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EFFECT OF SALTY WATER ON PERFORMANCE OF CEMENT-BASED MATERIALS

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ABSTRACT

The use of salty water for production of cement-based materials is one of the most efficient strategies to solve freshwater shortage problems in desert areas. The purpose of this paper is to investigate the effects of salty water from oil industry on fresh and hardened properties of the cement mortar. Semi-adiabatic calorimetry was used to study changes to cement reaction due to salt amendments. Incorporating salty water reduced the workability, slightly increased the wet density and no considerable effect on the air content compared to the control. Furthermore, salty water accelerated the hydration reactions, reduced setting time and enhanced the compressive strength at early ages, but later the enhancement decreased with curing time. It was apparent that the degree of impact depended on salinity level and ionic composition of the salty water.

Keywords: Salty Water; Mortar; Hydration; Calorimetry; Compressive Strength.

I. INTRODUCTION

In the construction projects, water is the key component in cement-based materials. It is mainly needed for the hydration process, as well as for workability, strength development and durability of the resultant products. Finding water of a suitable quality for their construction work can be a challenge for contractors in arid areas, particularly in the desert. However, during oil operations, a lot of salty water is generated [1, 2].

Al wahat region in Libya contains limited sources of fresh water that can be used in construction operations, but at the same time they are surrounded by a large number of petroleum fields that produce large amounts of gas and oil associated with huge quantities of salty water which are separated upon reaching the surface using production separators. The characteristics of this salty water, also known as production water, formation water, or brine water, depend on the geological structure, the type of hydrocarbons extracted from the earth and its amount can even vary with the lifetime of the reservoir [2]. At the end point of oil and gas production, salty water rates increase and can be over 98 percent of the total fluid flow [3, 4].

Salty water disposal is generally divided into discharge and injection operations. Most onshore salty water is reinjected into injection wells for either enhanced recovery, whereas, a lack of disposal capacity has led, many countries (e.g. Libya), to use a large pits as a method of disposal.

Discharging salty water during petroleum operations can have negative environmental and socio-economic impacts. The environmental impacts on agriculture (e.g. in Libyan Oasis) can arise from salts incorporated from discharge of salty water. Accumulation of soluble salt restricts the crop growth rate by disturbing the balance of nutrients present in the soil. On the other hand, fresh water sources are also vulnerable to potential adverse impacts due to the high pH and salt content. As a result, environmental regulatory authorities have banned its discharges in most onshore or near-shore locations [4].

Proper management of salty water resulting from petroleum operations is required not only control the short term adverse environmental impacts but also to avoid any long term problems associated with salty water disposal. Reusing salty water in the construction industry is one of the possible solution.

Cement-based materials (concrete, mortar) are the most widely used building material in the world. Finding an alternative building material that meets the same criteria for durability and affordability as that of such material is challenging [5]. The quality of the mixing water plays a vital role in the preparation of cement-based materials. Chloride, sodium, magnesium and calcium ions are the main contributor to the properties of the resulted product [6].

The presence of free chloride ions in the Portland Cement (PC) pore solution can cause many adverse effects such as low ultimate strength [7]. However, chlorides (sodium chloride) have two different effects based on

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their level. At low levels (up to 4% by weight of cementitious materials) they can act as an accelerator whereas at the higher level (8%) they can act as a retarder [8, 9].

Chloride can be bound by physiosorption to the cement hydrated products [10]. When chlorides are physically immobilised, they are adsorbed onto the surface of the amorphous CSH gel [11] causing a more permeable structure due to the ability of chlorides to deflocculate colloidal CSH particles [12]. It has been reported that CSH accounted for approximately 50% of the chloride binding capacity in a typical PC paste [13]. Furthermore, chloride can be bound chemically during cement hydration by the reaction of NaCl with CH to produce a $CaCl_2$ that reacts with the aluminates to be converted into the insoluble chloride form of Friedel's salt [11, 14].

