

The Effect of Salinity Concentrations and Curing Temperatures on Cement Class G for Carbon Storage

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Abstract

Carbon Capture and Sequestration (CCS) was recently introduced to combat global warming challenges, and one of the methods is storing supercritical carbon dioxide (CO₂) in abandoned oil wells. As a result, the integrity of the well cement is compromised by CO₂ reaction with formation water, the formation of carbonic acid as a result of cement degradation, and salinity conditions. As a result, the goal of this research was to determine the combined influence of temperature and salinity on well cement mechanical behaviour. Various salinity concentrations (0 ppm, 10,000 ppm, 20,000 ppm, and 30,000 ppm of NaCl) and curing times were used to cure sulphate resistant Class G cement samples (7 days and 30 days). The Young's modulus ranged from 1.6 GPa to 2.2 GPa. The mechanical behaviour of well cement was investigated by examining its uniaxial compressive strength and Young's modulus under these variable salinity and temperature conditions. Images of degraded samples taken using a scanning electron microscope (SEM) revealed microstructural changes induced by the degradation process. The proportion of chemical ions in the degraded cement samples was also determined using EDX (Energy Dispersive X-Ray Spectroscopy) testing. The test results demonstrated that the samples' uniaxial compressive strength initially increases up to an optimal salinity of 10,000 ppm, then steadily falls as salinity increases. The best temperature for 7 days of curing is 45°C, and the optimum temperature for 28 days of curing is 65°C, according to the compressive strength data. Overall, the resistant well cement attained its maximum strength at 60°C and 10,000 ppm NaCl concentration.

Keywords: Cement Class G; Young's Modulus; Uniaxial Compressive Strength; Salinity; Well Integrity.

1. Introduction

According to World Meteorological Organization (WMO) in May 2020, carbon dioxide (CO₂) concentration emission was recorded to breach more than 400 ppm, above the targeted 350 ppm and projected increase of about 2 ppm of the previous year [1]. Several attempts have been made to reduce or mitigate the effects of climate change caused by the gradual increase of CO₂ emissions into the atmosphere. This includes the conversion of industrial machinery to use lower-carbon fuels, limiting energy consumption, promoting renewable energy, instituting carbon tax laws, CO₂ capturing and storage [2-3]. CO₂ capture and sequestration (CCS) is a successful proven method of injecting supercritical CO₂ and storing it in concrete, abandoned hydrocarbon well, reservoir fields, saline aquifers, and/or undermined coal beds [4-5]. CO₂ injection into abandoned hydrocarbon reservoirs is considered amongst the most appropriate options of storage due to safety, proven permanent hydrocarbon traps and economical locations which are supported by infrastructure availability, low risk related integrity and well-organized operations [6]. CO₂ stored in these formations are securely trapped in the same manner hydrocarbons are held in the reservoirs for extended periods of time with little probability of leaking into neighbouring formations. Unfortunately, a variety of leaking issues from

tube and casing failure, water and sand particle production, leakage incidence in zonal isolation, ageing wells, ineffective cementing methods, and cement degradations have been linked to abandoned well reservoirs [7–9]. One of the most challenging obstacles is the ability of the wellbore cement properties to effectively accommodate the isolated zone for the entire life of the wellbore.

Cement degradation is a conventional problem in CCS and affected by several factors such as temperature, pressure and presence of salt. In addition, cement degradation or deterioration can be referred to as a change of porosity due to geochemical alteration under stationary conditions [10]. In terms of corrosion, there are several types of well completion components involved such as cement, casing, packer and other completion components located in down-hole. Typically, there are steel casing strings to be set in the middle of subsurface reservoirs with cement slurry filtrated into annulus space in between casing and formation. The existence of CO_2 in the aqueous phase, will dissolve in water to yield carbonic acid (H_2CO_3) which will react with the steel casing resulting in metal embrittlement [11]. On the other hand, the Portland cement mostly used for conventional hydrocarbon wellbore may lead to pathways for leakage (Figure 1) and reduce cement mechanical strength when exposed to supercritical CO_2 and poor cementing jobs [12].

