



Nanosilica effects on composition and silicate polymerization in hardened cement paste cured under high temperature and pressure



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ABSTRACT

Cement pastes of water to cement ratio (w/c) of 0.45 with and without nanosilica are hydrated under two conditions, room condition (20 °C with 0.1 MPa pressure) and an oil well condition (80 °C with 10 MPa pressure) for 7 days. For the cement pastes with nanosilica, 1% and 3% of cements weights were replaced by nanosilica. The composition of the hardened cement pastes is investigated using X-ray diffraction (XRD). Nuclear magnetic resonance (NMR) experiments are used to quantify the silicate polymerization in hydrated cement paste. Microstructural phases are identified according to the corresponding mechanical property using nanoindentation. The results showed that under room curing conditions, hardened cement paste with 1% nanosilica has the highest level of calcium silicate hydrate (C–S–H) polymerization. However, under high temperature and pressure curing conditions, hardened cement paste with 3% nanosilica has the highest level of C–S–H polymerization. A new relatively stiff microstructural phase is observed in cement pastes incorporating nanosilica and cured under elevated pressure and temperature conditions. The significance of curing conditions and nanosilica content on the polymerization and stiffness of hydrated cement pastes are discussed.

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1. Introduction

The 2010 fracture of oil wells in the Gulf of Mexico and the associated environmental impact with the loss of billions of oil barrels necessitates investigation to produce reliable and durable cementing materials for oil well operations. Producing sustainable oil wells is a major concern for countries with large oil production like the Kingdom of Saudi Arabia (KSA). KSA has 267 billion barrels of proven oil reserves with 100 major oil and gas fields and more than 1500 active oil wells. Many of these reserves are relatively deep and maintain high temperatures with elevated pressure.

In oil and gas well drilling operations, the cementing of well casing is typically performed by incorporating oil well cement (OWC) slurry to aid in the drilling process. The composite system formed is vital for precluding a host of undesirable processes which can severely impact the production process. Stringent performance requirements are therefore placed on the cement slurries used, including low viscosity, rapid strength development, low permeability and enhanced resistance to aggressive fluids. A number

of recent research investigations have been conducted to develop high performance OWC [1–3], however, only limited results are available. It is important to note that OWC is very similar in composition to Type I, Type II and Type I/II ordinary Portland cements (OPC) while incorporating limited amount of fluidity admixtures [4].

The major cement hydration product that makes up approximately 67% of hydrated OPC paste is calcium–silicate–hydrate (C–S–H) [5]. It is suggested that C–S–H, which consists of nano-structured colloidal layers, can be altered by incorporating nanoparticles such as nanosilica and nanoalumina. Recent studies have shown that a great potential exists to improve the mechanical characteristics of cement paste by incorporating nanosilica, nanoalumina and carbon nanotubes [6–8]. Developments in nanotechnology over the past few years have paved several avenues for development of new materials that can alter properties of cement mortar and concrete. Early efforts in nanotechnology were directed to understand the fundamental phenomena of cement hydration and cement degradation mechanisms [9]. Studies in nano-modified cements, self-healing cement and other nano-based cementitious materials have been in progress for the last decade [10]. It has been shown by several researchers that traditional cement based

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materials showed radically enhanced properties when engineered at the nanoscale [11]. Moreover, nanoparticles such as TiO_2 , ZnO_2 , fullerenes, carbon nanotubes, silica, alumina, magnesium, calcium and clays have been examined to alter strength, stiffness and ductility characteristics of cementitious materials [8,11].

In this study, a basic science investigation was conducted with the objective of understanding the significance of elevated temperature and pressure curing conditions on the hydration of OPC in the presence of nanosilica. OPC cement pastes with water to cement ratio (w/c) 0.45 without and with nanosilica contents of 1% and 3% are hydrated for 7 days under two conditions: room condition of 20 °C and 0.1 MPa pressure and an elevated condition of 80 °C with 10 MPa pressure. Type II OPC was used for its resemblance to OWC composition specifically with respect to C–S–H hydration. Using ^{29}Si magic angle spinning (MAS) nuclear magnetic resonance (NMR), the silicate polymerization of hydrated cement is investigated. The microstructural compounds in the hardened cement pastes are also investigated by X-ray diffraction (XRD) analysis. The mechanical characteristics of the microstructural phases in the hardened cement pastes represented by the modulus of elasticity are investigated using nanoindentation. The results shed light on the significance of curing conditions on the hydration products.

2. Methods

2.1. X-ray diffraction (XRD)

XRD is a well-known methodology in cement chemistry [12] and is used to detect various compounds in a cement paste. XRD is usually used to characterize crystalline materials. Using XRD spectra, several compounds in hydrated cement paste such as tricalcium silicate (C_3S), dicalcium silicate (C_2S), ettringite (Aft), calcium hydroxide (CH) and calcium silicate hydrate (C–S–H), can be detected. There exists a wealth of information in the literature on XRD observations of cement hydration products [12–14].

