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# Cement degradation in CO<sub>2</sub> storage sites: a review on potential applications of nanomaterials

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## Abstract

Carbon capture and sequestration (CCS) has been employed to reduce global warming, which is one of the critical environmental issues gained the attention of scientific and industrial communities worldwide. Once implemented successfully, CCS can store at least 5 billion tons of CO<sub>2</sub> per year as an effective and technologically safe method. However, there have been a few issues raised in recent years, indicating the potential leakages paths created during and after injection. One of the major issues might be the chemical interaction of supercritical CO<sub>2</sub> with the cement, which may lead to the partial or total loss of the cement sheath. There have been many approaches presented to improve the physical and mechanical properties of the cement against CO<sub>2</sub> attack such as changing the water-to-cement ratio, employing pozzolanic materials, and considering non-Portland cements. However, a limited success has been reported to the application of these approaches once implemented in a real-field condition. To date, only a few studies reported the application of nanoparticles as sophisticated additives which can reinforce oil well cements. This paper provides a review on the possible application of nanomaterials in the cement industry where physical and mechanical characteristics of the cement can be modified to have a better resistance against corrosive environments such as CO<sub>2</sub> storage sites. The results obtained indicated that adding 0.5 wt% of Carbon NanoTubes (CNTs) and NanoGlass Flakes (NGFs) can reinforce the thermal stability and coating characteristics of the cement which are required to increase the chance of survival in a CO<sub>2</sub> sequestered site. Nanosilica can also be a good choice and added to the cement by as much as 3.0 wt% to improve pozzolanic reactivity and thermal stability as per the reports of recent studies.

**Keywords** CO<sub>2</sub> storage sites · Cement design · Carbonation · Nano materials · Degradation

## Introduction

Carbon dioxide (CO<sub>2</sub>) sequestration is the technology developed in the past decades to reduce the amount of greenhouse gases increasingly released into the atmosphere. In this technique, which is also known as Carbon Capture and Storage (CCS), CO<sub>2</sub> as a dominant greenhouse gas is captured in industrial sites and injected into deep geological formations for thousands of years (Gaurina-Medimurec et al. 2010). The CCS technology has been initiated 20 years ago when a million metric tons per year of CO<sub>2</sub> was injected into an aquifer beneath the North Sea (Benson and Cole 2008). Canada and

Algeria followed the footsteps and sequestered over 20 million tons (Mt) of CO<sub>2</sub> in their deep geological sites ever since (Benson and Cole 2008; Takase et al. 2010). According to the International Energy Agency (IEA), the CCS can help to reduce more than 13% of cumulative greenhouse gas emission by 2050 and restrict the global increase of temperature. By 2015, there were 15 large-scale facilities around the world capturing 27 million tones (Mt) of CO<sub>2</sub> every year (International Energy Agency 2015). This would be a significant contribution into the reduction of global greenhouse gas emissions needed to restrain climate change.

Coal beds, saline aquifers, and depleted oil reservoirs are often chosen for CO<sub>2</sub> storage purposes, among which depleted reservoirs are the best options due to their geological history, integrity and infrastructures. Abundant and closed wells of these reservoirs are the best conduits to inject CO<sub>2</sub>, where the injection interval is cemented to avoid leakages. However, chemical degradations and mechanical failures induced due to the reaction with supercritical (Sc)

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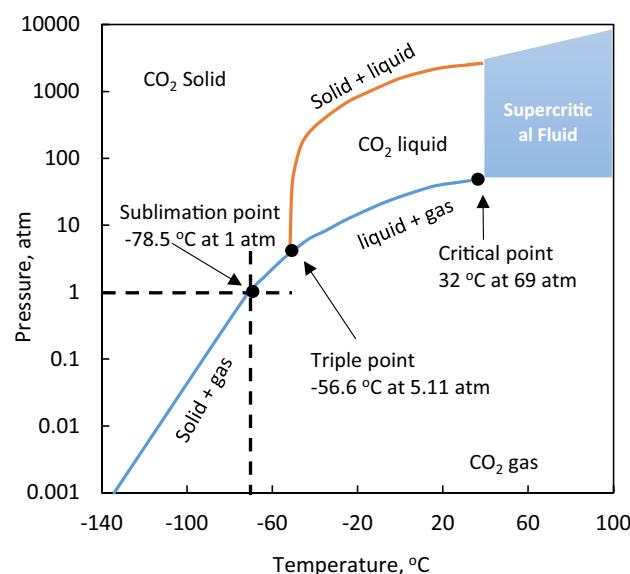
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$\text{CO}_2$  and carbonic acid under reservoir conditions may create leakage paths in the cement, causing seepage of  $\text{CO}_2$  to the surface and other valuable subsurface resources (Xu et al. 2007; Bachu and Bennion 2009; Zhang et al. 2013; Ansarizadeh et al. 2015). In fact, the class G cement, which is commonly used in the primary or secondary cementing stages, is very vulnerable and may lose its durability once exposed to supercritical (sc)  $\text{CO}_2$ . As a result, its mechanical and physical properties such as compressive strength, permeability, and porosity might change unfavorably, resulting in leakage of  $\text{CO}_2$  from the storage sites (Zhang et al. 2013; Ansarizadeh et al. 2015).