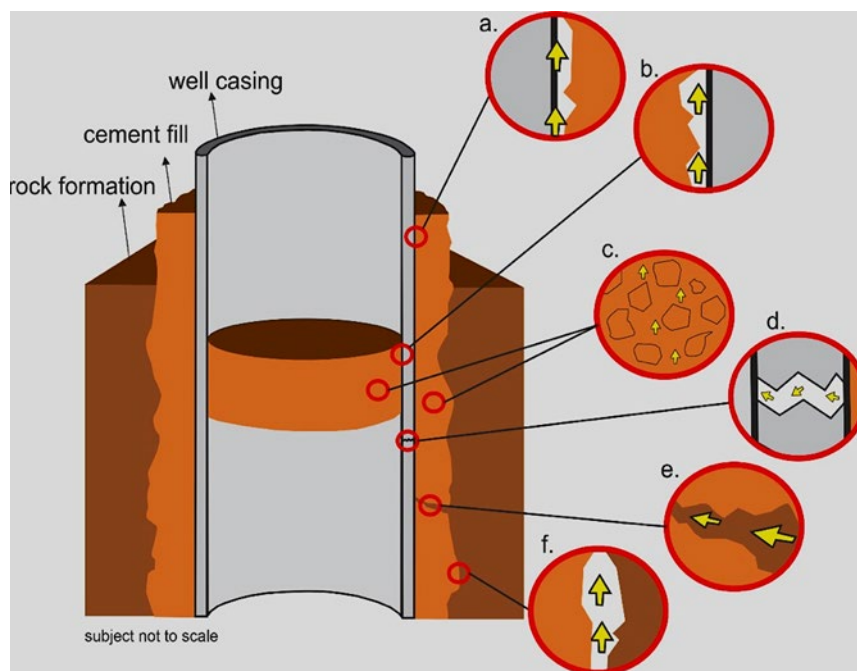


Figure 1. Schematic diagram of leakage pathways (yellow arrows) occurs in plugged abandoned reservoir well. (a) between cement and well casing, (b) between plug (cement) and well casing, (c) across cement pores, (d) across well casing, (e) fractured cement and (f) across rock and cement (modified after Randhol *et al.* [17])

This happens when carbonic acid reacts with Dicalcium Silicates (C_2S) to produce calcium hydroxide or portlandite ($\text{Ca}(\text{OH})_2$) and calcium-silicate-hydrates (C-S-H) gel which are the main composite in hydrated Portland cement [13]. Injecting supercritical CO_2 and storage in geological formation such as sedimentary basin is favorable at depths more than 800 m with geothermal gradient is $25^\circ\text{C}/\text{km}$ [14]. However, the Malay basin has a relatively high geothermal gradient of $45^\circ\text{C}/\text{km}$ [15]. Therefore, the cement for oilfield applications should withstand the minimum requirement of about 8.0 MPa (1160 psi) and 43°C because it is located at a depth of more than 800 m with average maximum salinity of 30 000 ppm [16].

The use of seawater to mix cement slurries for down-hole cementing operations is increasingly becoming significant due to the challenges of access to fresh water, which may increase expenses. For cementing operations, the presence of saline in seawater can compromise the

mechanical qualities of the cement. When exposed to different saline concentrations, the cement's strength may decrease due to porosity changes, loss of silica content and capacity to withstand high pressure and temperature [18]. As a result, the quality of the cement job is jeopardized. The American Petroleum Institute (API) recommended eight varieties of high or moderate sulphate resistant cement (class A to H) with specified specifications for hydrocarbon wellbore cementing jobs. The main objective of it being sulfate resistant is to prevent cement deterioration from water in reservoir formations. The enhancement of the API class was Portland-based in terms of composition, tolerance to high temperatures and acidic environments [19]. Most cementing jobs in the oil and gas industry use class G which is suitable for deep downhole jobs.

Prior to any CO₂ storage operation, the wellbore cement integrity needs to be assessed, and the two key parameters which affect cement integrity during curing time are temperature and salinity. Ordinarily, Young's Modulus, a materials elasticity metric, is used for compressive testing of cement integrity. A high Young's modulus suggests that a cement system would become brittle and fracture with increasing pressure and temperature, whereas a low Young's modulus suggests that the cement system will stay ductile, stiff, and intact. In a correlation study between cement compressive strength and salinity levels, Emmanuel *et al.* [20] reported that after 28 days of curing with seawater (pH: 6.5), the strength of the cement increased owing to the suppression of hydrate reaction. In another study, Gowthaman *et al.* [21] evaluated the addition of sulphates to ordinary Portland cement, which was near the API Class G cement standard. According to their findings, the Young's modulus and strength of the cement decreased with increasing salt concentration and aging time due to retardation in the hydration process. SEM Images revealed that, NaCl penetration and deposition increased substantially with increasing concentration and aging time. Kutchko *et al.* [22] observed that the solubility of Ca(OH)₂ decreased with increasing temperature during 28 days of curing Class H Portland-based cement under pressures ranging from 0.1 MPa to 30.3 MPa and temperatures ranging from 22°C to 50°C. Additionally, they observed that the curing temperature and pressure altered the mechanical properties of hydrated cement. Nasvi *et al.* [23] documented that the optimum cementing temperatures for class G should be between 50°C to 60°C and the general mechanical strength required for well cementing should be greater than 14 MPa [24]. The integrated effect of temperature and salinity concentration on cement class G integrity to fit Malaysia reservoir conditions particularly, is illusive in the literature. Therefore, the purpose of this study was to assess the mechanical properties (strength and stiffness (Young's Modulus)) of these integrated components.

