

# 1                    **Shelf Life of Alkali Activated Cement: Effects of Storage** 2                    **Condition and Duration**

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## 4                    **Abstract**

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7    Hydraulic cements based on the alkali aluminosilicate chemistry tend to be more reactive than  
8    Type I Portland cement. An experimental investigation was conducted in order to evaluate the  
9    effects of storage in air and in sealed condition on the properties and performance of alkali  
10    aluminosilicate cements. Different storage durations up to 28 days were considered in this  
11    experimental study. Hydraulic cements were evaluated based on their morphology, chemistry and  
12    mineralogy, hydration kinetics, strength development characteristics, and microstructure of the  
13    resultant hydration products. The early-age hydration kinetics and strength development  
14    characteristics of the alkali aluminosilicate cement were found to be altered by storage, especially  
15    when the cement was exposed to air. Older cements also produced hydration products that were  
16    more susceptible to microcracking when dried under vacuum. Longer-term strength development  
17    characteristics of cements were less influenced than their shorter-term strength development  
18    characteristics with duration of storage in sealed or exposed condition.

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22    **Keywords:** shelf life, aging, alkali **activated** cement, cement clustering, mechanical properties

23 **1. Introduction**

24 Portland cement offers significant performance and cost advantages which have allowed concrete  
25 to assume a prevalent position as the primary material of construction in diverse fields of  
26 application. The significant consumption of Portland cement, and the high growth rate of its global  
27 production have drawn attention to the sustainability of Portland cement manufacturing. The  
28 relatively large carbon footprint and energy content of Portland cement have prompted efforts to  
29 make quantitative and qualitative changes in cement chemistry and production methods. The  
30 industry has been successful in implementing steps that have quantitatively reduced the CO<sub>2</sub>  
31 emissions of cement manufacturing plants, and improved their energy-efficiency without altering  
32 the fundamental chemistry of Portland cement [1]. Parallel with these efforts, initiatives have been  
33 launched to develop new classes of hydraulic cements with inherently sustainable processing  
34 methods and chemistries [2, 3].

35 The Portland cement chemistry is based largely on calcium silicates which, upon hydration, form  
36 calcium silicate hydrate with desired binding qualities. An alternative chemistry which can compete  
37 with Portland cement in mainstream construction applications is based on alkali aluminosilicates  
38 that, upon binding of water, yield alkali aluminosilicate hydrates with highly desired binding  
39 qualities [4, 5]. This alternative cement chemistry is highly robust, and can take advantage of  
40 different (abundant) natural raw materials (e.g., clay and soda ash) and industrial byproducts (e.g.,  
41 coal fly ash and metallurgical slags). It has a strong resemblance to some abundant natural  
42 aluminosilicate minerals [6, 7]. Investigations of some historic structures (e.g., Pantheon in Rome),  
43 that have withstood the test time for about 2000 years, suggest that they have also been based on  
44 the alkali aluminosilicate chemistry [8]. Alkali aluminosilicate hydrates offer characteristically high

45 levels of impermeability, weathering resistance, and thermal and chemical stability [9, 10]; they  
46 effectively protect reinforcing steel against corrosion [11].

47 One important drawback of the common approach to production of alkali aluminosilicate hydrate  
48 binders is its “two-part” nature where an alkaline solution is added to an aluminosilicate precursor  
49 [12-14]. This approach contrasts the normal approach to production of Portland cement binders  
50 where water is added to cement to induce hydration reactions. Efforts have been initiated in recent  
51 years to produce hydraulic cements based on the alkali aluminosilicate chemistry; these “one-part”  
52 cements undergo hydration reactions upon addition of water [15-18]. The combination of  
53 aluminosilicate precursors and alkalis, however, is highly reactive. There would thus be concerns  
54 about initiation of hydration reactions by simple exposure to the humidity in air. This would reduce  
55 the ‘shelf life’ of hydraulic cements based on the alkali aluminosilicate chemistry.

56 This investigation was undertaken to assess the ‘shelf life’ of an alkali aluminosilicate cement  
57 exposed directly to air or stored in a sealed container. The effects of storage time on the chemical  
58 and mineralogical characteristics, microstructure, and some key engineering properties of the  
59 hydraulic cement were evaluated.

## 60 **2. Materials and methods**

### 61 **2.1 Materials**

62 The alkali aluminosilicate cement used in this investigation was produced via mechanochemical  
63 processing (using ball milling) in previous work of same authors [19]. The proportions of the raw  
64 materials used for production of the alkali activated cement were coal fly ash at 45 wt.%, slag at 25

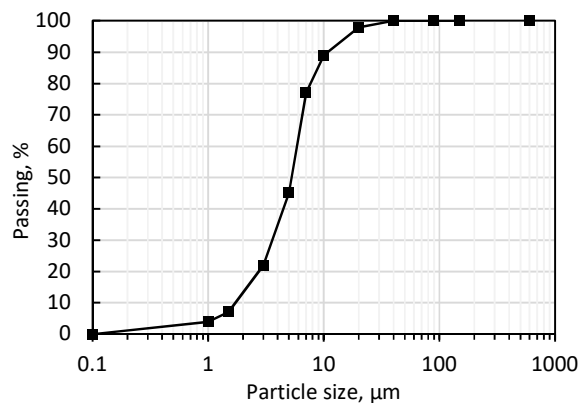
65 wt.%, albite at 15 wt.%, sodium silicate 7 wt.%, sodium hydroxide at 3 wt.% and borax at 1 wt.%.  
66

66 The chemical compositions and Blaine fineness for the cement are presented in Table 1.

67 Table 1. Chemical compositions (wt.%) and Blaine fineness of the alkali **activated** cement used in  
68 this investigation.

SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	Blaine Fineness, cm <sup>2</sup> /g
35.2	28.1	13.6	4.03	3.73	1.14	8.89	0.53	3960

69 The particle size distributions of the alkali **activated** cement are presented in Figure 1. The median  
70 particle size was 7.4 μm.



71  
72 Figure 1. Particle size distributions of the alkali **activated** cement used in this investigation.

## 73 2.2 Methods

74 The hydraulic cements were stored in two conditions: (i) sealed in a Ziploc bag where 250 g of  
75 cement was placed in a bag of 950 cm<sup>3</sup> volume, and the bag was squeezed by hand in order to  
76 remove air prior to sealing (Figure 2a); and (ii) directly exposed to air (Figure 2b) as a 10 mm layer  
77 of cement with about 175 cm<sup>2</sup> exposed surface area, placed in a shallow plastic container. The  
78 cement bags and plastic containers containing the cement samples were stored at 23±2°C and

79 55±5% relative humidity. Fresh cement (tested within 8 hours after production) was used as control  
80 in this experimental work.

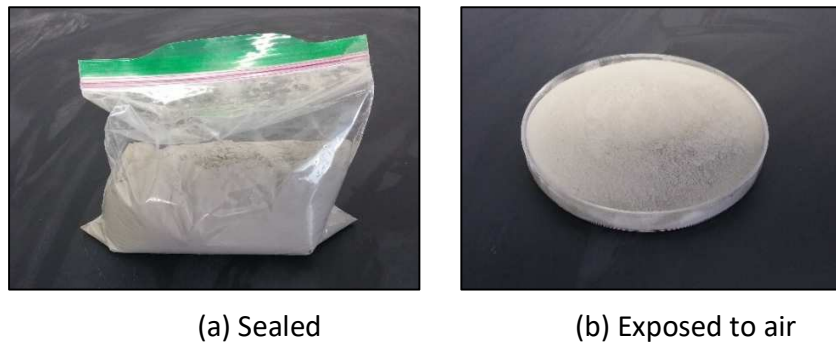


Figure 2. Alkali **activated** cement in sealed and exposed conditions.

84 Thermogravimetric analyses were conducted on the fresh and aged cement using a Perkin Elmer  
85 TGA 4000 with temperature raised from 50 to 950°C at a heating rate of 15°C/min in nitrogen  
86 atmosphere.

87 The pH of cement in solution, conductivity and total dissolved solids (TDS) were evaluated by  
88 adding the cement to deionized water at 1% concentration by mass in a vial, and placing the  
89 solution on shaking table for 1 hour. A pH meter (ORION STAR A215) was used to determine the  
90 pH, conductivity and TDS of solutions.

91 The cement mineralogy was evaluated via x-ray diffraction (XRD) spectroscopy using a Bruker D8  
92 daVinci diffractometer equipped with Cu x-ray radiation operating at 40 kV and 40 mA. Powder  
93 XRD was carried out at a reflection angle range of  $2\theta = 10-80^\circ$ , **with a scanning duration of 45**  
94 **minutes.**

95 Scanning electronic microscopy (SEM) of the test specimens was performed using a JOEL-5000  
96 instrument on the cement and its hydrated pastes. The specimens were first coated with gold-

97 palladium alloy using a sputter coater (DESK II), and then imaged in high-vacuum mode at an  
98 accelerating voltage of 10 or 15 kV.

99 The heat of hydration reactions was measured by performing the calorimetry test (ASTM C1679),  
100 using an I-cal 2000 HPC calorimeter. For performance of this test, cement was mixed manually with  
101 water for 30 seconds at a water/cement ratio of 0.35. The paste was placed inside the calorimeter,  
102 and its heat release was monitored over 7 days.

103 The compressive strength tests were performed per ASTM C109 on 50-mm cubic mortar specimens  
104 after 1, 7 and 28 days of curing in **sealed condition (>95% relative humidity) at room temperature.**

105 The initial set time of pastes was measured per ASTM C191 using the Vicat needle apparatus. The  
106 amount of water mixed with cement for measurement of set time was selected to produce a  
107 normal consistency per ASTM C187.

108 **The effects of aging on the alkali activated cement and binder characteristics have been considered**  
109 **up to 28 days; the duration was chosen based on experimental studies that have shown the highly**  
110 **reactive cement powder (due to presence of highly reactive alkalis, such as sodium hydroxide,**  
111 **calcium oxide).**