There have been several methods proposed in recent years to improve the mechanical and transfer characteristics of the cement used for sequestration practices. Changing the water-to-cement ratio, applying pozzolanic materials and employing non-Portland cement are the most important approaches proposed so far with limited success once tested under reservoirs conditions (Abid et al. 2015). Employing nanoparticles might be another approach that worth consideration as a solution for reinforcing the cement. This is mainly because chemical and mechanical properties of materials are changed when their particle size is reduced. It is also known that Calcium–Silicate–Hydrate (C–S–H), which is one of the main components required to resist against the  $\text{CO}_2$  attack, is widely available in a high order structure at the nanoscale (Abid et al. 2015; Arina and Irawan 2010). The benefits of using nanoparticles, such as nanosilica ( $\text{SiO}_2$ ), nanoalumina ( $\text{Al}_2\text{O}_3$ ), clay nanocomposites, nanotitanium oxide ( $\text{TiO}_2$ ), carbon nanotubes (CNTs), and nanoglass flake (NGFs) have been widely presented in the building and polymer industries (Abid et al. 2015; Jahangir and Kazemi 2014; Lee 2012; Ghadami et al. 2014). However, there have been a very limited discussion on their potential applications in the oil and gas industries, especially for  $\text{CO}_2$  sequestration sites. The aim of this paper is to provide a review of the characteristics of different nanoparticles and evaluate their potential applications in the cement used for the storage sites. This may shed some lights as to how the physical and mechanical characteristics of the cement can be improved for a safer storage of  $\text{CO}_2$ .

## Background

In this section, attempts are made to provide a better insight into the interactions taking place in storage sites between the cement and  $\text{CO}_2$ . The importance of  $\text{CO}_2$  sequestration as well as physical and chemical phenomena involved are discussed and approaches developed so far to resist the  $\text{CO}_2$  attack are presented.



**Fig. 1**  $\text{CO}_2$  phase diagram [modified after (Oldenburg 2007; Waseem Arshad et al. 2017)]

## CO<sub>2</sub> sequestration in deep geological formations

$\text{CO}_2$  storage sites are often referred to as deep geological formations with a storage capacity of 675–900 billion tons (Ansarizadeh et al. 2015; Benson and Cole 2008). It is a common practice for the oil and gas industry to inject  $\text{CO}_2$  into particular deep (more than 800 m) geological formations (reservoirs) to increase the petroleum production, which is also known as Enhanced Oil Recovery (EOR). When these reservoirs are completely depleted and verified as a safe geological storage sites,  $\text{CO}_2$  sequestration is considered as part of the CCS technology in the field (Gaurina-Medimurec et al. 2010).

Once injected into the storage sites,  $\text{CO}_2$  must be monitored carefully as it appears in different phases under diverse temperature and pressure conditions (see Fig. 1). For instance, at the ambient temperature,  $\text{CO}_2$  appears as a gas, but it becomes a supercritical fluid under the temperature of 32 °C and the pressure of 7 MPa (IPCC 2005; Oldenburg 2007), which often happens at the depth of greater than 800 m.

## Cement systems for $\text{CO}_2$ sequestration

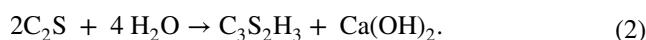
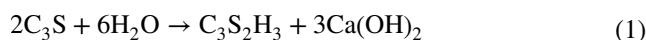
Secondary cementing is done to seal off the wells used to inject  $\text{CO}_2$  in the reservoir. A good well cement should have an appropriate thickening time, a good rheology, a low water

**Table 1** Major components of Portland cement (modified from Adams and Charrier 1985)

Compound	Chemical composition	Cement chemist notation	Concentration (wt %)	Purpose
Tricalcium silicate	$(\text{CaO})_3 \cdot \text{SiO}_2$	$\text{C}_3\text{S}$	55–65	Enhances the strength and develops early strength
Dicalcium silicate	$(\text{CaO})_2 \cdot \text{SiO}_2$	$\text{C}_2\text{S}$	15–25	Hydrates slowly, strength generated over extended period of time
Tricalcium aluminate	$(\text{CaO})_3 \cdot \text{Al}_2\text{O}_3$	$\text{C}_3\text{A}$	8–14	Promotes rapid hydration, affects thickening time and initial setting of the cement, and makes the cement vulnerable to sulfate attack
Tetracalcium aluminoferrite	$(\text{CaO})_4 \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$	$\text{C}_3\text{AF}$	8–12	Responsible for slow hydration

loss efficiency, and no free water bleeding (Lesti et al. 2013). There are eight classes of cements listed in the American Petroleum Institute (API)<sup>1</sup> standard, categorized based on their specifications and functionality. Among all classes, Class G (Portland Cement) is the most common one. In fact, Portland cement is commonly used on many occasions due to its accessibility and adaptability to different subsurface conditions. This type of cement consists of four main components, including Tricalcium Silicate ( $\text{C}_3\text{S}$ ), Dicalcium Silicate ( $\text{C}_2\text{S}$ ), Tricalcium Aluminate ( $\text{C}_3\text{A}$ ), and Tetracalcium Aluminoferrite ( $\text{C}_3\text{AF}$ ), which give certain functionality to the cement such as enhancing the strength or changing the hydration rate. Table 1 gives a summary of the functionality provided by the cement components.

Tricalcium silicate ( $\text{C}_3\text{S}$ ) is the most abundant component of the cement, hydrating faster than others (Nelson 1990). When water is mixed with the cement, hydration takes place and the compressive strength develops, which expressed as (MacLaren and White 2003; Omosebi et al. 2016):

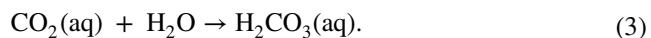


Hydration of aluminate, tetracalcium aluminoferrite ( $\text{C}_3\text{AF}$ ), is similar to tricalcium aluminate ( $\text{C}_3\text{A}$ ), which forms ettringite when it reacts with gypsum. As a result, the production of calcium–silicate–hydrate ( $\text{C}-\text{S}-\text{H}$ ) is more than  $\text{Ca}(\text{OH})_2$  due to the abundance of  $\text{C}_3\text{S}$ . This calcium–silicate–hydrate is a very important component acting as the binder of the cement.

## Portland cement degradation: carbonation and bicarbonation

To understand the chemical reactions taking place between supercritical carbon dioxide ( $\text{scCO}_2$ ) and Portland cement, Kutchko et al. (2007) carried out an experimental study to simulate a real reservoir condition (i.e., the temperature of 50 °C and the pressure of 30.3 MPa with a pH of 12.3). They indicated that the cement degradation is linked to the structural transformation of  $\text{C}-\text{S}-\text{H}$ , carbonation of portlandite ( $\text{Ca}(\text{OH})_2$ ) and the leaching of calcium carbonate ( $\text{CaCO}_3$ ).