2. Materials and methods

2.1 Materials

2.1.1. Saline water preparation

Saline water with different concentration (10 000 ppm, 20 000 ppm and 30 000 ppm) were prepared by adding NaCl to distilled water. This was calculated using Equation 1. The amount of NaCl in g/1 000 mL added in order to achieve the desired concentration is shown in Table 1.

$$\text{Weight of NaCl (g) in 1 000 ml (distilled water)} = \frac{1\,000\text{ ml} \times \text{ppm}}{1\,000\,000 - \text{ppm}} \quad (1)$$

Table 1. Weight of NaCl at different salinity concentrations in ppm

Salinity concentration (ppm)	Weight of NaCl (g/1000 mL)
0 (control)	-
10 000	15.23
20 000	20.41
30 000	30.93

2.1.2. Cement sample preparation

The cement class G sulfate resistance powder used was donated by Kemaman Supply Base. It had a specific gravity of 3.2 g/cc and the chemical composition obtained from the manufacturer is tabulated in Table 2. Cements slurry samples were prepared according to API recommendations practice 10B, which is 0.44:1 water to cement ratio (w/c) [25]. Cement powder was then added bit by bit into the cement container and mixed evenly for 15 seconds. Next, the cement mixer was set at a speed of 12, 000 RPM to mix the cement slurry for 35 seconds. All sides of the cube moulds were then greased prior to filling with cement slurry. The cement slurries were then poured into 150 mm x 150 mm cube moulds as shown in Figure 2 and left for 24 hours under atmospheric conditions (1 atm and 27°C). All cement samples were cured in different temperature and salinity for 7 - 30 days. A total of 80 of cement samples were prepared.

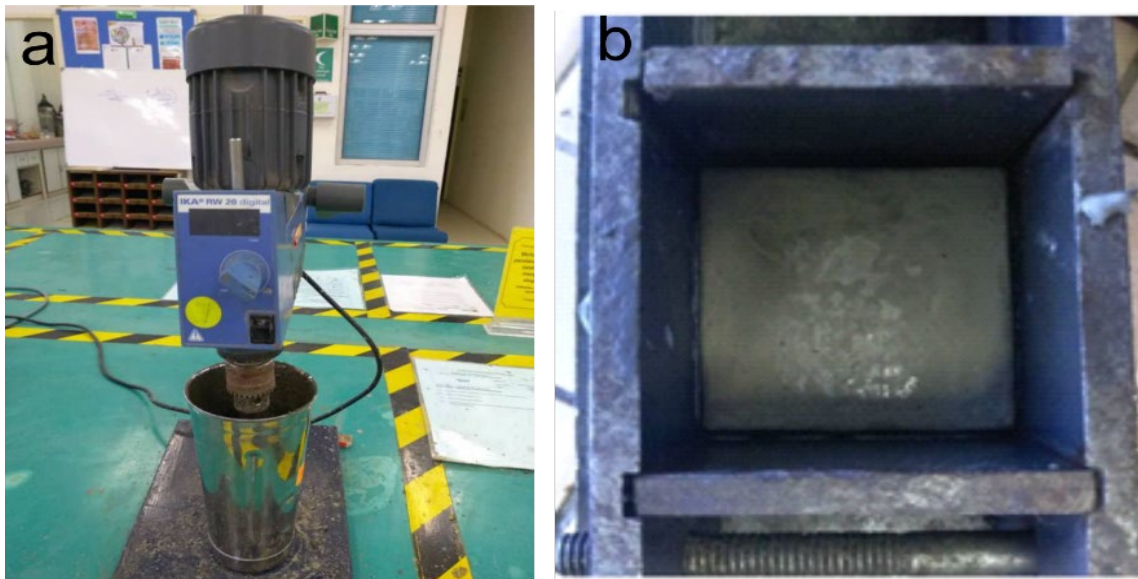


Figure 2.(a) Preparation of cement slurry using heavy duty mixer and (b) Casting cement slurry in the cube moulds