2NaCl	+	**CH**	$\longrightarrow$	**$CaCl_2$**	+	**2NaOH**
Sodium chloride	+	Calcium hydroxide	$\longrightarrow$	Calcium chloride	+	Sodium hydroxide
$CaCl_2$	+	**C_3A**	$\longrightarrow$	**$C_3A.CaCl_2.10H_2O$**		
Calcium chloride	+	Aluminate	$\longrightarrow$	Chloro-aluminate/Friedel's salt		

Cement type is the most influential factor for chloride immobilization [15]. In general, the more the aluminate (C_3A) clinker in the cement, the more chloride binding occurs [14, 16]. Normal PC can bind chloride significantly more than sulphate – resisting Portland cement (SRPC). This can be attributed to the SRPC's lower C_3A content.

Furthermore, there is an inverse relationship between pH and the stabilised chloride concentration since it has been reported that significant binding of high levels of chlorides is usually achieved at high pH values [15].

Moreover, the effect of the water/binder ratio on chloride binding has been investigated by several researchers. The conclusion in regard to this issue is somewhat controversial. Arya and Buenfeld [15] suggested that the binding of chloride would increase as the water/binder ratio increase, however, other researchers showed that the total amount of bound chloride was independent of the water to binder ratio [17].

Also curing temperature and hydration time have an effect on the binding of chloride. Arya and Buenfeld [15] cited studies that supported the fact that chloride binding increases as the curing temperature increases due to the faster reaction rates at the high temperatures. In addition, Arya and Buenfeld [15] reported that the majority of chloride binding in PC mixes occurs during the first 28 days.

Many substances are known to act as accelerators, however, calcium chloride ($CaCl_2$) is the most effective and widely used accelerator for all classes of cement [18, 19]. Thus, the suggested mechanism explored in relation to the effects of $CaCl_2$.

Juenger and Monteiro [18] reported that the more effective accelerating ions increased flocculation of colloidal particles. The CSH which formed on the surface of the particles in the case of C_3S hydration would become more flocculated in the presence of these ions. Consequently, it was proposed that the structure should contain larger pores to provide easier diffusion pathways through the CSH layer forming around the particles (Figure 1) and more rapid hydration during the early stages.

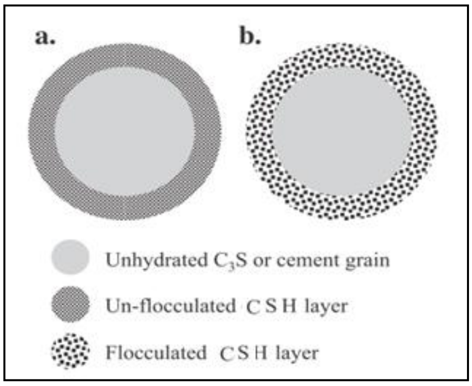

Figure 1. Effects of accelerating ions on flocculation of CSH, a) unflocculated system, b) flocculated CSH as may occur in the presence of $CaCl_2$

Overall, as noted by different works of past researchers, there are contradictory results regarding the impact of salty water on the performance of cement-based materials in terms of fresh and hardened properties (Table 1).

Table 1. Past observations on the effect of salty water on properties of cementitious materials