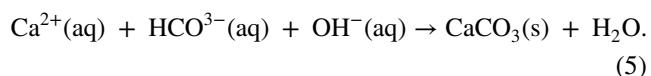
In fact, when  $\text{CO}_2$  is injected into a storage site, it dissolves into the brine, which is often left in the reservoir after production, and forms carbonic acid ( $\text{H}_2\text{CO}_3$ ) as expressed in Eq. (3). This leads to a significant reduction of pH:



As carbonic acidic diffuses into the hydrated cement, Portlandite is attacked, as expressed in Eq. (4), at a very fast rate due to its higher reactivity (Omosebi et al. 2016). This interaction brings an equilibrium to the solution:

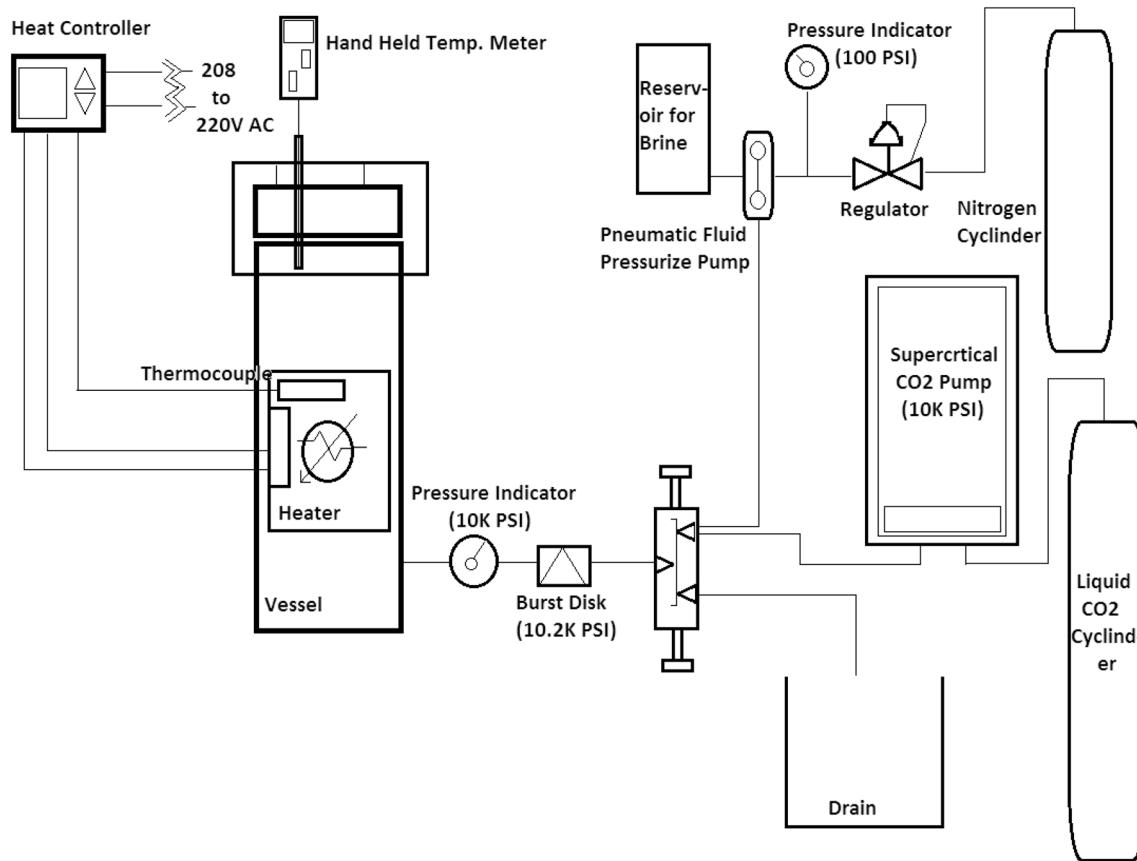


However, due to the consummation of Portlandite and leaching of  $\text{Ca}^{2+}$  out of the cement matrix, the porosity of the cement increases and calcium carbonate ( $\text{CaCO}_3$ ) is precipitated, as addressed by Eq. (5). Under these circumstances,  $\text{CaCO}_3$  acts like a filler and occupies the pore space of the cement, causing a significant reduction in porosity. In fact, formation of  $\text{CaCO}_3$  not only decreases the porosity and permeability by densification of the cementitious matrix, but also increases the compressive strength. This process, which is known as carbonation, is thermodynamically favored and cannot be avoided (Santra and Sweatman 2011):



Although carbonation improves the cement resistance to  $\text{CO}_2$  attack, the crystallization of  $\text{CaCO}_3$  would lead to cracking and volume expansion (Abid et al. 2015). This

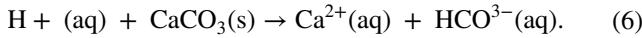
<sup>1</sup> API standard is a practice that is an accepted worldwide standard which is followed by most of the oil and natural gas companies.



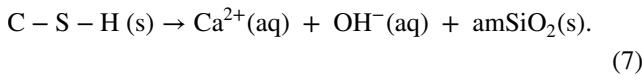
**Fig. 2** Schematic view of the static reactor designed and installed at Curtin University Malaysia

would provide a route for  $\text{CO}_2$  to migrate easily from the storage site.

