

# The silica-alkaline reaction of aggregates is more realistic than the alkaline-carbonate reaction of aggregates

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## Abstract

The main objective of this research is shedding light on the siliceous and carbonate reactions. Is the use of alkali-silica reactions suitable for aggregates containing limestone? Alkali-silica reaction (ASR) and the related deformation are significant issues with concrete construction durability that date back to the 1940s. This harmful reaction results in excessive expansion and fissures, which can seriously damage concrete structures. Understanding the ASR mechanism is still difficult because of complicating processes and reactions, despite long-standing observation and several ASR investigations. Therefore, the alkali mortar bar test was carried out for five samples containing limestone and dolomitic stone so that it could obtain results within 56 days, compared to obtaining results in 360 days by using the carbonate alkaline reaction test. In this study, the alkali mortar bar test and the alkali carbonate test were determined on three limestone samples and two dolomitic stone samples. Casting of mortar bars with the specified aggregate (2.36 mm- 150 µm), cured in 1 N of sodium hydroxide solution at 80 °C and a concrete prism of alkali carbonate reaction with aggregate of (19 mm - 12.5 mm), stored at 38 °C over water. The accelerated mortar bar reaction gave a higher rate of expansion in a short period than the carbonate reaction, although the carbonate reaction proved that the aggregate was reactive after a long period and it was not exposed to high temperature and sodium hydroxide solution. The results showed that the accelerated mortar bar reaction gave a higher rate of expansion in a short period than the carbonate reaction, although the carbonate reaction proved that the aggregate was reactive after a long period and it was not exposed to high temperature and sodium hydroxide solution. The results of the accelerated mortar bar test and the alkali-carbonate test were applied to SEM analysis, which confirmed that calcite and brucite were produced as a result of the reaction between the lime stone and dolomite crystals and the alkali content in the cement. Keywords: alkali silica reaction, alkali carbonate reaction, expansion, mortar bar, concrete prism Kulcsszavak: alkáli-szilikát reakció, alkáli-karbonát reakció, duzzadás, habarcs hasáb, beton hasáb

## 1. Introduction

By creating concrete mixtures that minimize reaction harm and creating technologies to reduce the negative effects of negative expansion in existing buildings, it is essential to have a thorough understanding of the ACR technique in order to choose the best method for mitigating negative growth in new concrete structures. Despite the fact that these ACR and ASR cause concrete to degrade in similar ways, the reaction products that distress the concrete are different from one another, according to the knowledge currently available (ASR gel for ASR vs. brucite formation for ACR). The major several accepted theses that describe how ACR works and how aggregates expand as a result: (1) expansion-free dedolomitization (Swenson & Gillott, 1964); and (2) dedolomitization-induced increase (Hadley, 1964a, Tang et al., 1987; Ozol, 1994), 3) Katayama's Alkali Silica Reaction (Katayama, 2010; Gr (Fecteau et al., 2012) and (4) The alkali-silica reaction (ASR), which happens in cementitious materials, is caused when siliceous aggregate particles mix with alkali-sodium and alkali-potassium created during Portland cement

hydration. When pore fluid is absorbed, a gelatinous substance is produced, which causes expansion and internal stress in the concrete. The gel will damage the concrete if the following damage takes place: 1) there is a sufficient supply of reactive silica in the aggregate. 2) The concentration of OH<sup>-</sup> ions in the concrete's solution increases. 3) Water can be obtained from an outside source. Alkali-silica reactivity seems to have been a problem that was resolved by: 1) applying non-reactive aggregate. 2) Using cement that contains less than 0.6 percent by mass of sodium oxide (Na<sub>2</sub>O) equivalent low-alkali cement. 3) The maximum cement content is 500 kg/m<sup>3</sup>. 4) Using silica fume, slag, and other substitutes to partially replace cement. 5) Using the salts with ASR dominance. 6) Avoiding any contact between concrete and a moisture source. The products of the alkali silica reaction fling the aggregates, increase calcium, and form the chemical formula C-S-H (Katayama T., 2010; Thaulow et al., 1996; Leemann, A et al., 2013). Li, Q et al. (2014), Hou, et al. (2013), Hou, et al. (2004), Hou, et al. (2005), and Hou, et al. ASR crystallization's subsequent steps were established through carefully monitored mortar mixture studies. It dissolves and reacts with calcium hydroxide (CaOH)