## 112 **3. Results and discussion**

### 113 **3.1 Effect of storage duration and condition on the dehydration kinetics of cement**

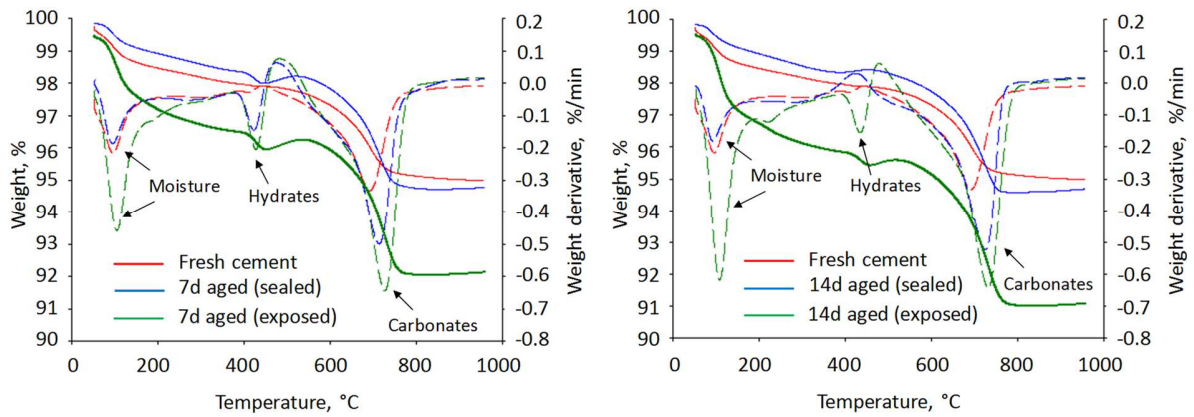
114 Figure 3 compares the TGA/DTA test data generated for fresh cement and the cement aged in  
115 sealed and exposed conditions over different time periods. Three main stages of mass loss were  
116 identified in the TGA data, with the corresponding DTA peaks occurring at: (i) ~140°C attributed to

117 loss of moisture; (ii)  $\sim 450^{\circ}\text{C}$  attributed to decomposition of hydrates; and (iii)  $\sim 750^{\circ}\text{C}$  attributed to  
118 decomposition of carbonates. The changes in these peaks as a result of different storage conditions  
119 and durations are discussed in the following.

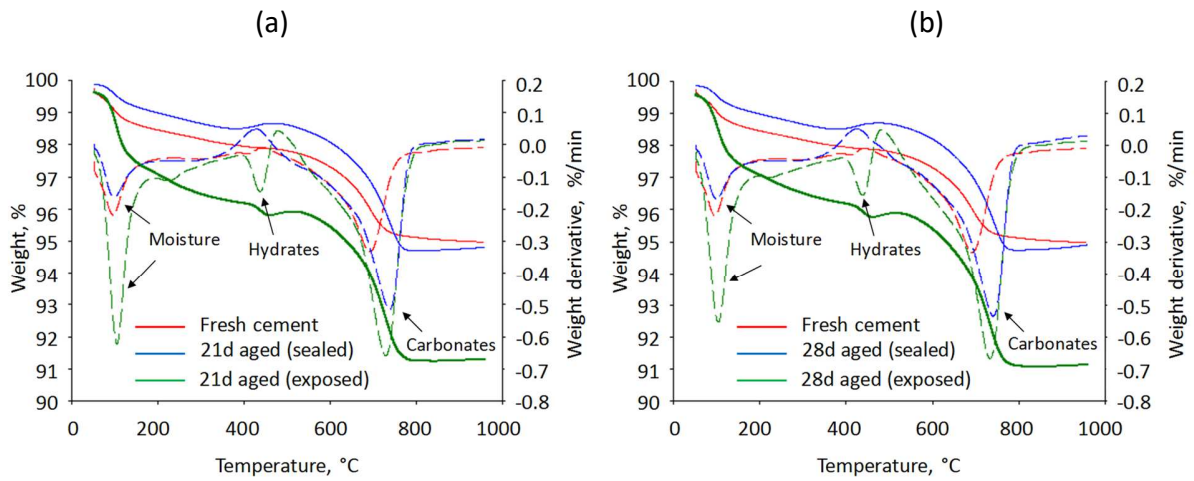
120 The moisture content of the fresh cement was observed to be less than 1 wt.%. Storage of cement  
121 in sealed condition for 7 days produced a small reduction in moisture content (Figure 3a); further  
122 reductions were noted after 14, 21 and 28 days of storage in sealed condition (Figures 3b, 3c and  
123 3d, respectively). This trend could be explained by minor hydration of cement which binds moisture  
124 into the products of hydration. For the cement exposed to air at  $55\pm 5\%$  relative humidity, there  
125 was a rise in moisture content over time. The measured value of moisture content was 2.9 wt.%  
126 after 7 days of storage (Figure 3a), which increased after 14 and 21 days of storage (Figures 3b and  
127 3c, respectively), and reached 3.2 wt.% after 28 days of exposure to air (Figure 3d). It seems that  
128 the absorption of moisture from air (at  $55\pm 5\%$  relative humidity) more than compensated to any  
129 loss of moisture to hydration reactions.

130 The cement stored for 7 days in both sealed and exposed conditions (Figure 3a) exhibited a new  
131 peak at  $\sim 450^{\circ}\text{C}$  (corresponding to decomposition of hydrates), and also a rise in the carbonate peak  
132 at  $\sim 750^{\circ}\text{C}$ . These DTA peaks were sharper after 7 days of storage for cement exposed to air (versus  
133 that kept in sealed condition). These trends are indicative of minor hydration and carbonation  
134 reactions occurring during the storage time, which tend to be more pronounced for the cement  
135 exposed to air. While the DTA peak associated with the decomposition of hydrates was missing in  
136 fresh cement, that corresponding to the decomposition of carbonates was present in fresh cement.  
137 This could be due to the presence of carbonates among the cement raw materials or the formation  
138 of carbonates in the course of processing of the hydraulic cement. The carbonate peak appeared to

139 be sharper for longer storage durations, which could be due to the formation of more stable forms  
 140 of carbonates over time. The carbonate peak got more pronounced over the first 14 days of  
 141 storage, after which it was more stable probably due to exhaustion of the readily available species  
 142 that are prone to carbonation.