Salt	Concentration	Type of mix	Observation	Reference
Sea water	Na = 4130 mg/L Cl = 7620 mg/L Ca = 1900 mg/L Mg = 5420 mg/L	Cement Paste	The chloride ions in sea water increased the hydration degree. Sea water mixing enhanced the compressive strength at early ages but later the enhancement decreased	[20]
Sea water	Cl = 9344 mg/l	Concrete	The 28 days compressive strength of concrete mixed with sea water increased only by 2% as compared with portable water	[21]
Salty water	Salinity = 35000 mg/L	Concrete	The compressive strength of concrete mixed with salt water increased by 6, 11 and 7% at 7, 14 and 28 days respectively as compared with portable water	[22]
Salty water	Salinity = 875 mg/L	Concrete	Reduced 28 days compressive strength by 6%. The compressive strength increased by 33% and 54% at 28 and 90 days respectively as compared with compressive strength at 7 days. Delayed setting time by 3%.	[23]
Ocean water	NaCl = 46000 mg/L	Concrete	Reduced 28 days compressive strength by 22%. Gained compressive strength steadily up to 150 days but with values lower than the rate of the sample with fresh water.	[24]
Ocean water	Salinity = 35000 mg/L	Concrete	Reduced 28 days compressive strength by 29.4%	[25]
Sea water	NaCl = 8000 mg/L	Concrete	Increased 7 days compressive strength by 3.4% and decreased 28 days compressive strength by 7.8% as compared to tap water	[26]
NaCl	1% and 4% by weight of cementitious materials	Mortar	Higher NaCl content, lower acceleration effect. Accelerated setting time by 50% and 20% respectively	[9]
NaCl	8% by weight of cementitious materials	Mortar	Reduced 28 days compressive strength by 14%. Delayed setting time by 140%.	[9]
NaCl	2.9% by cement weight	Concrete	Increased 28 days compressive strength by 28%.	[27]
NaCl	35 grams/ liter of water	Concrete	Increased 28 days compressive strength by 6%	[28]
NaCl	45 grams/ liter of water	Concrete	The rate of the strength gain in fresh water is slow as compared with the salt water. Increased 7 days compressive strength by 5%.	[29]
NaCl	50 grams/ liter of water	Concrete	Reduced 28 days compressive strength by 13%	[30]

Salty water	Salinity = 35000 mg/L	Concrete	Increased 28 days compressive strength by 6%	[5]
Salty water	Salinity = 82500 mg/L	Concrete	The hydration rate increased with increasing salinity. Within the first week, the compressive strength increased by 53%. Improvement after 7 days was only 13%.	[31]
Production and Brackish water	Minimum chloride = 2040 mg/L	Paste, mortar and concrete	All mixes met or exceeded the requirement 28-days design compressive strength of 30 MPa.	[1]
	Maximum chloride = 44500 mg/L		Gained compressive strength up to 18 months. Decreased the initial setting times of paste mixes. Decreased concrete slump. Increased concrete wet density.	
$CaCl_2$	10% by weight of cement	Paste	Accelerated setting time by 45%	[32]
$CaCl_2$	4% by weight of cement	Concrete	Increased 7 and 28 days days compressive strength by 21.3% and 10.4% days respectively.	[33]
$CaCl_2$	0.75% by weight of cement	Paste	Accelerated cement hydration. Improved compressive strength with time as compared to control samples.	[34]
$CaCl_2$	4% by weight of cement	Concrete	Concretes with calcium chloride have higher compressive strength compared with those without calcium chloride. Increased 7 and 28 days days compressive strength by 36% and 7% days respectively.	[35]
$CaCl_2$	4% by weight of cement	Concrete block	Increased 28 days compressive strength by 42%.	[36]

The mechanism of the effect of salty water on the properties of cement-based materials have been investigated previously; however, none of the theories can explain all the observed phenomena, therefore, further research is required. The objective of this research is to understand the impact of incorporated salty water generated from petroleum processes on the properties of the resultant cement mortar. This knowledge is needed to determine the appropriate end-use of these materials.

II. EXPERIMENTAL METHODOLOGY AND DESIGN

2.1. Materials

2.1.1 Cement

Table 2: Percent chemical composition of El-Borge cement analyzed by XRD method

Parameter	Content (%)	
	El-Borge cement	ASTM C150
CaO	62.36	61 – 67
SiO_2	22.06	19 – 23
Al_2O_3	4.8	2.5 – 6.0
Fe_2O_3	3.68	6 Max

MgO	2.01	5 Max
K_2O	0.63	<1.0
SO_3	2.47	3 Max
Na_2O	0.08	<1.0

ASTM type I cement (El-Borge, Zliten (BZ) from Arab Union Contracting Company (AUCC), Libya which meets GPC requirements [37] was used to produce the mortars. General purpose cement is preferred because the observation of mortar properties can be done during the normal hydration process, hence the effect of salty water can be noticed.