When portlandite is completely consumed,  $\text{CaCO}_3$  starts to dissolve due to its continuous reaction with fresh carbonic water, which leads to further leaching of  $\text{Ca}^{2+}$  and domination of  $\text{HCO}_3^-$ , as expressed by Eq. (6). This dissolution of  $\text{CaCO}_3$  is called bicarbonation:



Without  $\text{CaCO}_3$ , the remaining C-S-H, which acts as the binding component in the hydrated cement, is converted into amorphous silica gel ( $\text{amSiO}_2$ ), as expressed by Eq. (7). As a result, the amount of  $\text{Ca}^{2+}$  gradually increases and more pores are created within the cement matrix, which leads to the loss of zonal isolation and migration of  $\text{CO}_2$  to the surface and subsurface resources (Kutchko et al. 2007; Zhang and Talman 2014):



## Neat cement degradation

### Experimental conditions

Many of the laboratory experiments carried out to evaluate cement integrity in a  $\text{CO}_2$  rich environment were done using an HPHT vessel. Half of this pressure vessel is often filled with brine (salt water) to have brine saturated  $\text{CO}_2$  (carbonic acid), while the upper half contains only sc $\text{CO}_2$  which is known as wet  $\text{CO}_2$  (Barlet-Gouedard et al. 2009). This configuration allows to simulate a storage site and initiate carbonation of the cement in the presence of  $\text{CO}_2$ . Figure 2 shows a schematic view of the pressure vessel installed at Curtin University, Malaysia.

The period of the experiment may vary from a month to a year during which the samples are constantly monitored to evaluate the carbonation rate. To achieve the best result, the carbonation tests under these circumstances are carried out under the static condition where the amount of brine/fresh water initially added to the vessel is maintained (Kutchko et al. 2008). It should be noted that employing fresh water rather than brine would accelerate the degradation of the

**Table 2** Developed approaches to improve cement resistance in CO<sub>2</sub> sequestration sites (modified after Abid et al. 2015)

Approaches	Result
Using Pozzolanic material, e.g. fly ash, silica fume, bentonite	Reducing the permeability and quantity of the portlandite. As permeability decreases, the ingress of CO <sup>3-</sup> and carbonation slows down (Kutchko et al. 2008, Duguid and Scherer 2010) Reducing the water content in the cement and decreasing the Ca/Si ratio, causing the creation of a longer chain of C-S-H, which increases the strength of the cement An excessive amount of pozzolanic materials may result in a poor strength development (Kutchko et al. 2008, Laudet et al. 2011)
Decreasing water/cement ratio	Increasing the unhydrated cement clinker which eventually decreases the permeability Increasing the density which may increase the fracture possibilities May result in creating fractures in heavy weight cement (Brandl et al. 2010)
Using non-portland cements	It is not sufficient for a long-term wellbore integrity and not generally recommended because of its accessibility (Bai et al. 2015) The hydration products are resistant to CO <sub>2</sub> (Takase et al. 2010)
Using special additives	Latex, for example, improves the bonding strength and controls the filtration loss. It allows a good strength development (Duguid and Scherer 2010). The resistance against CO <sub>2</sub> attack will, however, not be significantly improved due to a low quantity of CaCO <sub>3</sub> present Epoxy resins will chemically coat the cement but it was degraded when tested at 90 °C and 28 MPa for 31 days (Brandl et al. 2010)
Using nanomaterials	Nanosilica, for example, improves the microstructure and the strength of the cement by decreasing the porosity and permeability of mortar (Zhang et al. 2014) Polymer/clay nanocomposites increase the tensile strength (Barlet-Gouédard et al. 2007) Nanoiron enhances the compressive strength (Barlet-Gouédard et al. 2012)

cement due to the faster rate of CO<sub>2</sub> getting dissolved in fresh water.

## Experimental studies

Experimental studies carried out to evaluate cement degradation are often conducted by an HPHT chamber (vessel) to simulate reservoir conditions. These studies are categorized into two classes: (1) cements are exposed to scCO<sub>2</sub> saturated brine and (2) cement samples are solely tested against scCO<sub>2</sub>, which is also known as wet scCO<sub>2</sub> (Kutchko et al. 2007, 2008; Barlet-Gouédard et al. 2009; Arina and Irawan 2010; Duguid and Scherer 2010; Laudet et al. 2011). Having done such tests, Barlet-Gouédard et al. (Barlet-Gouédard et al. 2009) suggested that brine should be used for the carbonation test rather than fresh water. They used a 0.4M NaCl brine solution and observed a dramatic fall in the propagation rate of the cement samples after 2 days of exposure to CO<sub>2</sub>-saturated brine. To investigate the effect of temperature and pressure on the cement degradation, Arina and Irawan (2010) conducted an experiment by preparing the neat Class G cement according to the API recommended practice. They cured the cement slurry for 8 h at different temperatures (40 and 120 °C) and pressures (10.5 and 14.0 MPa) where it was found that the HPHT condition reduces the compressive strength, causing densification of C-S-H, which would increase the rate of CO<sub>2</sub> penetration. Barlet-Gouédard et al. (2009) found out that the degradation of the cement is faster under high-temperature (90 °C) and high-pressure (20.68 MPa) conditions. They indicated that

the cement morphology is modified by temperature under these circumstances. Laudet et al. (2011) carried out a carbonation test using neat Class G cements exposed to scCO<sub>2</sub> for about 90 days, at the pressure of 8 MPa and two different temperatures of 90 and 140 °C. A faster carbonation front was found at 140 °C due to the mineralogical nature of the hydrates which reduces the cement's transport properties and ultimately limits the carbonation process. They emphasized that the wellbore temperature and pressure should be monitored before the cement design.

To further understand the behavior of Portland cement under sequestered environments, Kutchko et al. (2007, 2008) carried out a series of experiments in which neat class H cement samples were exposed to scCO<sub>2</sub> under the reservoir condition (i.e., the pressure of 30.3 MPa and the temperature of 50 °C). They observed that the cement resistance depends mainly on the curing environment. In fact, the cement cured under HPHT conditions for 28 days had the least amount of CO<sub>2</sub> penetration due to the formation of calcite. This study was further investigated by Kutchko et al. (2008) in which the period of carbonation test was extended from 9 days to 1 year. The results obtained indicated that the carbonation reaction is a diffusion controlled phenomenon for samples exposed to the scCO<sub>2</sub> environment. Duguid and Scherer (2010) did a series of experiments to study the relationship between the cement degradation and pH variation. There was no degradation in the samples exposed to scCO<sub>2</sub> having a leaching solution of pH 5. Hence, they concluded that if CaCO<sub>3</sub> can be dissolved into the formation water, the degradation would stop. This could be the reason

why sandstone reservoirs have a greater carbonation front compared to carbonates. However, this experiment was conducted under dynamic conditions and it may not be a true representative of reservoir conditions.