2.2. ^{29}Si magic angle spinning nuclear magnetic resonance (^{29}Si MAS NMR)

Silicate polymerization represents the number of bonds generated by the silicate tetrahedron. The description of a silicate tetra-

hedron with various sharing oxygen atoms is shown in Fig. 1. A silicate tetrahedron having the number of n sharing oxygen atoms is expressed as Q^n where n is the number sharing oxygen atoms ranging from zero to four. As described in Fig. 1, Q^0 is observed due to the remaining C_3S and C_2S in hydrated cement while Q^1 (end-chain group), Q^2 and Q^3 (middle-chain group) in silicate are typically detected due to the layered structure of C–S–H. Q^4 is the polymerization of the quartz and can be observed in silica rich products such as fly ash and silica fume. The intensity of the silicate Q connections can be investigated using ^{29}Si MAS NMR technique. Using the intensity fractions of Q^n 's, the degree of hydration D_h of a hydrated cement paste can be calculated as [15]

$$D_h = I_Q^1 + I_Q^2 + I_Q^3 \quad (1)$$

where I_Q^n represents the corresponding intensity of Q^n connection respectively. If a silica rich material is used for a hydrated cement paste, the pozzolanic activity coefficient D_{pa} is calculated by comparing the intensity fraction of Q^4 at time t_0 and t as [1]

$$D_{pa} = \frac{I_Q^4(t) - I_Q^4(t_0)}{I_Q^4(t_0)} \quad (2)$$

The average degree of C–S–H connectivity D_c , which can represent the silicate polymerization, in a hydrated cement paste is also calculated as [1]

$$D_c = \frac{I_Q^1 + 2I_Q^2 + 3I_Q^3}{I_Q^1 + I_Q^2 + I_Q^3} \quad (3)$$

High values of D_c represent high polymerization of C–S–H. From the extensive studies of the structure of C–S–H by ^{29}Si MAS NMR, it was shown that the polymerization of C–S–H depends on its compositional C/S ratio (high polymerization for low C/S ratio) and the humidity in interlayer water (high polymerization for low interlayer water) [15,16].

The relative quantity of sharing oxygen atoms of a silicate tetrahedron in a silicate material can be detected by ^{29}Si MAS NMR and this information is used to interpret the polymerization of a silicate tetrahedron. The typical chemical shift peaks for Q^0 , Q^1 , Q^2 , Q^3 and Q^4 appeared in hydrated cement are near -74 , -80 , -84 , -95 and -110 (pozzolanic silica), respectively [1,17]. As 'the next nearest neighbor' of a silicate tetrahedron can affect chemical shift, the chemical shifts for silicate tetrahedral are varied. The ^{29}Si chemical

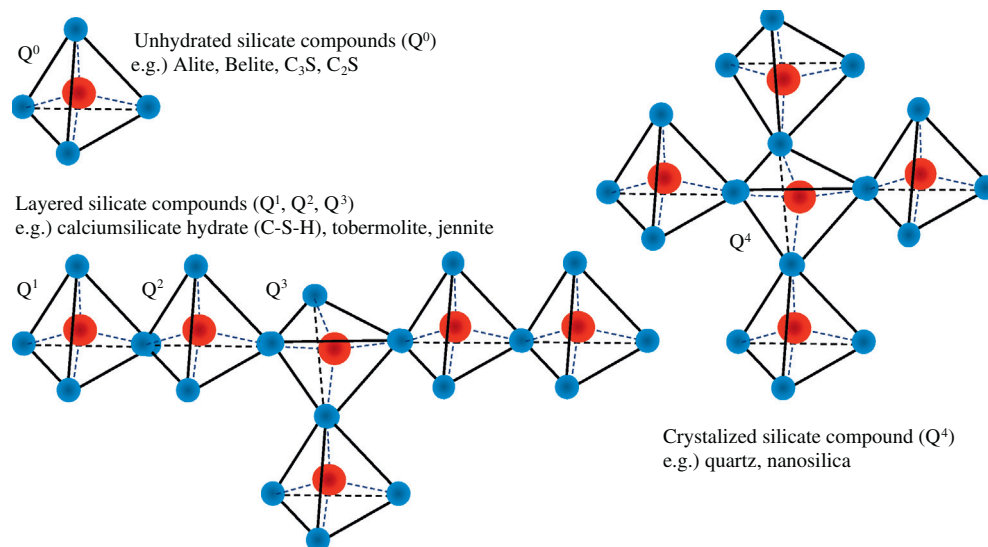


Fig. 1. Silicate connections.

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