2.1.2 Fine aggregates

Fine aggregates used were locally available silica sand procured from Awjilah town and called El-Borge sand with an absorption capacity of 0.20%, specific gravity of 2.60. Prior to use, the fine aggregates were dried in ambient conditions to eliminate any free water. The particle size distribution by sieving method specified in ASTM C 136 [38] is illustrated in Figure 2.

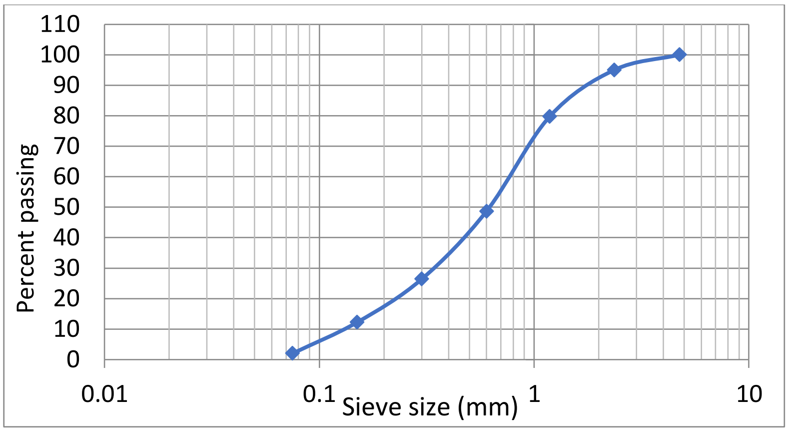

Figure 2: Particle size distributions (Sieving method) of El-Borge sand

2.1.3 Mixing water

Fresh water

Water drinking grade tap water (TW) (pH 7.4; 2.29 µS/cm) was used and conditioned at 22±2 ºC prior to use.

Salty water

A total of five water samples was obtained from different Libyan oil fields. These samples represent formation water from separators and manifolds. A total of five water samples (including tap water) was analyzed for certain impurities that could affect mortar mixes (Table 3).

Table 3: Chemical analysis results of water samples

Parameter (mg/L)	Tap water	Sample 1	Sample 2	Sample 3	Sample 4
Chloride	250	34200	67100	111000	140200
Sodium	200	20000	23600	31500	57000
Calcium	78	7100	8820	14000	23500
Magnesium	50	1050	1200	2100	5100
TDS	578	623500	100720	158600	225800

2.2 Mortar composition

The composition of the mortar was in accordance with ASTM C 270 [39] the mix proportions being 1 part of cement and 3 parts of sand (by mass) at a fixed water/cement ratio (w/c) of 0.50. Each mortar batch comprised cement (225 g), fine aggregate (675 g), water (112.5 g). For each mix, different water type was used.

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2.3 Preparing, casting and curing of test specimens

The mixing process followed the procedure described in ASTM C 305 [40] using the Hobart mixer (model N-50 G). The cement mortar specimens were cast using cubes (50 mm) from steel molds. The moulds were sealed using zip lock plastic bags to prevent water from evaporating and stored in a moist atmosphere for 24 h using a large plastic box. Demoulding take place after that and thereafter placed in a curing tank filled with water saturated with lime [41] for up to 28 days at a temperature of 22.0 ± 0.5. Water not saturated with calcium hydroxide (high-calcium hydrated lime) may affect test results due to leaching of lime from the test specimens. All laboratory work was conducted at 22 ± 2 °C.