Table 2. Chemical composition of class G sulfate resistance in this study

Material	wt%	Material	wt%
Calcium oxide (CaO)	64.0	Sulfate trioxide (SO ₃)	2.7
Silica oxide (SiO ₂)	21.5	Magnesium oxide (MgO)	2.4
Aluminium oxide (Al ₂ O ₃)	3.4	Potassium oxide (K ₂ O)	0.38
Iron oxide (Fe ₂ O ₃)	4.6	Sodium oxide (Na ₂ O)	0.41

2.2 Cement integrity test

2.2.1. Uniaxial compression strength

NL Scientific uniaxial compressive strength (UCS) testing machine was used to measure the uniaxial compression strength. The cement samples were tested using a 3000 kN capacity compression and 0.6 kN/s constant loading. Once compression started, the value of compressive strength displayed on the monitor next to the machine was recorded. The test was conducted after the cement samples were cured for 7 days and 30 days in different temperatures and salinity. The Young Modulus was calculated according to Equation (2):

$$E = \frac{\sigma}{\varepsilon} \quad (2)$$

$$\varepsilon = \frac{\Delta L}{L_0} \quad (3)$$

where E is Young's modulus (GPa); σ is stress from UCS (MPa); ε is strain (ratio); ΔL is change of sample length and L_0 is original length.

2.2.2. Morphology structure analysis

The morphology structure of the cement samples was obtained using Scanning Electron Microscope (SEM) Hitachi SU8020 while X-ray diffraction (XRD) patterns were measured using a Rigaku SmartLab, Japan diffractometer. With a Cu_K- beta radiation at 40 kV and 30 mA to obtain the crystalline phase of the cement after identifying optimum salinity concentration and temperature.

3. Results and discussions

Figure 3 shows the distribution of temperature (30°C, 45°C and 65°C) against the Uniaxial Compressive Strength (UCS) for different salinity concentrations of the cement class G samples (0 ppm, 10 000 ppm, 20 000 ppm and 30 000 ppm) for curing in 7 days and 30 days. From Figure 3a, it can be seen that the variation of salinity (NaCl) concentrations had a marginal effect on the mechanical strength of cement when cured at 7 days, the mechanical strength ranged between 30 MPa to 23 MPa as temperature increased from 30 °C - 65 °C. On other hand, the compressive strength increased significantly as the temperature increased from 30°C to 45°C and remained constant as the temperature increased from 45°C to 65°C. The sample with a salt concentration of 10,000 ppm exhibited the maximum compression strength in comparison to the other samples. The increment of compressive strength is attributed to the precipitation of hydration products such as C-S-H and $\text{Ca}(\text{OH})_2$ in the alkaline environment [20]. According to the experiments, the optimum temperature and salinity concentration for 7 days curing period are 45°C and 10 000 ppm respectively.