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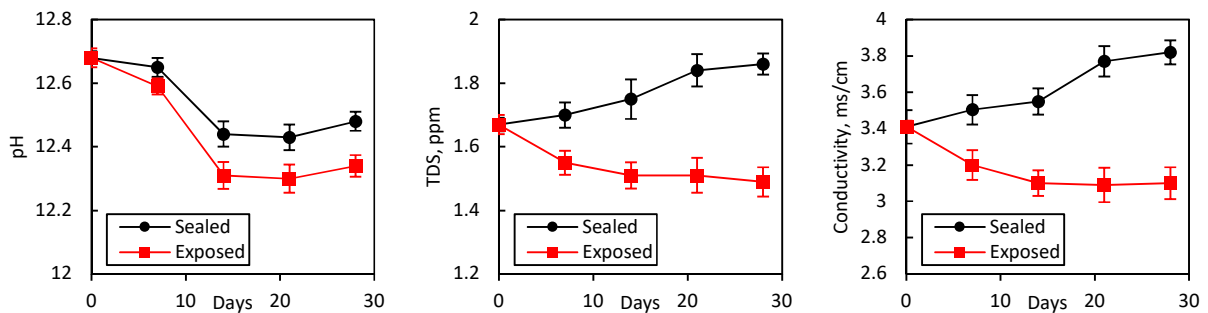
147 Figure 3. TGA/DTA test results for fresh cement and cement stored for (a) 7 days, (b) 14 days, (c) 21  
 148 days and (d) 28 days in sealed or exposed condition.

149 **3.2 Effect of storage duration and condition on the pH, total dissolved solids (TDS) and**  
 150 **conductivity of the cement in water**



151 Figure 4 compares the pH, TDS and conductivity test result for cement in fresh state and also after  
 152 different durations of storage in either sealed condition or exposed to air. As shown in Figure 4a,  
 153 the pH of cement dropped slightly after 1 week of storage in both sealed and exposed conditions.  
 154 After 14 days, however, pH dropped significantly for both storage conditions; this drop was more  
 155 pronounced for exposed cement. The pH of cement remained reasonably stable beyond 14 days.  
 156 The drop in pH can be attributed to carbonation of cement (supported by the TGA data presented  
 157 in Figure 3) [20] and probably by hydration (alkali activation) phenomena that could bind alkalis  
 158 into aluminosilicate hydrates. Carbonation and hydration reactions are enabled by exposure to the  
 159 humidity and carbon dioxide in air.

160 For the cement exposed to air both TDS and conductivity decreased with storage time (Figures 4b  
 161 and 4c, respectively). An opposite trend was observed for cement stored in sealed condition.  
 162 Carbonation and hydration reactions can be used to explain the drops in TDS and conductivity for  
 163 the cement exposed to air. One could hypothesize that the rise in TDS and conductivity for the  
 164 cement stored in sealed condition is due to the (surface) alkali activation of the aluminosilicate  
 165 precursors releases aluminum and silicon ions to the solution [21], thereby raising the TDS and  
 166 conductivity values. This trend could have been overshadowed by carbonation reactions for the  
 167 cement exposed to air.

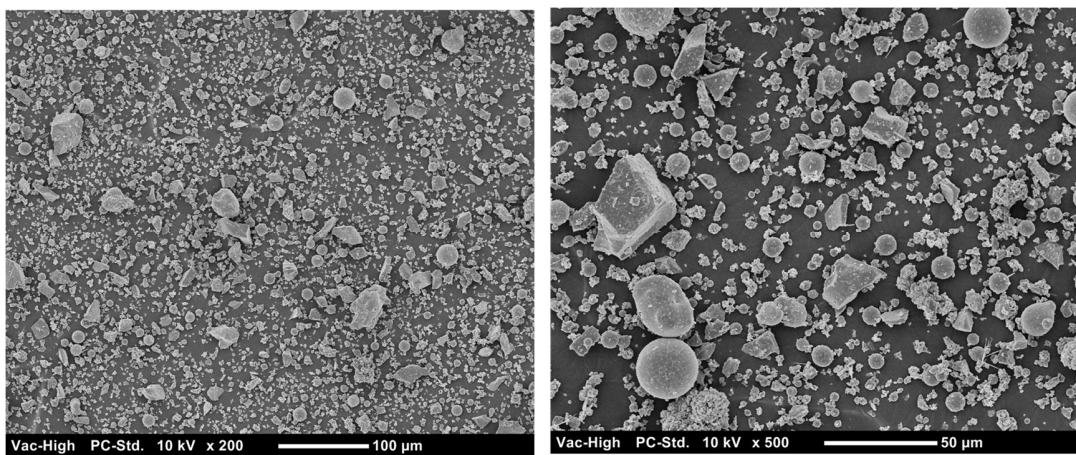


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169 Figure 4. pH, total dissolved solids (TDS) and conductivity of fresh and aged cement in water.