Knowing that cement degradation taking place under CO<sub>2</sub>-rich environments, a number of attempts were made in the past decade to resolve this issue by providing different approaches, which are discussed in the next section.

## Developed approaches to improve cement resistance

As per the discussion provided, the carbonation of Portland cement is unavoidable and, hence, a few methodologies were proposed to enhance the cement resistance as reported in Table 2. Looking at Table 2, it seems that nanomaterials have gained the attention of many researchers in the past few years considering their remarkable functionality and proven applications in the construction and building-related applications. It is undeniable that other supplementary materials can improve the overall performance of the cement if properly selected/used. However, the improvement achieved by nanomaterials used so far is significantly higher than the other materials, mainly because of their large surface areas, fast interactions, and favorable pozzolanic components. In the next section, different nanomaterials which were already used or may have applications to improve the efficiency of cements and concretes are presented together with their advantageous and shortcomings. This may provide a deeper insight into the potential applications of these nanomaterials in the oil well cementing.

## Nanomaterials

Modification of cement-based materials using nanoparticles is currently recognized as an active research area for the construction and civil industry. In this technique, nanosize ( $< 10^{-9}$ ) additives are included in the mixing procedure to improve the desired properties of the cement and concrete (Lee 2012). This is mainly because nanoatoms can much easily attached to the surface of each particle and increase the surface area-to-volume ratio, which potentially increases the mechanical strength and reduces the porosity of concretes. Moreover, adding nanoparticles promotes the hydration process at the early stages due to the large surface area of particles (Zhang and Li 2011; Choolaei et al. 2012; Meng et al. 2012). Besides, cement is composed mainly of nano/microsize crystals and amorphous calcium-silicate-hydrate (C-S-H). As mentioned earlier, C-S-H acts as the binder

of the cement, which is one of the key components governing the cement's durability. Hence, the nanosized C-S-H particles with an average size of 5–10 nm can significantly reduce the porosity and permeability of the cement (Sobolev 2015). Sanchez and Sobolev (2010) highlighted the following advantageous of adding nanoparticles to the cement:

- Well-dispersed nanoparticles can help to suspend the cement grains and aggregate by increasing the viscosity of the liquid phase. At the same time, they can improve the segregation resistance and workability of the system;
- Nanoparticles fill the voids between the cement grains, ceasing the movement of “free” water;
- Well-dispersed nanoparticles can accelerate the hydration by acting as the centers of the crystallization of cement hydrates;
- Nanoparticles favor the formation of small-sized crystals and small-sized uniform clusters of C-S-H;
- Nanoparticles enhance the structure of the aggregates' contact zone, resulting in a better bond between aggregates and the cement paste;
- Nanoparticles can provide crack arrest and interlocking effects between the slip planes, which improves the toughness, shear, tensile, and flexural strength of the cement-based materials;
- The tremendous surface area/volume ratio of nanomaterials alters the chemical reactions of hydrating cements and enhances their mechanical strength.

However, it should be noted that the nature of nanoparticles, their composition, and dosage may cause some unfavorable changes in the matrix of the cement. As such, cautions must be taken to ensure that the nanoparticles chosen for oil well cementing does not pose any negative impact on the physical and mechanical characteristics of the cement once used. In the next section, some of the most common nanoparticles used in the cement and concrete industry are presented and their potential application for oil well cementing is discussed.

## Current state of the art

In this section, recent studies carried out to improve the physical and mechanical characteristics of Portland cement are presented. It should be noted that many of these studies were done in the civil industry and there are a limited number of research works conducted to modify of oil well cements using nanomaterials. However, since a very same type of the cement is used in the civil and construction industry, the potential benefits and disadvantageous of these nanomaterials in the primary/secondary cementing of wells can be understood.



### Nanosilica ( $\text{nanoSiO}_2$ )

Nanosilica has been used in many studies for cement-based materials as it is the cheapest oxide nanoparticles (Ershadi et al. 2011; Choolaei et al. 2012). Qing et al. (2007) found that 3% wt nanosilica can reduce the amount of calcium hydrate (portlandite), and improves the compressive and bonding strength at the early stage of hardening. Ershadi et al. (2011) conducted an oil well cementing experiment by adding nanosilica into the class G cement. The water-to-cement ratio of 0.6 was used to produce a cement slurry with a large thickening time, high porosity, and permeability and a low compressive strength. They indicated that adding nanosilica improves the rheological and mechanical properties of the cement, while the porosity and permeability decreases by 33 and 99%, respectively. This could be due to the filler characteristics of nanosilica, which can enhance the microstructure and promote further pozzolanic reactions (Ershadi et al. 2011; Choolaei et al. 2012). The results obtained from the study of Choolaei et al. (2012) also emphasized on the great increase of the compressive strength after adding different portions of nanosilica into an ordinary Portland cement. They also indicated that the porosity and permeability of the cement decreases which was subjected to the quantity of nanosilica used. They concluded that a certain quantity of nanosilica must be added to the cement to achieve the desired functionality. A very similar conclusion was made by Mendes et al. (Mendes and Hotza, Repette 2014) as they highlighted that a large amount of nanosilica would reduce the performance of the cement, while a small amount would not make any significant changes. Choolaei et al. (2012) proposed to use 1 wt% and Oltulu and Şahin (2011) suggested 2 wt %, whereas Mendes et al. (Mendes and Hotza, Repette 2014) recommended 3 wt% nanosilica for being mixed with the cement to have the best performance. Nevertheless, these portions significantly increased the viscosity of the cement slurry by preventing the separation of cement particles.