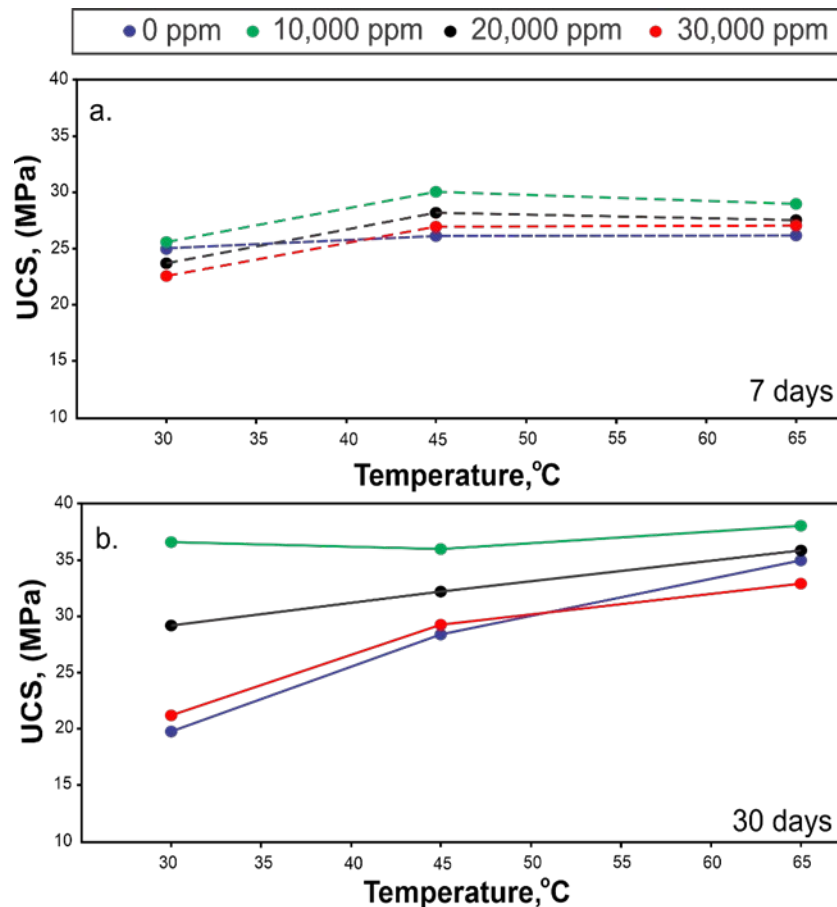


Figure 3. Distribution of UCS with different temperatures and salinity concentrations (a) curing in 7 days and (b) 30 days

Meanwhile, for Figure 3b where the cement samples were cured for 30 days, the variation of salinity (NaCl) concentrations had a substantial effect on the mechanical strength. The mechanical strength ranged between 20 MPa to 39 MPa, which are all above the recommended mechanical strength requirement of 14 MPa [24].

Salinity in NaCl concentrations has significant effects that related to mechanical strength in between 39 MPa to 20 MPa which are above mechanical strength requirement 14 MPa [24]. The findings are consistent with the recent work of Nasvi *et al.* [23], which found that the optimal temperatures for this type of cement ranged between 50°C to 60°C. For all the samples the mechanical strength increased with increasing temperature. In the presence of NaCl, the mechanical strength increased with increasing temperature due to the reaction of Ca^{2+} ions in C-S-H gel which were enough to precipitate along with Cl^- ions which enhanced the dissolution of C-S-H [21]. For 30 days curing, the optimum salinity concentrations and temperature were found to be 10 000 ppm and 65°C respectively.

The Young's modulus is a component of the elasticity modulus, also known as effective stiffness. It is the most significant for material mechanical properties, particularly in cement integrity studies. The temperature and pressure in the wellbore for CO_2 storage are significantly high in the subsurface compared to the surface where confinement occurs for the cement that laterally shrinks away from the well or expands due to strain effects. Figure 4 depicts the distribution of the Young's modulus (E) with varying temperature and salinity concentrations.