2.4 Test Methods

F- Cal 4000 semi-adiabatic calorimeter (Calmetrix Incorporation, USA) was used for monitoring the temperature evolution of mortar mixes while the hydration reaction takes place. F- Cal 4000 is designed for up to four standard (100 mm x 200 mm) plastic cylinders with lids as sample vials. Due to heat sink effect, an extra mortar sample mass was needed to help create a robust and good thermal profile. The large sample size 2kg mortars (approximately ½ volume of a 100 mm x 200 mm cylinder) were prepared according to ASTM C270. The F-Cal's lid was left open during testing, to avoid heat transfer between samples. All cylinders were capped to avoid accidental spillage. Data generated by the F-Cal 4000 was analysed using CalCommander Calorimetry Software. Test age was in the order of 50 hours to minimize testing error (due to limiting random errors that accumulate with longer test ages) and to ensure the main hydration peak had decomposed back to the baseline and reached a steady state. The test period of not more to 3 days was recommended by Poole [42] as this time frame probably best describes cement's potential for causing thermal-stress problems and also helps to minimise testing errors due to limiting random errors that accumulate with longer test ages (up to 7 days). The reported calorimetric data was an average of triplicate samples taken from a single batch.

The fresh mortars were tested in accordance to ASTM C 1437 [43] for flow, ASTM C138 [44] for wet density, air content (TESTING Bluhm & Feuerherdt GmbH, ASTM C 231 [45] and ASTM C 807 [46]for setting times (H-3085 Humboldt Vicat Tester). Mortar specimens (50 x 50 x 50 mm) were tested at the age of 3,7 and 28 days for compressive strength. An ADR –Auto V2.0 250/25 compression testing machine from ELE International, UK was used. The compressive strength was followed the listed procedures of the test method ASTM C109/C109M [47]. Vertical load at a rate of 0.99 kN.s-1 was exerted on the specimens and the maximum load indicated by the testing machine (load at failure) has been recorded. Each value of the results presented in this report is the average of three test samples.

III. RESULTS AND DISCUSSION

3.1 Influence of salty water on flow of mortar mixes

The flow was found to decrease with increased concentration of salinity (Figure.3). A reduction in flowability was between 8 -22% compared to the control mortar.

The use of salty waters at different salinity have been reported in the literature to cause a reduction in the workability of cement-based materials [1, 2].

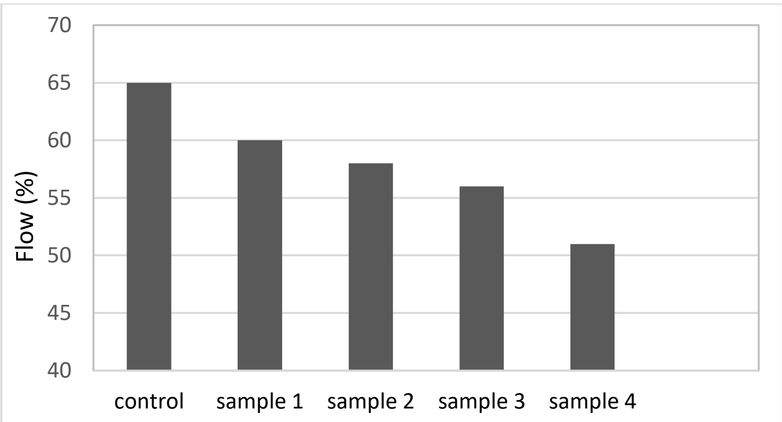

Figure 3: Effect of salty water on flow of mortar mixes

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3.2 Influence of salty water on wet density and air content of mortar mixes

For the mortar mixes of fixed w/c ratio, wet densities were noted to increase with increasing salinity compared to control mix (Figure 4). However, the percentage increments in the wet density were only between 0.5 – 1.3% among the various samples. These increases suggest that the higher the TDS contributes to higher wet densities. The increase in the wet density of cementitious materials when incorporating salty water has been noted in other studies [1, 2].