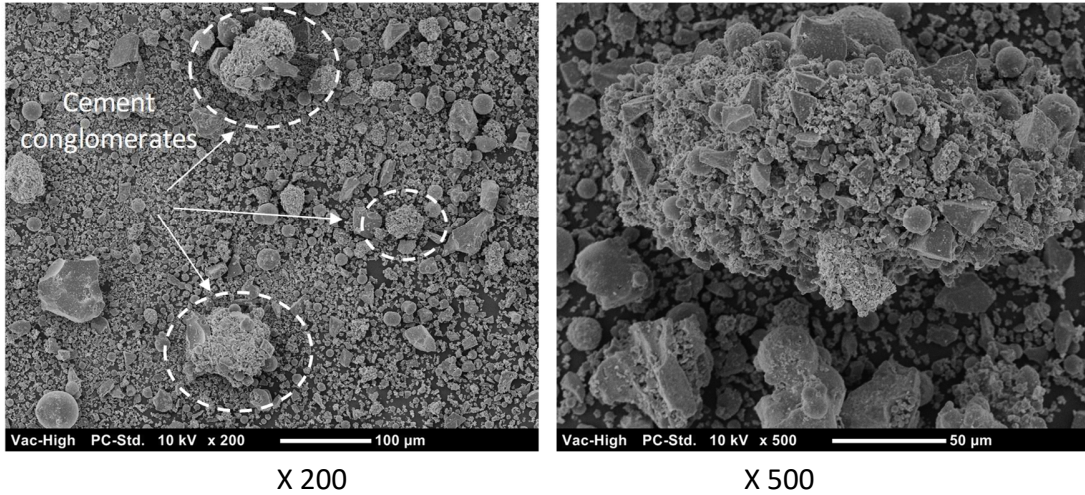
170 **3.3 Effect of storage duration and condition on the cement morphology**

171 Figure 5 presents SEM images (at two magnifications) for the cement aged for 28 days in sealed  
172 and in exposed conditions. The cement stored in sealed condition (Figure 5a) appeared as  
173 individual particles that were mostly 10 to 20 micrometer in size. The cement stored in exposed  
174 condition, on the other hand, formed clusters of cement particles that were 100 micrometer in size  
175 (referred to as cement conglomerates in the SEM image of Figure 5b). The formation of these  
176 agglomerates could be due to minor near-surface hydration/carbonation reactions of cement  
177 particles that were exposed to air (as noted in the TGA/DTA results in Figure3).



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X 200 X 500  
(a) Cement stored in sealed condition for 28 days



181  
182  
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184

X 200 X 500  
(b) Cement exposed to air for 28 days

Figure 5. SEM images for aged cement in sealed and exposed conditions.

185 Figure 6 shows the cement stored for 28 days in sealed and exposed conditions. Some lumps  
186 (about 5 mm in size) were formed when cement was stored in exposed condition; such lumps did  
187 not appear in cement stored in sealed condition.



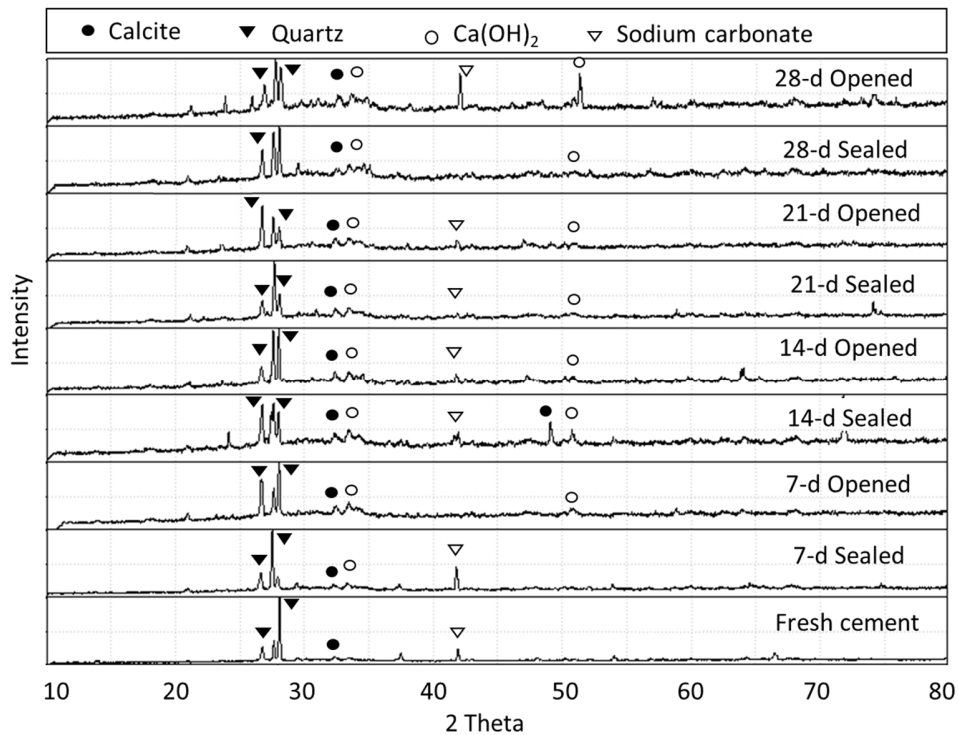
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(a) (b)

Figure 6. Images of the cement stored for 28 days in sealed (a), and in exposed (b) conditions.