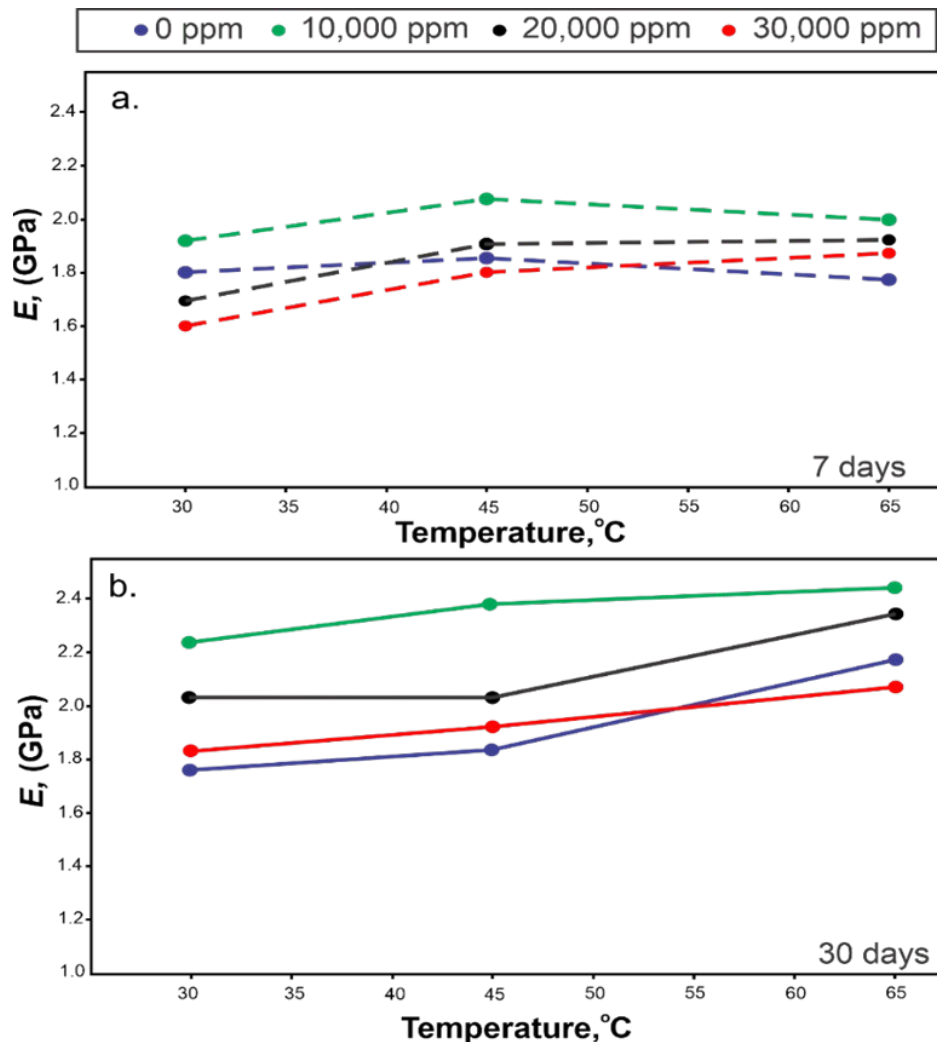


Figure 4. Young Modulus with various temperatures and salinity concentrations (a) curing in 7 days and (b) 30 days

Figure 4a shows the cement samples which were cured for 7 days. The maximum and minimum Young's modulus value were 1.6GPa and 2.2GPa for samples with 30,000 ppm and 10 000 ppm at 30°C and 45°C, respectively. With increasing temperatures, the young modulus increased sharply and then decreased moderately. The initial increment at optimum salinity of 10,000ppm was from 1.9 to 2.2GP before gradually decreasing to 2.0GPa. The effect of temperature on the young's modulus is a related to the UCS values. Higher Young's modulus values will result from higher UCS values. Accordingly, the young's modulus decreased above the optimum temperature similar to that UCS values. In other words, below the optimal temperature, the initial elasticity decreased due to delay in the hydration process as Cl^- enters the cement matrix, leaching Ca^{2+} out of the cement matrix, whereas at the optimal temperature, the elasticity increased, most likely due to reduced $\text{Ca}(\text{OH})_2$ solubility [21]. The elasticity modulus of the samples with 20 000 ppm and 30 000 ppm NaCl slightly increased due to the reaction of cement components with NaCl, in which it decreased the material strength by precipitating Ca ions. Furthermore, the C-S-H molecules get denser as the temperature rises and are unable to properly fill the capillary pore spaces. As a result, the strength reduces as the temperature rises. The Young's modulus varies in a similar way to the uniaxial compressive strength.

Figure 4b depicts 30-day curing period with Young's modulus values ranging from 1.75GPa to 2.4 GPa. The overall trend showed that elasticity increases with increasing temperature and the sample with salinity concentration of 10,000 ppm had the highest elasticity (2.4GPa) compared to the other samples. These findings are consistent with previous findings [24] which showed that low NaCl concentrations accelerate hydration whereas high NaCl concentrations slow hydration and affect the elasticity modulus. Furthermore, The range of elasticity values for 30-days curing period was found to be greater than that of 7-days curing period, consistent with the USC data obtained. the results revealed that after 30-days curing the optimum temperature of the samples increased. Therefore, curing time increased the USC, elasticity modulus and resistance to temperature.

Scanning Electron Microscope (SEM) SEM analysis was performed to determine the microstructural variation and detailed morphology structure of the cement samples. Figure 5 depicts SEM micrographs of selected optimum cement samples after 7 days and 30 days of curing at different temperatures (45°C and 65°C) and NaCl concentrations. From the SEM images, it is possible to distinguish the different microstructures at different NaCl concentrations, curing periods, and temperatures. The formation of C-S-H gel is clearly visible at 20 000 ppm NaCl for both 7-daya and 30-days curing periods at different temperatures. The images suggest that the porosity for both curing periods decreased with increasing NaCl concentrations and should have improved mechanical strength [21]. This is because C-S-H gel dissolves in the presence of NaCl, forming Portlandite ($\text{Ca}(\text{OH})_2$), which fills the available pore spaces in cement, making it less permeable and stronger [22]. However, according to our results, 10 000 ppm had the highest compressive strength compared to the other samples. The possible reason could be that the NaCl microcrystalline absorption process occurs on the surface of the fibrous C-S-H gel and acts to bridge into two C-S-H particles but does not assimilate with the C-S-H structure, as suggested by Suzuki *et al.* [26].

