 40% silica, reducing the C/S ratio to about 1.0. Besides, silica flour (SF) appeared to be a good pozzolanic material which can react with $Ca(OH)_2$ (CH) [41–45]. As a result, the hydration products will change and form a spatial structure which is high compressive strength and low permeability.

Chemically, nanosilica and silica flour are the same as SiO_2. So NS may be capable of regulating C/S ratio and changing the hydration products of cement like SF, which can effectively prevent the strength retrogression of cement under high temperatures. But NS possesses much smaller particle size and larger specific surface area than SF, which may affect the cement in different ways. Some researchers have compared the different influence of SF and NS on cement. Their results indicated that NS could improve the cement strength more significantly under normal curing conditions [17,19,21,24,28,32,46,47]. But no one has verified whether NS is able to prevent the strength retrogression of oil well cement under the curing condition of high temperature and high pressure (HTHP).

Therefore, in this study, we aimed to verify whether nanosilica would prevent the strength retrogression of oil well cement under high temperature through experiments and reveal its mechanism. As above mentioned, the different types and sizes will give NS various physical and chemical properties [28,30,48,49]. And we adopted the amorphous nanosilica sol (NSS) which was commonly used in petrochemical industry. The depth of deep oil wells in Western China is usually 5000–8000 m. And the bottom hole temperature and pressure are about 120–180 °C and 50–120 MPa. So we set the curing condition to 150 °C/65 MPa. Firstly, the effect of NSS on the compressive strength of oil well cement was investigated. Then, the microstructure and the composition of samples were analyzed through scanning electron microscope (SEM) and X-ray diffraction (XRD). In order to confirm the pozzolanic reaction, we determined the reaction products of NSS and $Ca(OH)_2$ through EDS. In addition, we also investigated the effect of NSS on the cement containing 35% SF under high temperature. Based on the experimental results, we hoped to reveal the effect and mechanism of nanosilica sol on oil well cement under high temperature. And this will lay a foundation for the application of nanosilica and other nanomaterials as cement additives, and also provide a new perspective for high-temperature well cementing design.

2. Experiment materials and methods

2.1. Experimental materials

The experimental materials included “Sheng Wei” brand Class-G oil well cement (produced by Shengli Huanghe Cementing Corp., China, its clinker compositions are given in Table 1), nanosilica sol (NSS, produced by Shandong Peak-tech New Material Co., China, its technical specifications are given in Table 2), silica flour (SF, 50–100 μm, produced by Shengli Huanghe Cementing Corp., China), dispersant (produced by Shengli Huanghe Cementing Corp., China), anhydrous alcohol (analytic pure, produced by Shanghai Branch of China Pharmaceutical Group).

2.2. Experimental methods

2.2.1. Slurry preparation

Nanosilica sol, silica flour and other additives were added to the Class-G oil well cement slurry at a certain mass fraction by weight of cement (% BWOC). The cement slurries without and with silica flour were prepared with a water-to-cement ratio (W/C) of 0.44 and 0.38, respectively, by following the API recommended practice 10B-2 standards “Recommended practice for testing well cements” [50].

2.2.2. Determination of compressive strength

The compressive strength of set cement was tested after the prepared cement slurry being poured into a copper mold ($50 \times 50 \times 50\ mm^3$) and cured in a house-made high-temperature and high-pressure curing pot under condition of 150 °C/65 MPa. After the set curing time, the cement stone was taken out from curing pot. The compressive strength of cement stone was determined by a pressure measuring instrument (WEW-300B, Shandong Huace Mechanical Equipment Corp., China). In order to get more accurate data, each test was repeated three times, and the average value of compressive strength was calculated.