190 **3.4 Effect of storage duration and condition on the cement crystalline phases**  
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192 Figure 7 presents the XRD spectra of the fresh cement and the cement stored for 7, 14, 21 and 28  
 193 days in sealed and exposed conditions. Calcite was formed in cement after 7 days of storage in  
 194 both sealed and exposed conditions. The calcite peak was observed to be more pronounced with  
 195 increasing duration of storage. Portlandite peak at  $2\theta$  of  $51^\circ$  was observed in cement after 7 days of  
 196 storage in both condition; this peak was more pronounced after 28 days storage in exposed  
 197 condition. The XRD results are consistent with the observation made in analysis of the TGA results  
 198 (see Figure 3).



199  
 200 Figure 7. XRD spectra for fresh cement and cement after different duration of storage in sealed and  
 201 exposed conditions.

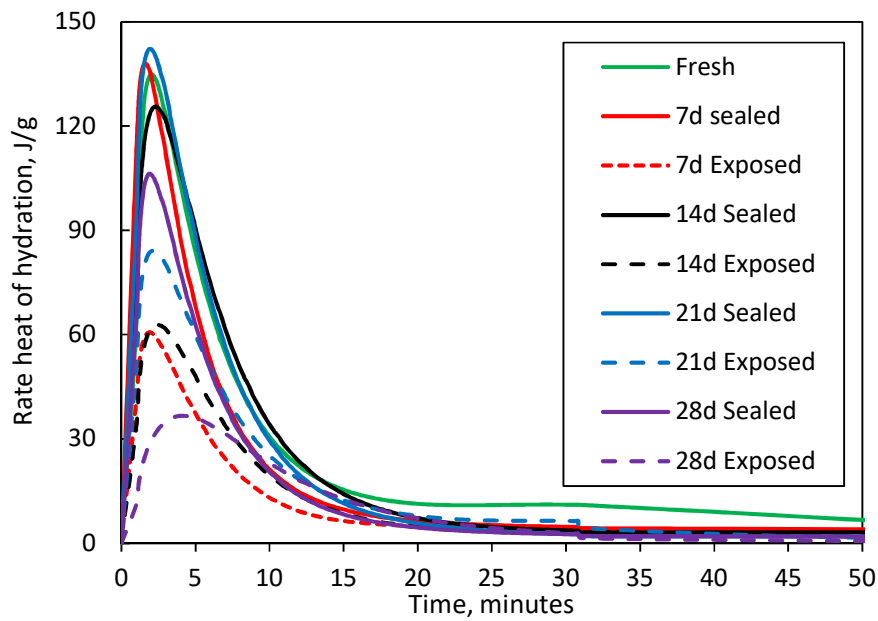
### 202 3.5 Effect of storage duration and condition on the cement hydration kinetics

203 The heat of hydration test results (up to 7 days) for fresh cement and cements stored for different  
 204 durations in sealed and exposed conditions are presented in Figures 8 and Table 2. Hydraulic

205 cements generally exhibit an instantaneous exothermic peak upon addition of water. This peak  
206 could be attributed to the exothermic processes involving instantaneous adsorption of the solution  
207 on the surface of cement particles, and breakdown of the Si-O-Si and Al-O-Al bonds which release  
208 ionic species of Si and Al into the solution [22]. In the case of Portland cement, this instantaneous  
209 peak is followed by a dormant period before the primary exothermic peak appears after few hours.  
210 In the case of the alkali **activated** cements considered here, however, the initial peak is (in most  
211 cases) the only exothermic peak. In other words, any instantaneous exothermic phenomena are  
212 followed (in a continuous way) by exothermic hydration reactions without the appearance of a  
213 dormant period. This exothermic peak is reached within few to several minutes. It should be noted  
214 that, in spite of its distinct heat release (and hydration kinetics) attributes, the cement produces  
215 mortar and concrete materials with acceptable fresh mix workability retention over time and set  
216 time.

217 Exposing the alkali **activated** cement to air greatly impacted its exothermic heat release attributes.  
218 The main exothermic peak weakened significantly with duration of storage in exposed condition.  
219 This peak was sharper for both the fresh cement and the cement stored in sealed condition. The  
220 intensity of the exothermic peak decreased with increasing storage duration even for the case  
221 involving storage of cement in sealed condition. The fresh cement exhibited a second broad and  
222 weak peak at about 30 minutes, which did not appear for cements stored in sealed or exposed  
223 conditions; **which could also reflect the longer heat release for the paste prepared with fresh**  
224 **cement**. This second peak of fresh cement could correspond to the exothermic reactions when the  
225 alkaline solution is intensively adsorbed, and the OH<sup>-</sup> anions begin to attack the Ca-O, Si-O and Al-  
226 O bonds in cement particles to form alkali aluminosilicate hydrates [23].

227 The total values of heat release (Table 3) indicate that the cement stored in exposed condition  
 228 experienced significant loss of exothermic heat release (about 80% after 28 days of storage) when  
 229 compared with the fresh cement. The cement stored in sealed condition, on the other hand,  
 230 experienced minor loss of the total heat released (less than 25% loss for 28 days of storage) when  
 231 compared with the fresh cement.



232

233 Figure 8. Rate of heat release test results.

234 Table 3. Total heat release of cement in fresh state and after different periods of storage in sealed  
 235 or exposed conditions.