Figure 6 shows the XRD pattern for NaCl at 10 000 ppm with various curing times and temperatures. It can be seen that the most abundant mineral is $\text{Ca}(\text{OH})_2$, which was formed during the initial development of C-S-H, followed by dicalcium silicates (C_2S) and calcium carbonate (CaCO_3). $\text{Ca}(\text{OH})_2$ and C-S-H were hydration products of C_2S , whereas CaCO_3 appeared due to carbonization process from carbonic acid reaction [11]. The hydration of C_2S in an alkaline or saline environment can speed up the process of hydration curing time period [13]. Therefore, the increase in C-S-H is important for improving the mechanical properties and durability of cement. Furthermore, this process can absorb significant amounts of CO_2 which is important for environmental protection [13].

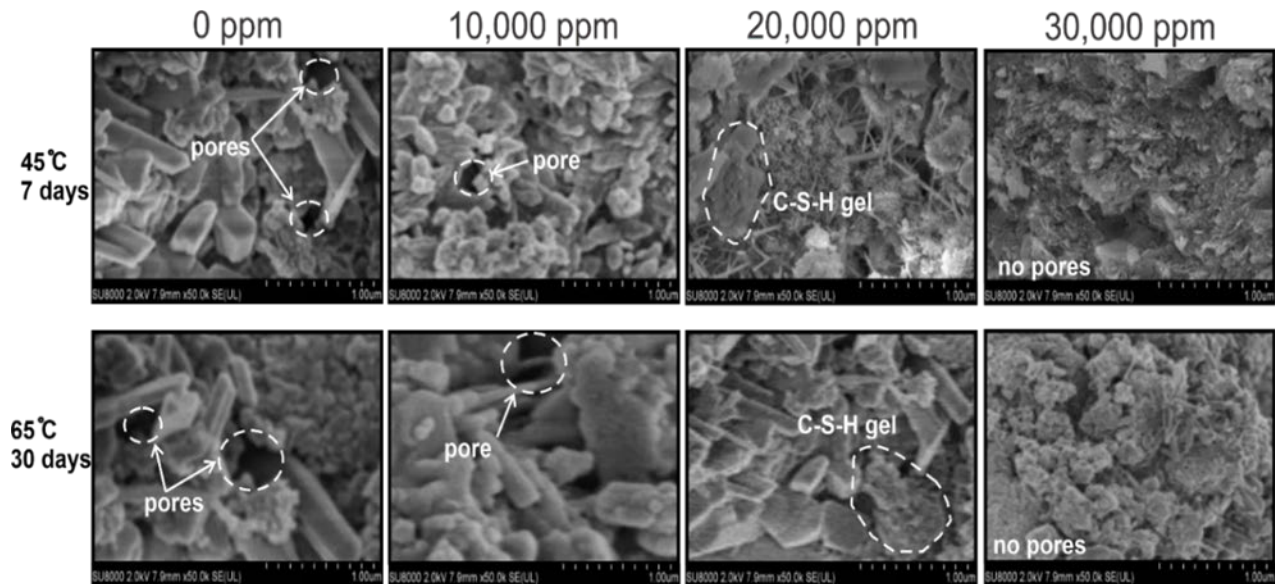


Figure 5 –SEM images of the samples at optimum temperature 45 °C (7 days) and 65 °C (30 days) with various salinity concentrations