However, the observation made by Ghafoori et al. (2016) was not aligned with the previous findings. They replaced their Portland cements (Class I) with 6 wt% nanosilica/microsilica and fully submerged the cement samples in a 5 wt% chemical sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) solution for 1 year. The results obtained indicated that the microsilica-based cement expands by 0.043%, whereas the nanosilica-based cement could swell by 0.054%. It was also observed that the compressive strength of 6 wt% nanosilica-based cement (~44 MPa) is lower than that of the microsilica-based cement (~52 MPa). Their mercury intrusion porosimetry (MIP) testing showed a higher volume of pores in the nanosilica mixture. These could be linked to the agglomeration effect of the dry nanosilica during mixing. They indicated that agglomerated nanosilica failed to be a nucleation site

for densifying the cement paste. Li et al. (2006) found that water could be trapped between the agglomerated structure of nanoparticles during mixing which later became a porous zone. This indicated the fact that agglomerated nanosilica had weakened the cement paste matrix. Hence, a proper dispersion would be needed to fully harness the nanoparticles in the cement slurry.

### Nanoalumina ( $\text{nanoAl}_2\text{O}_3$ )

Several studies have been conducted to investigate the application of nanoalumina in the cement and concrete industry. Oltulu and Şahin (2011) studied the single and combined effects of nanopowders (i.e., nanoalumina and nanosilica) on the cement strength and capillary permeability. They added 0.5, 1.25, and 2.5 wt% binder amount of nanoparticles to the cement and tested the compressive strength at the early (i.e., 3 and 7 days), standard (i.e., 28 days), and late stages (i.e., 56 and 180 days), whereas the capillary permeability was only determined after 180 days. The best result for the compressive strength and the capillary permeability were observed when 1.25 wt% nanoalumina was used. They concluded that nanoalumina is a better option compared to nanosilica when it comes to the improvements of the physical and mechanical properties of the cement. They also indicated that a combination of these two nanoparticles would lead to agglomeration and reduces the overall performance of the cement mortar. Later, in a similar study, Oltulu and Şahin (2013) highlighted that 1.25 wt% single nanopowder would be good enough to maximize the compressive strength and minimize the permeability of the cement. Mendes et al. (Mendes and Hotza, Repette 2014) also pointed out that nanoalumina improves the abrasion resistance and thermal shock as well as resistance against any drastic changes in temperature. Heikal et al. (2015) partially replaced cement with 1, 2, 4, and 6 wt% nanoalumina to study their influences on the cement strength. Polycarboxylate-based superplasticizer was also used as part of this study to maintain the rheology of the cement slurry. The samples were cured for 28 days in a water bath and the results revealed that adding nanoalumina enhances the hydration of the cement by accelerating the initial and final setting times. The compressive strength was also increased for the slurry having superplasticizer. They concluded that 1 wt% nanoalumina is the optimum amount to achieve the desired properties.

To study the effect of sulfate attack on the cement, Jahan-gir and Kazemi (2014) added 0.1 kg nanosilica and 0.05 kg nanoalumina to the cement, cured the samples at the room temperature for 24 h, and exposed them to 10 wt% sulfuric acids for 3 to 28 days. Their study indicated that the compressive strength increases by 50% when nanoparticles are used. It was also found that a combination of nanoalumina

and nanosilica may result in a lesser expansion, since nanoalumina is a great gelatinous preserver.

### Nanotitanium dioxide (nanoTiO<sub>2</sub>)

Nanotitanium dioxide has been used in several studies with cement-based materials due to its functionalities, such as removal of volatile organic compounds and self-cleaning, which are commonly known as photocatalytic properties (Lee and Kurtis 2010; Chen et al. 2012). Unlike nanosilica, it is a non-reactive filler and has no pozzolanic activity (Chen et al. 2012). Zhang and Li (2011) conducted a test on the concrete and demonstrated that a small quantity of nanoTiO<sub>2</sub> may have a better performance than adding a large volume. According to them, adding 1 wt% nanotitanium dioxide would increase the compressive strength by 118%. This quantity could also reduce the porosity from 11 to 9%. They concluded that the finer the pore structure of the concrete is, the higher the resistance of the concrete would be against the chloride penetration. Senff et al. (2012) prepared cement samples with 12 wt% nanotitanium dioxide based on a water/binder weight ratio of 0.5 and did rheological and flow table measurements. They found that the torque, yield stress, and plastic viscosity of mortars increase significantly by this modification. However, changes in the mechanical properties, such as the compressive strength, were not obvious. According to the study carried out by Meng et al. (2012), where 0, 5, and 10 wt% nanoTiO<sub>2</sub> were mixed by the cement, the compressive strength decreases once 10 wt% nanotitanium dioxide were added to the cement. Similar results were shown by Perez-Nicolas et al. (Pérez-Nicolás et al. 2017) where they found that after 28 days of curing, increasing the amount of nanometrically structured TiO<sub>2</sub> in the cement decreased the compressive strength due to the increase of mixing water. Chen et al. (2012) used a similar percentage as Meng et al. (2014) and showed that the compressive strength increases at all ages. Besides, cement samples could withstand the corrosion and flame abrasion (Lee and Kurtis 2010). Mohseni et al. (2016) studied the application of nanoTiO<sub>2</sub> on rice hush ash-based cement composites. The percentage of nanoparticles used varies from 1 to 5 wt% of the binder, and the water-to-binder ratio of 0.4 was used to prepare the slurry. Improvements of the compressive strength and durability were recorded, especially for the mixture having 10 wt% rice hush ash and 5 wt% nanoTiO<sub>2</sub>. They also observed reductions in the transfer properties with the increase of nanoadditives in the chloride permeability test.