The results indicated that there is no significant relationship between salinity and air content. All mixes showed a slight reduction in the air content compared to the control mortar (Figure 5).

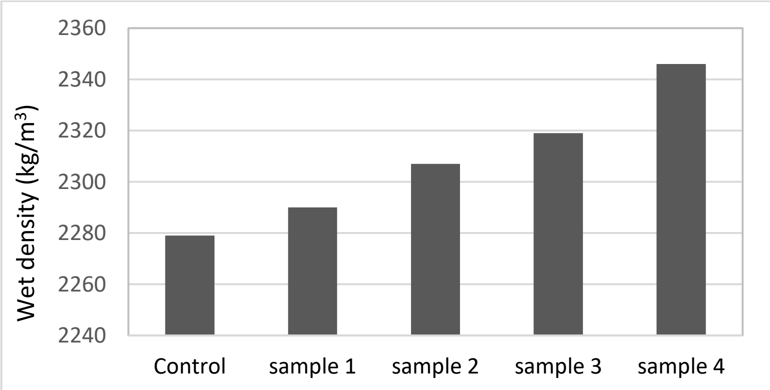

Figure 4: Effect of salty water on wet density of mortar mixes

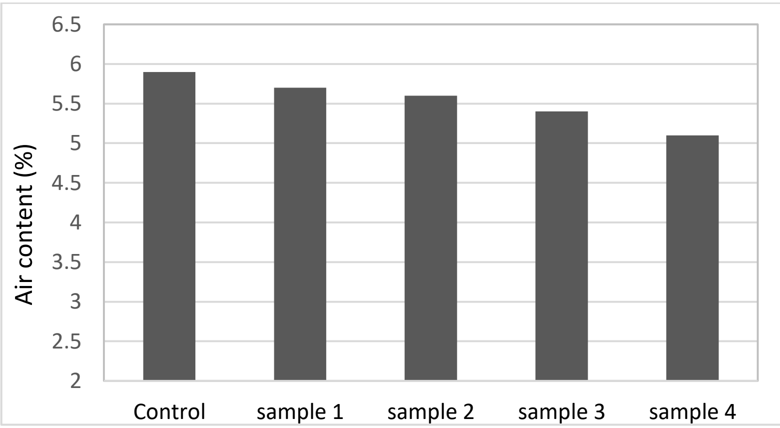

Figure 5: Effect of salty water on air content of mortar mixes

3.3 Influence of salty water on setting time of mortar mixes

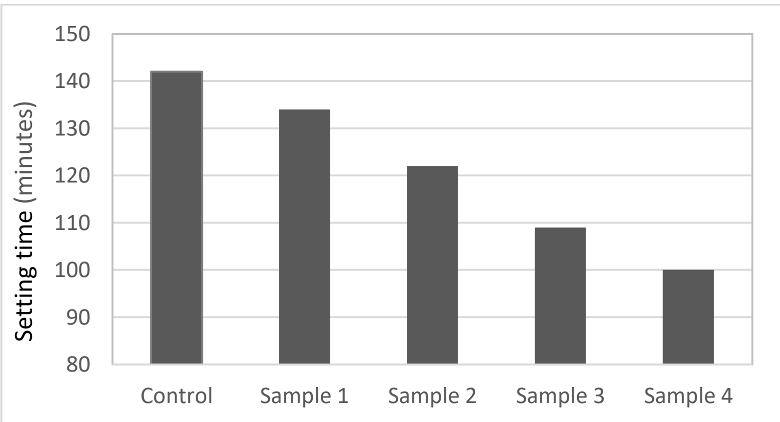

Figure 6: Effect of salty water on setting time of mortar mixes

Results revealed that the accelerating effect of salinity on setting time was noticed for all mixes but to varying extents compared to the control. (Figure 6). It was found that the setting time for all mortar mixes were

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decreased with increasing salinity. Acceleration percent relative to control mortar were 22,26,29 and 33% for sample 1, 2, 3 and 4 respectively. The accelerating impact of salinity on the setting time of cement-based materials was previously reported [2, 9, 32].