to generate calcium-silicate-hydrate (C-S-H) when OH<sup>-</sup> ions attack silica, and this process continues until the nearby CH is consumed locally. The C-S-H at this stage is silica-rich and extremely limited. A hydrous ASR gel has a tendency to develop at this stage. This result supports Taylor's thesis that the chemical formula of ASR is mostly that of a pozzolanic reaction (Taylor, 1997). The phases of the reaction were confirmed by Li et al. (2014), who showed that the decrease of CH and the C-S-H reaction at 80 °C were necessary, that is the temperature that the widely accepted Standard ASTM test C1260 indicates. This reaction sequence provides a starting point for the microstructural variations discussed below. ASR happens when hydroxides in cementations materials react with high-reactivity silica in concrete. Alkali-silica gel can expand due to a reaction mechanism when the surrounding Portland cement's surrounding humidity is absorbed. The expansion may produce cracks in concrete. Most countries impose a limit on the typical amount of hydroxides (such Na<sub>2</sub>O) in cementations materials to control or prevent ASR in concrete. However, it was later shown that the necessary ratio of 0.6 percent equivalent alkalis was insufficient to prevent damage to the ASR. If concrete buildings' alkaline cement rates have only been as low as (0.45-0.50) percent equivalent Na<sub>2</sub>O. Stark, D. (1995). Aggregates that are reactive and that, when combined with sodium hydroxide, produce alkali silica gel as a consequence of the interaction between silicate and the alkali metal (NaOH). They consequently absorb water from their surroundings, stressing the concrete as a result. The existing alkaline content limitation of 0.6 allows for the control of the alkali aggregating reactivity. When applied to concrete pozzolanic materials, a different approach of controlling the alkali aggregate reaction yields stronger strength and less overall porosity. Rougher aggregate surfaces have higher strengths and smaller total porosities that come into touch with the cement paste than do rounded particles, which results in weaker concrete.

2. Materials and methods

2.1 Materials

The materials used in this study are aggregates, Ordinary Portland cement (OPC) and sand. Five samples of aggregates are examined in this study by XRD analyses and XRF analyses, which are mainly composed of calcite (CaCO<sub>3</sub>), dolomite (CaMg(CO<sub>3</sub>)<sub>2</sub>) and quartz (SiO<sub>2</sub>). Three limestone aggregate samples are supplied from El- Alameen area and the two dolomitic stone aggregate samples are derived from Attaqa area, Egypt. The cement is ordinary Portland cement with a grade of 42.5. (Suez Cement Company). Sand is quarried and obtained from Cairo's open quarries and El-Wahat El-Baharaya Road. The chemical compositions of the cement and aggregates were tested as shown in *Tables 1* and *2*. *Fig. 1* gives the chemical and mineralogical compositions of limestone aggregate samples (L) and the chemical and mineralogical of dolomitic stone aggregate samples (D) are derived from Attaqa area are given in *Fig. 2*. *Table 4* displays the physio-mechanical properties of limestone and dolostone aggregates.

Oxide content	Cement
L.O.I	2.92
MgO	1.45
SiO <sub>2</sub>	20.47
Al <sub>2</sub> O <sub>3</sub>	4.48
SO <sub>3</sub>	2.80
K <sub>2</sub> O	0.29
CaO	60.88
Fe <sub>2</sub> O <sub>3</sub>	5.49
Na <sub>2</sub> O	0.33

Table 1 Chemical compositions of cement (wt. %)
   
 1. táblázat Cement kémiai összetétele (m %)

Oxide content	Samples				
	E2	E3	E4	D1	D2
Al <sub>2</sub> O <sub>3</sub>	1.39	1.39	1.34	3.03	4.45
CaO	48.35	53.60	53.30	36.50	46.78
Fe <sub>2</sub> O <sub>3</sub>	1.18	0.93	0.87	0.62	1.13
MgO	1.47	0.92	1.71	13.90	6.02
L.O.I	29.79	33.20	33.20	43.60	39.60
SiO <sub>2</sub>	16.5	8.79	8.41	3.03	4.45

Table 2 Chemical compositions of aggregates (wt. %)
   
 2. táblázat Adalékanyagok kémiai összetétele (m %)