	0 days	Storage duration							
		7 days		14 days		21 days		28 days	
Fresh cement		Sealed	Exposed	Sealed	Exposed	Sealed	Exposed	Sealed	Exposed
Total heat, Kcal/kg	180	174	118	159	91	160	72	140	35

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237 **3.6 Effects of the storage duration and condition the initial set time and compressive strength**



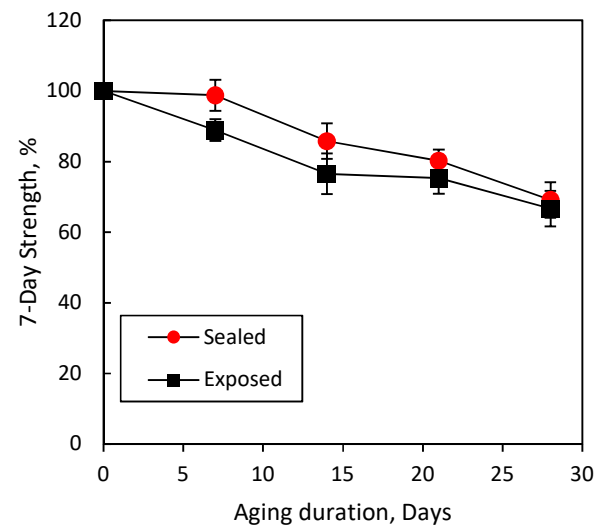
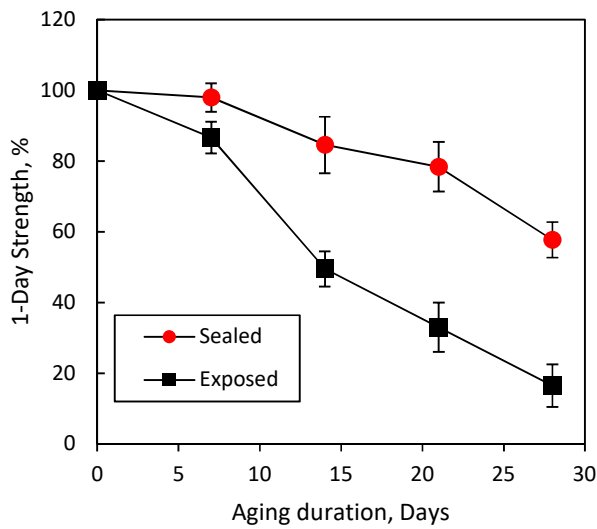
238 Table 4 presents the initial set time test results for cements tested immediately after production  
 239 and after different duration of storage in exposed or sealed conditions. The initial set time is  
 240 observed to decrease with increasing duration of storage, especially when the cement is exposed  
 241 to air during storage. After 28 days of storage in exposed condition, the initial set time of cement  
 242 dropped to about half of the cement tested immediately after production. The corresponding drop  
 243 in set time was about 20% after 28 days of storage in sealed condition. The drop in set time with  
 244 increasing storage duration could be attributed to: (i) reduction of pH (see Figure 4) for the cement  
 245 exposed to air, considering that low pH values enhance the solution of calcium compounds, leading  
 246 to rapid formation of calcium silicate hydrates [24, 25]; and (ii) exposure of cement to air could  
 247 lead to formation of some partially soluble carbonates that release  $\text{CO}_3^{2-}$  ions to the solution in the  
 248 course of hydration that induce accelerated carbonation reactions [26, 27].

249 Table 4. Initial set time test results obtained after different durations of storage in exposed and  
 250 sealed conditions.

		Storage duration									
		0 days		7 days		14 days		21 days		28 days	
Fresh cement		Sealed	Exposed	Sealed	Exposed	Sealed	Exposed	Sealed	Exposed	Sealed	Exposed
Initial set time, min	53 ±3	55 ±3	43 ±2	48 ±3	30 ±2	46 ±3	27.5 ±2	44 ±3	26 ±2		

251 The compressive strength test results for mortar specimens prepared using alkali **activated** cement  
 252 are presented in Figure 9 (as percent of the strengths obtained with fresh cement). The percent  
 253 loss in compressive strength with storage duration was more pronounced at early ages for the  
 254 cement stored in exposed condition. The differences between cements stored in exposed versus  
 255 sealed condition diminished when compression test was performed at 28 days of age. The losses in  
 256 compressive strength with increasing storage duration can be attributed to the partial hydration of

257 cement particles due to exposure to humid air, considering the reactive nature of alkali **activated**  
 258 cements. The loss in compressive strength at later ages could be less significant due to the bonding  
 259 of the new cement hydrates to those that formed during storage which may accompany further  
 260 hydration of the cement that hydrated partially in storage. The results also point at the importance  
 261 of using properly sealed bags for the cement developed in this work. Storage in silo could provide  
 262 the hydraulic cement with reasonable protection against exposure to humid air [25].