### Polymer/clay nanocomposites

Nanoclay is another nanomaterial which can be considered as a potential alternative for the modification of

cement-based materials. Hakamy and Shaikh (Hakamy et al. 2014) carried out an experiment on hemp-fabric-reinforced nanocomposites by partially substituting ordinary Portland cement (OPC) with 1, 2, and 3 wt% nanoclay. The water-to-cement ratio was considered as 0.48, and it was found that 1 wt% nanoclay would be the optimum quantity required to improve the hemp-fabric, nanomatrix adhesion, and thermal stability. It would also reduce the porosity and water absorption, and increase the flexural strength, fracture toughness, and impact strength. The reduction of transport properties was highlighted by Surendra et al. (Surendra et al. 2015), as well. According to them, nanoclay has a two-layer structure which helps to block the water molecules transport and reduce the permeability of the cement mortar, which increases the compressive strength by 12% when 1 wt% kaolinite is added. The flexural strength of the cement paste was increased when 1 wt% kaolinite was added to the cement. Baueregger (2015) studied the use of nanoclay on the early cement strength. The kaolinite was used by different portion as the nanoclay. Their results showed that nanokaolin clay could boost the early compressive and tensile strength of the cement without negatively impacting the final strength after 28 days. They also pointed out that a proper dispersion technique and an optimum size selection would be critical factors to improve the overall performance of the cement. Hakamy et al. (2015) studied cement nanocomposites reinforced with hemp-fabric and calcined nanoclay (CNC) under the NaOH treatment. They reported that 1 wt% CNC reduces the porosity and water absorption and increases the flexural strength, fracture toughness, impact strength, and thermal stability. They also stated that a significant amount of CNC being used in the cement would cause agglomeration. As such, a proper dispersion method must be considered to ensure that clay nanoparticles can reinforce the cement structure.

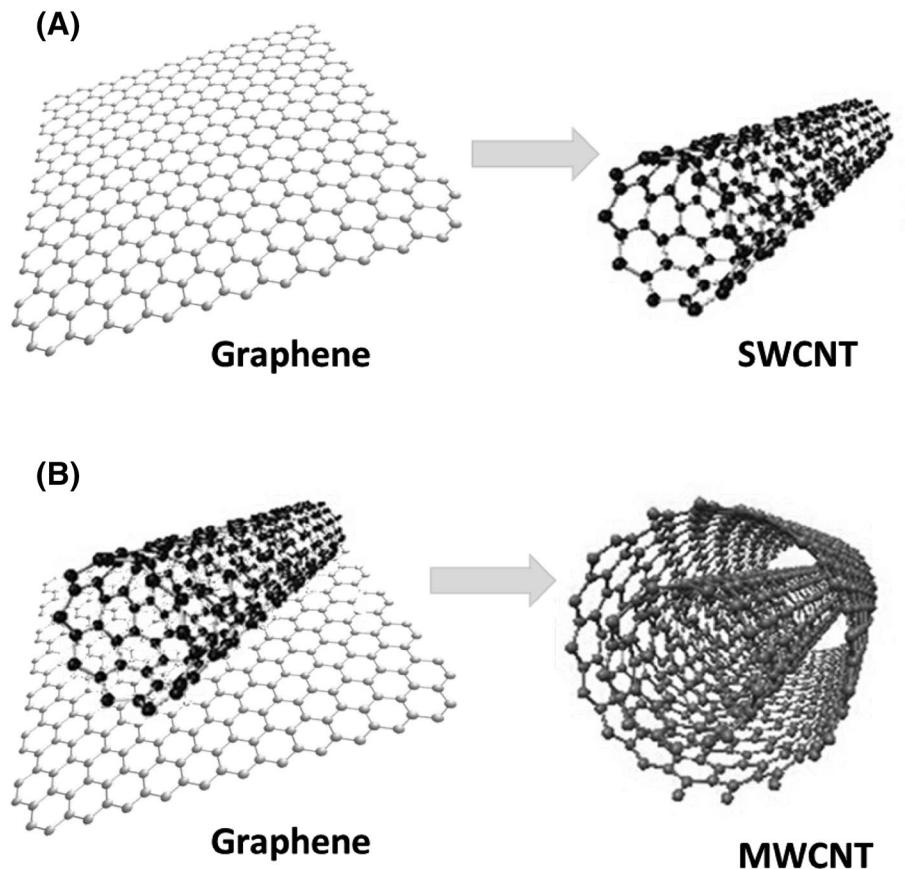
### Carbon nanotubes (CNTs)

Carbon nanotubes (CNTs) are hollow tubular channels, which are a rolled up version of the single or multiple layer graphene (Ferro et al. 2011). Their length is not restricted but often in micrometer size, while their diameters are something between 0.4 and 10 nm for a single-walled CNT (SWCNT) or from 4 to 100 nm for a multi-walled CNT (MWCNT). Figure 3 shows a schematic view of SWCNT and MWCNT.

CNTs were discovered by Iijima (1991) as the materials exhibit outstanding mechanical, thermal, and conductive properties. This ultralight weight material has been involved in different studies ranging from medicinal and constructions to buildings of structures ever since. There have also been a few studies on the application of CNTs in the oil well cementing where improvements in the compressive strength (Nasibulina et al. 2010), ductility (Abu Al-Rub et al. 2012),



**Fig. 3** **a** SWCNT and **b** MWCNT structures (<http://www.nanocarbon.cz>)

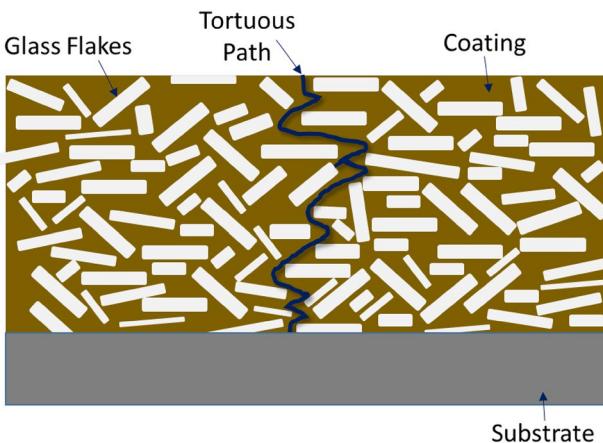


resilience (Yazdanbakhsh et al. 2009), and Young's modulus (Sáez de Ibarra et al. 2006) were reported.