3.4 Influence of salty water on compressive strength of mortar mixes

When compared to the control, the compressive strength is stronger at 3 days and weaker at 7 and 28 days (Figure 7). This is due to the early stages of hydration reactions being accelerated by the salty water. Additionally, the C_2S reaction with saline water occurs earlier and, as a result, strength reduces in the later stages [48]. Furthermore, the long term compressive strength loss of mortar can be attributed to the crystallisation of salt in the salty water.

Several studies have been carried out to investigate the effect of salty water on the compressive strength; however, some contradictory outcomes were noted with a tendency for some studies to show that salty water decreases the strength while others indicate increases in strength (Table 1).

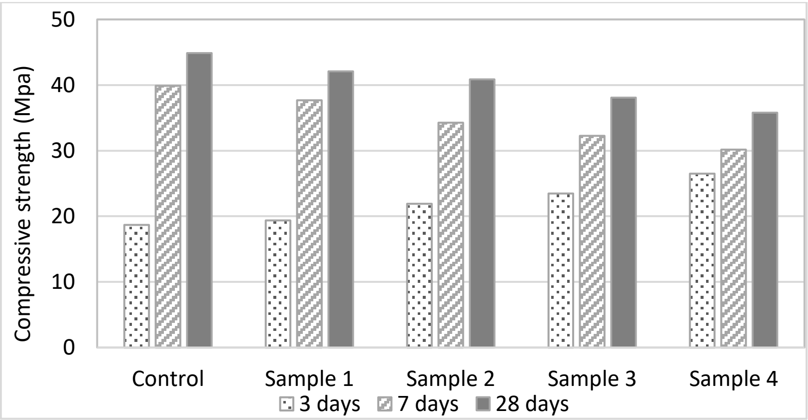

Figure 7: Effect of salty water on compressive strength of mortar mixes

3.5 Influence of salty water on hydration of mortar mixes

The thermal profile resulted from the semi-adiabatic calorimeter suggested that the incorporation of salty water with mortar mixes accelerated the hydration reactions. All mortar mixes exhibited an increase in the peak heights of evolved temperature and reached a peak that occurred earlier than for control (Table 4 and Figure 8). The peak temperature for all mortar mixes were increased with increasing salinity.

Mortar mixes incorporating salty water with TDS equal 225800 mg/L (sample 4) exhibited the greatest acceleration where the peak temperature increased by 31% and the time to peak accelerated by 42% compared to the mortar mixed with fresh water.

The above observations are generally consistent with the results from existing studies [2, 31, 49-51].

Table 4: Summary of hydration characteristics from semi-adiabatic for mortar containing salty water

Mix	TDS (mg/L)	Peak temperature (°C)	Time to peak temperature (minute)
Control	682	15.54	600
Sample 1	623500	15.82	500
Sample 2	100720	18.96	450
Sample 3	158600	19.72	400
Sample 4	225800	20.35	350