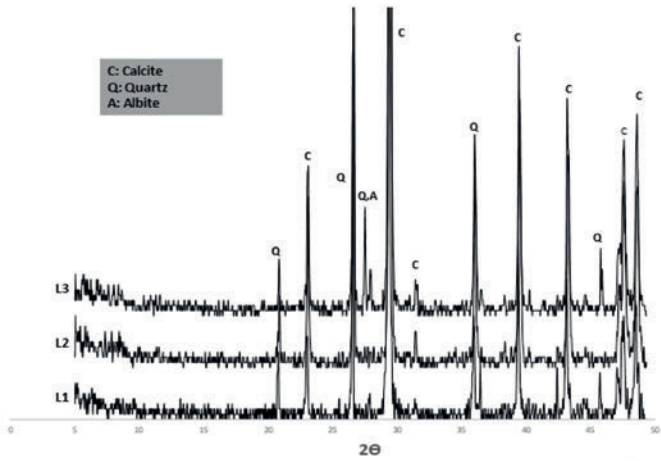


Fig. 1 The XRD pattern of limestone aggregate samples
   
 1. ábra Mészkeő adalékanyag XRD mintája

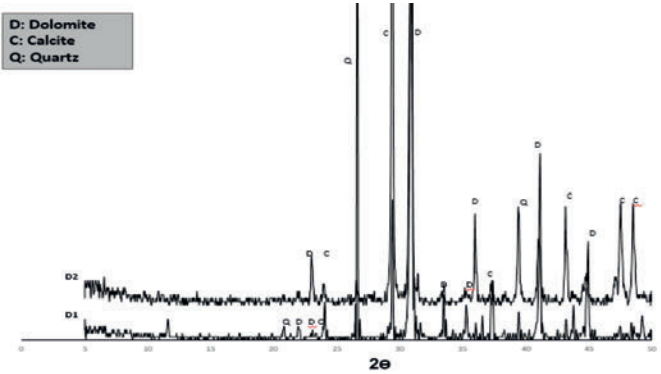


Fig. 2 The XRD pattern of dolomitic aggregate samples
   
 2. ábra Dolomit adalékanyag XRD mintája

Physical parameters	L2	L3	L4	D1	D2
Water absorption ( $W_a$ ) %	3	3.4	2.1	2.4	3.7
Specific gravity ( $G_{od}$ )	2.8	2.9	2.6	2.7	3
Aggregate impact value (AIV) %	35	37	20	31	39
Flakiness index ( $I_F$ ) %	41	41.5	20	24	43
Elongation index ( $I_E$ ) %	29	32	23	23	32
Los Angeles abrasion value (LAV) %	42	42	25	26	43

Table 3 Physico mechanical properties of limestone and dolostone  
3. táblázat Mésző és dolomit fizikai-mechanikai tulajdonságai

$W_a$ ,  $G_{od}$ , AIV,  $I_F$ ,  $I_E$ , and LAV have all been physical properties to their own minimum, maximum, and mean values measured in Table 3, Limits of roads and concrete aggregates are defined by BS and ASTM specifications, and also related BS and ASTM standards. According to ASTM-127, the  $W_a$  content of aggregate used in cement concrete will be less than 2.5 percent. This means these limestones meet the water absorption requirements for cement concrete. Calcite crystals have a specific gravity of 2.60 to 2.78, while concrete blocks require a lower limit around 2.60. It signifies that the specific gravity results from these stones are in the limits of ASTM C127. The physical qualities of flakiness and elongation index are connected to the geometry of aggregate fragments. High values cause reduced strength and anisotropic properties if used as aggregates in roadway and cement construction. The  $I_F$  and  $I_E$  value of the limestones vary around (12.20–1930) and (10.10–28.91), respectively, and are constrained by (BS 882 and 812:105. 2.) AIV is an indicator method for testing the compaction resistivity of an aggregate to rapid stress (Smith, Collis 2001). The range of LAV for these limestones is 14.08–25.12%, less than 40% as specified by ASTM C-131, which implies that these rocks can be used as road aggregate.

## 2.2 Methods

### 2.2.1 Alkali Silica Reaction

The accelerated mortar bar test (AMBT) is a technique employed by Oberholster and Davies at the National Building Research Institute in South Africa (1986). The proportion of cement to aggregates is 1:2.25, while the proportion of water to cement is 0.47. Casting mortar bars with the specified aggregate (coarse or fine) that has been treated to a defined gradation was part of the test. The concrete specimens were poured into (25x25x285) mm<sup>3</sup> cubic steel moulds in two equal levels with compacting each layer. The mortars bars were taken from their moulds after 24hrs and immersed in water at room temperature, which was then heated to 80°C in a drier for the next 24 hours where the mortar bars be stored at this temperature. After extracting the samples from water, they have been measured for length before becoming immersed in a 1N NaOH solution for 56 days at 80°C. Regular length change measurements were done during the storage period. Although different agencies define different expansion limits, most standards use the total expansion at the end of the 56 days soaking period. ASTM for example, have defined the following expansion requirements: ASTM C 1260 expansion criteria:  
< 0.10% is considered innocuous  
0.10 to 0.20% is considered potentially reactive  
> 0.20% is considered reactive

### 2.2.2 Alkali Carbonate Reaction

The concrete prism test (ASTM C 1105) is widely regarded as a much more fast and reliable method of estimating aggregate performance characteristics. This test uses concrete with a cement content of 420 kg/m<sup>3</sup>. The equivalent alkali concentration of the cement should be between 0.8 and 1.0%. The proportions of water and cement were 0.42 to 0.45. The concrete prisms have dimensions of (75x75x285) mm<sup>3</sup>. Molds made of cubic steel that have been cured for 24 hours at 23 °C and then stored at 38 °C over water. When examining plain concrete, measurements of expansion were conducted at periodic times with no chemical admixtures. Aggregates that increased by more than 0.04 percent in a year were referred as reactive aggregates.