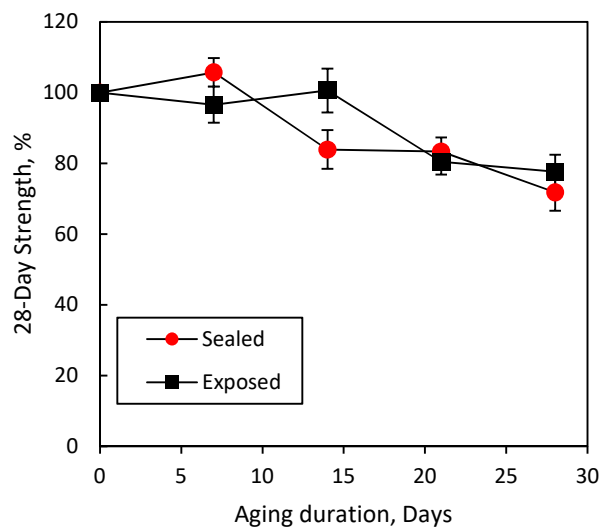


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(a) 1-day compressive strength

(b) 7-day compressive strength



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(d) 28-day compressive strength

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Figure 9. Compressive strength test results obtained at different ages versus the duration of

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storage in sealed and exposed conditions.

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### 3.7 Effects of the storage duration and condition on the microstructure of cement hydrates

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Figure 10 compares SEM images (taken after 28 days of room-temperature curing) for pastes

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prepared using fresh cement (Figure 10a), and cements stored for 28 days in sealed condition

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(Figure 10b) and in air (Figure 10c). A denser microstructure with less microcracking was observed

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in the case of the hydrated paste prepared with fresh cement. The paste prepared with aged

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cement (Figures 10b and c), on the other hand, exhibited microcracking and a structure of

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apparently reduced density. The SEM images pointed at the reactivity of the fresh cement and its

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ability to produce alkali aluminosilicate gel. Formation of hydrates and carbonates on cement

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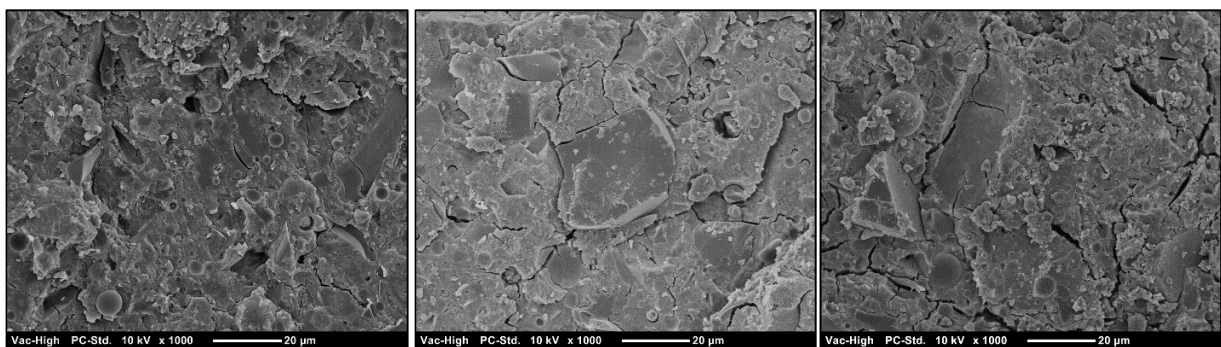
particles could explain the reduced reactivity of aged cements, and thus the lower integrity of their

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hydration products. Formation of microcracks in both sealed and exposed hydrated cement pastes

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could explain the reduction in the compressive strength observed in Figure 9.



280

(a)

281

(b)

(c)

282

Figure 10. SEM images of hydrated paste prepared using fresh cement (a), and cements aged for 28

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days in sealed (b) and exposed (c) conditions.

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285

## 4. Conclusions

286 An experimental program was conducted in order to assess the effects of storage in sealed and  
287 exposed conditions for periods up to 28 days on the qualities of an alkali **activated** cement. The  
288 effects of storage duration on the cement chemistry, morphology, hydration kinetics, set time,  
289 strength development characteristics, and the microstructure of the resulting hydrated cement  
290 pastes were evaluated. The following primary conclusions were derived based on the data  
291 generated in this experimental work.

- 292 • Storage in sealed condition had minor effects on the hydration kinetics of the alkali  
293 **activated** cement which were assessed by monitoring its heat of hydration and strength  
294 development characteristics. Some hydration (alkali activation) and carbonation reactions  
295 occurred during storage in sealed condition, **yielding significant drop in the total heat of**  
296 **hydration after 28 days of storage (140 Kcal/kg in sealed compared to 35 Kcal/kg in exposed**  
297 **condition)**. The morphology of cement particles did not experience notable changes after  
298 storage in sealed condition. Hydrated cement pastes prepared with the cement aged for 28  
299 days in sealed condition experienced more microcracking when subjected to vacuum drying  
300 when compared with pastes prepared with fresh cement.
- 301 • Exposure to air during storage of the alkali **activated** cement significantly reduced its early-  
302 age (1-day) strength development potential (**by approximately 80% after 28 days of storage**)  
303 . The effects on 7- and 28-day strength development characteristics were less pronounced  
304 (**32% and 27%, respectively after 28 days of storage**). The effects of storage in air on the  
305 hydration kinetics and microcracking tendency of the cement paste when subjected to  
306 vacuum drying was more pronounced in the case of the cement stored in exposed  
307 condition.

- 308 • The effects of aging of cement on its performance characteristics could be attributed partly  
309 to its partial hydration (alkali activation) and carbonation in the course of storage in sealed  
310 and especially exposed condition.
- 311 • While the work reported in this paper investigated the effect of two storage condition on  
312 the shelf life of alkali activated cement, other storage condition (such as temperature,  
313 moisture, etc.) must be investigated in future studies.

#### 314 **Acknowledgment**

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317

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