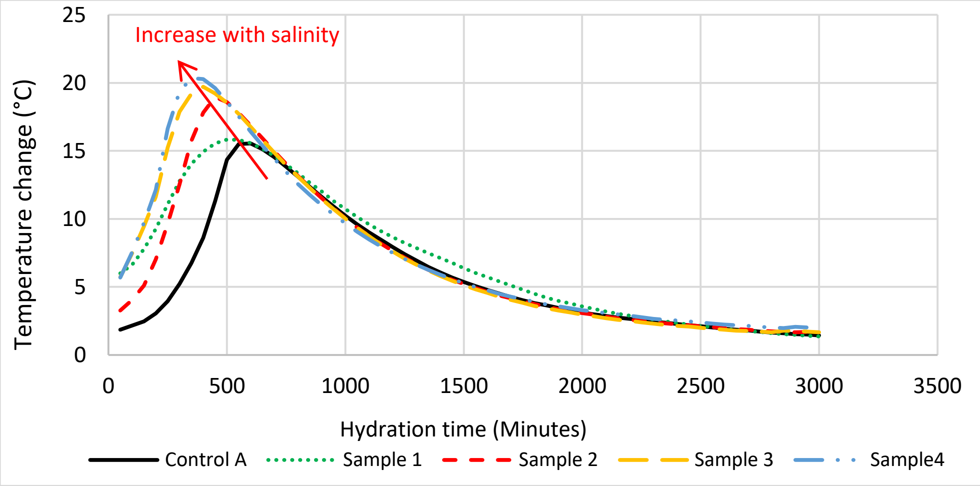

Figure 8: Thermal profile from semi-adiabatic calorimeter for mortar containing salty water

The mechanisms responsible for accelerating and enhancing the hydration reaction are not precisely known. However, several theories have been previously proposed for the acceleration caused by saline water.

Early age hydration is accelerated by the formation of calcium chloride (Ca Cl_2) by the reaction of calcium hydroxide (Ca (OH)2) in cement mortar pore solution with sodium chloride (NaCl) from salty water [52]. Furthermore, Vidick et al. [53] indicated that the acceleration phenomenon can be attributed to the ability of calcium chloride to shorten the time necessary to achieve supersaturation with respect to portlandite (CH) in cement.

Mattus and Gilliam [54] stated that the setting time of cement accelerates by calcium and magnesium cations due to the saturation of the cement pore solution regarding calcium. Saturation is achieved more quickly, and crystal growth is therefore promoted, with magnesium substituting for calcium to form its hydroxide. Furthermore, Machado et al. [32] showed that the penetration of $CaCl_2$ in the pore of cements could accelerate the reaction due to the hydration of silicates which reduced their crystallisation time, thus accelerating the setting of the materials. Lee [55] stated that sodium and potassium may accelerate the hydration of C_3S and may affect the rate of the precipitation of calcium hydroxide in cement pores. Moreover, Ramachandran [56] showed that the setting behaviour is accelerated when calcium chloride combines with the aluminate and ferrite chloride hydrate respectively. They may also combine in a finely divided form and provide nuclei for the hydration of the silicate phases. Hydration of C_2S by itself is known to be accelerated by calcium chloride.

Ramachandran also suggested several potential mechanisms for the accelerated cement hydration due to incorporation of salty water. Firstly, the accelerated hardening is due to the coagulation of the hydro silicate ions (possible polymerisation). Accelerated formation of CSH has also been considered. Secondly, In the solution of calcium chloride, higher dissolution of cement components and hydrated phases will occur. Chloride may combine with CH to form a basic chloride complex which, being a metastable phase within decomposition, can lead to increased dissolution of CH and concomitant acceleration of the hydration process. Thirdly, as a result of the diffusion of Cl ions through the initially formed hydrates and accelerated outward diffusion of OH ions, there is a quickening in the precipitation of CH and acceleration in the decomposition of calcium silicates. Finally, the presence of calcium chloride is more likely to produce the C_4AH_{13} instead of C_3AH_6 phase and this formation of crystalline C_4AH_{13} results in higher strengths.

IV. CONCLUSION

The incorporation of salty water reduced the workability of cement mortar by 8 – 22%, slightly increased the wet density (up to 1.3%) compared to the control whereas no relationship was found between salinity and air content. Furthermore, incorporating saline water with mortar accelerated the hydration reactions. Consequently, high early heat of hydration resulted in reduced setting time.

Data from the both calorimetry and compressive strength testing showed that the salty water had complex effects, including accelerated hardening, increased early strength (3 days) but reduced long term strength (7 days, 28 days). It was apparent that the degree of impact depended on salinity level and ionic composition of the salty water. Further work is needed to better identify and explain these trends.

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