For instance, Tyson (Tyson et al. 2011) indicated that CNT can increase the fracture toughness and prevent the creation of crack induced due to the expansion. It was also found that the rheology and stability of the cement slurry will not be altered if a sophisticated dispersion technique is employed (de Paula et al. 2014). Moreover, CNTs can increase the stiffness of C–S–H and decrease the porosity of the cement matrix, which ultimately reinforce the cement (Ferro et al. 2011). Rahimirad and Baghbadorani (2012) studied the use of CNT-reinforced cements in preventing gas migration, which is one of the cementing problems in gas wells. They concluded that the probability of having casing failure in oil and gas wells can be reduced, because CNTs have a high aspect ratio and, hence, would require significant energies to allow the crack propagation around a tube. However, to have an efficient CNT synthesized cement, a proper dispersion technique must be used and an optimum quantity of CNTs must be found. de Paula et al. (2014) dispersed the single-layered carbon nanotube (SWCNT) into the ground cement clinker using lignosulfonate. Although the results were promising, the scanning electron microscopic images did not show the perfect bonds between the cement matrix and SWCNT, which indicate the inefficiency

of the dispersion technique applied. Mendoza et al. (Mendoza Reales et al. 2016) indicated that addition of MWCNTs by the mass of cement up to 0.5% in an anionic surfactant can help to have a good dispersion. There was no negative impact or chemical affinity reported to the cement in that study, even at the temperature of 65 °C.

On the contrary, the study of Camacho et al. (2014) concluded that the incorporation of MWCNTs in the cement would lead to a higher corrosion rate. In their study, they considered a water-to-cement ratio of 0.5 with 0.05, 0.1, 0.25, and 0.5 wt% MWCNTs dosage to the cement. Distilled water was used for the sample preparation and the cement pastes were fabricated in 20 °C and 65% relative humidity (RH) for 28 days. Prismatic specimens were prepared for two corrosion tests: (1) chloride attack tests conducted by partially immersion of the specimens in a brine solution and (2) accelerated carbonation tests where samples were exposed to dry and wet CO<sub>2</sub>. The results obtained from both tests were similar and revealed that the increasing MWCNT dosage increases the corrosion rate. These could be explained with the depassivation of the steel surface which was due to a pH decrease induced by the cement carbonation. However, this corrosion study was based on the electrode interactions which could not be considered as an approach to directly observe the behavior of MWCNT-based cements.



**Fig. 4** Appearance of GFs in a coated substrate

#### Nanoglass flake (NGFs)

Glass flake (GF) substrates are defined as highly planar platelets with a very smooth surface. They are transparent with a transparent color tone. Nanoglass flakes (NGFs) were introduced early in 2010, having a thickness of 100–750 nm. Because of the layered structure, GFs have many advantages over other nanomaterials, such as providing a better interaction between filler and matrix, which improves the overall properties of the final cement product (see Fig. 4) (Nematollahi et al. 2010; Salehi et al. 2017).

As it is seen in Fig. 4, the laminar structure of NGFs creates a tortuous path, preventing any particles to intrude into the substrate easily. Since their introduction in the coating industry in the 1960s, GFs have been widely used in a variety of different applications due to their excellent material improvements. GFs can also improve the chemical and corrosion resistance properties of materials (Nematollahi et al. 2010; Ghadami et al. 2014). Moreover, they have been extensively used as an in-situ barrier for many industrial applications, such as external coating of high-temperature oil flow lines in Duri Oil Field, Indonesia (Watkinson 2009). Watkinson (2009) in his study on the concrete indicated that NGFs are capable of enhancing the chemical resistance and the compressive and tensile strength of materials. Considering the fact that NGFs can provide a tough impermeable barrier for steel and concrete surfaces by generating the tortuous paths, they might be a good option to improve the cement properties under the severe and abrasive conditions of CO<sub>2</sub> storage sites. According to Salehi et al. (2017), by adding 0.5 wt% GFs, a lesser amount of fillers would be required for the cement preparation, which reduces the manufacturing costs and fabricating substances in many industrial applications. However, there have not been any studies so far reporting the application of NGFs in the oil well cementing and, hence,

their behaviors under the HPHT condition of CO<sub>2</sub> sequestration conditions have not been fully understood.

## Conclusion

There have been many studies proposing solutions to improve the overall performance of the cements used in CO<sub>2</sub> sequestration sites, but none of these approaches were totally successful in resolving these issues, due, perhaps, to the severity of interactions between the cement and supercritical CO<sub>2</sub>. Nanoparticles have revealed promising results once added to the cement in various conditions, which might be due to their large surface area and reactivity. It seems that almost all types of nanomaterials can act like a filler to densify the microstructure, reduce the porosity, improve the transfer properties, and, eventually, enhance the mechanical strength. However, it appears that combination of two or more nanoparticles can lead to agglomeration and creates unfavorable changes in the cement properties. Superplasticizer, as a dispersant agent, may help to have a better and uniform dispersion of nanoparticles in the cement structure, but there is no established approach to determine the amount of nanoparticles required to have an efficient cement under different conditions.

Nanosilica might be one of the best nanoparticles for the cement probably due to its lower cost and pozzolanic activity. Nanoclay is also cheap, but it is not as much good as nanosilica due to its lesser pozzolanic activity. NanoAl<sub>2</sub>O<sub>3</sub> has a better performance compared to nanoSiO<sub>2</sub>, but it is not commonly used, perhaps, because of cost-saving purposes. NanoTiO<sub>2</sub> has no pozzolanic activity, but it has photocatalytic properties, which can help to decrease the migration of CO<sub>2</sub>. However, Nanotitanium dioxide may not be suitable for CO<sub>2</sub> sequestered sites due to its instability under high-temperature and high-pressure conditions. CNTs and NGFs can withstand harsh environments, but there are a limited number of studies reporting their applications in the oil well cementing. Thus, further studies are recommended to evaluate the application of these nanoparticles when they are mixed in the cement and exposed to CO<sub>2</sub>.

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