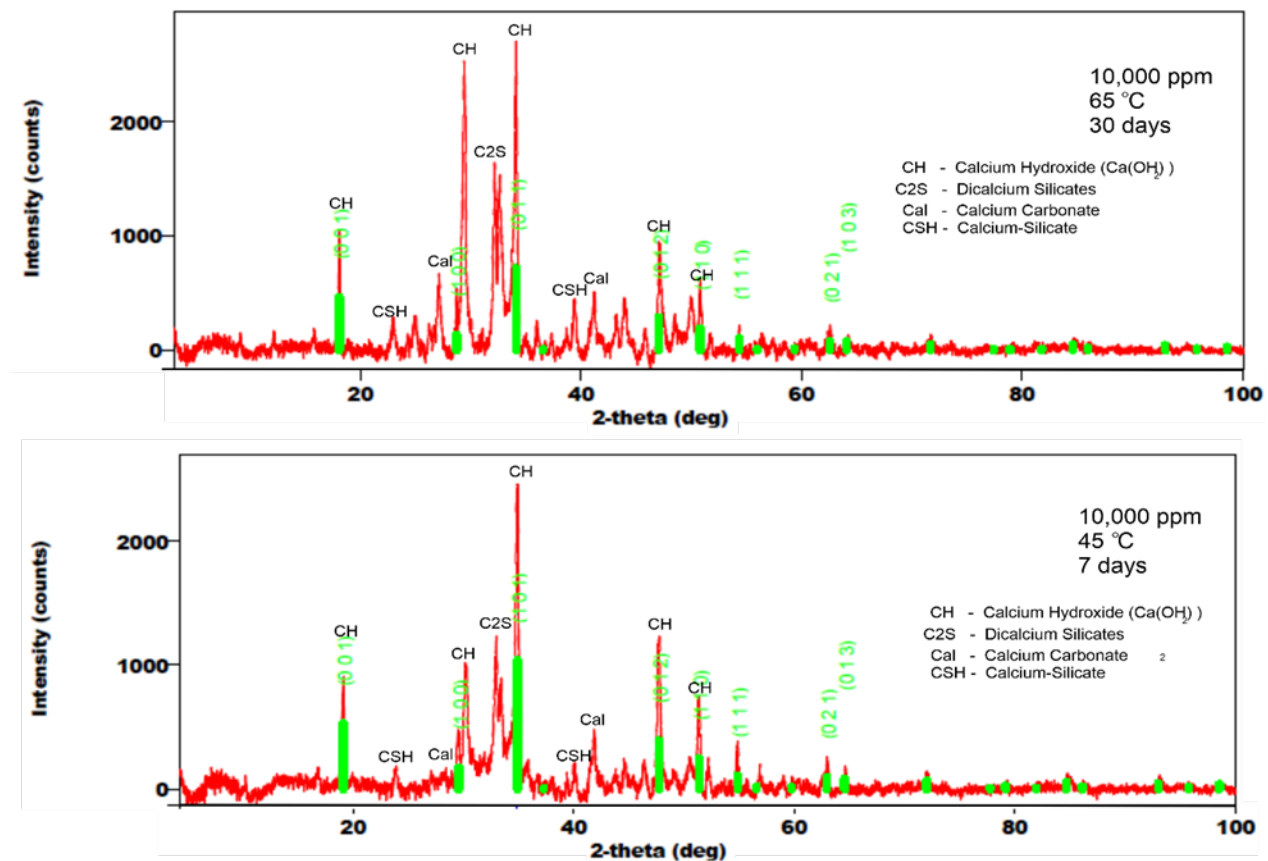


Figure 6. XRD patterns of the cements for 10 000 ppm NaCl concentration at different curing period (7 and 30 days) and temperatures (45°C and 65°C)

4. Conclusions

The integrated effect of salinity concentrations (0 ppm – 30 000 ppm) and temperatures (35°C – 65°C) befitting Malaysia reservoir conditions was used to determine the mechanical

strength, elasticity, and surface morphology of class G cement. The highest compressive strength of 39 MPa was attained using low salinity (10,000ppm) at a temperature of 65 °C after 30 days curing owing to NaCl absorption on the fibrous structure surface and retardation of hydration process. The Mechanical strength increased with increasing temperature owing to increased hydration rate and C-S-H gels bonding. All the cement samples used had a mechanical strength greater than 14 MPa, making them suitable for CO₂ storage. The elasticity modulus followed the same trends with that of UCS, the maximum elasticity (2.4GPa) was attained using low salinity (10,000ppm) at 65°C after 30 days curing. SEM images revealed the formation of C-S-H gel in the presence of NaCl, this reduced the porosity of the cement. This was also supported by XRD, which revealed that at 10 000 ppm, the gel began to form as a by-product of the hydration process.

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Nomenclature

ppm	parts per million
E	Young's Modulus
UCS	Uniaxial Compressive Strength
C-S-H	Calcium-Silicate-Hydrates
NaCl	Sodium Chloride (salt)
σ	Stress
ε	Strain
API	American Petroleum Institute
wt%	Percentage by weight

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