## 3. Results and discussion

### 3.1 Alkali Silica Reaction (ASR)

#### 3.1.1 Limestone aggregate from El-Alameen area (L)

The limestone aggregates used in the concrete originated from the El-Alameen region (D). The composition and quantity of gel had an impact on the expanding forces carried on by its presence in concrete. It appears that time has an impact on the reaction products' creation. ASR gel is produced in greater volume during a longer exposure time, resulting in a broader expansion (Kawamura M. et al., 1998) and (E. Gavrilenko et al., 2007), while the gel's composition changes over time. The gel initially took in water but not Na<sup>+</sup> or K<sup>+</sup> ions. The gel became increasingly viscous and expanded over the initial alkali-rich gel as the reactions continued and the gel's Ca<sup>2+</sup> concentration increased. (Kawamura M. et al., 1998) found that elevated temperatures significantly increased the overall amount of Ca<sup>2+</sup> in the concrete gel. This was thought to be necessary in order to produce expanding gel. Also, they found no evidence that the alkali silica gel's Ca<sup>2+</sup> content had an impact on the gel's development rate. Alkali-silica reaction gel's viscosity flow was decreased by increasing the alkali content inside the gel. Alkali gels' viscosity may not be high enough to produce enough expanding force to crack aggregate concrete as a result.

Curing time, days	Mix notation	Linear expansion, %		
		L1	L2	L3
3		0	0.035	0.074
7		0.002	0.041	0.085
10		0.014	0.068	0.092
14		0.027	0.098	0.167
16		0.046	0.151	0.247
28		0.058	0.223	0.381
56		0.064	0.354	0.496

Table 4 Linear expansion values of (L) mixes cured in 1N NaOH up to 56 days  
4. táblázat 56 napig 1N NaOH oldatban tárolt (L) keverékek lineáris tágulás értékei

Table 4 shows that the samples were left in the solution for longer than ASTM C 1260 specifies, and Fig. 3 shows the data as a function of curing time up to 56 days. Since L1 did not exceed the allowed limits and generated improved results after 56 days, it was determined that L1 is the sample that is most resistant to high temperatures and soaking in sodium hydroxide.



L2 and L3 samples that started out performing poorly improved considerably and visibly as time passed on throughout the next 14 days. The generation of a huge volume of silica gel over an extended period of time and a correspondingly significant increase in length made Sample L3 the most extended sample.

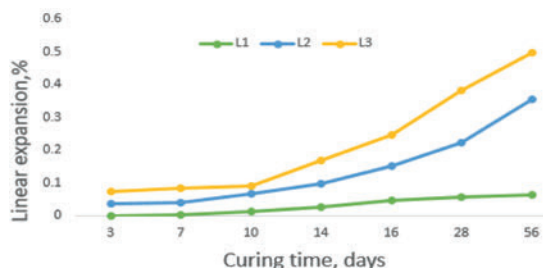


Fig. 3 Linear expansion values of (L) mixes cured in 1N NaOH up to 56 days  
3. ábra 56 napig 1N NaOH oldatban tárolt (L) keverékek lineáris tágulás értékei

### 3.1.2 Dolomitic stone aggregate from Attaqa area (D)

These aggregates were collected from the Attaqa region (A). The ASR products generated have different contents, according to experiments on materials that were soaked in various dissolved salts. KOH produced more crystalline gel, whereas NaOH was more aggressive and produced more gel. The expansion increased until a particular amount of alkalis was reached, at which point it continued to shrink as the concentration of alkalis increased. The linear expansion values of the aggregates taken from Attaqa (A) are listed in Table 5, which is displayed as a function of the curing time in Fig. 4.

Curing time, days	Linear expansion, %	
	D1	D2
3	0.018	0.072
7	0.021	0.079
10	0.038	0.093
14	0.041	0.158
16	0.056	0.228
28	0.083	0.401
56	0.094	0.479

Table 5 Linear expansion values of (D) mixes cured in 1N NaOH up to 56 days  
5. táblázat 56 napig 1N NaOH oldatban tárolt (D) keverékek lineáris tágulás értékei

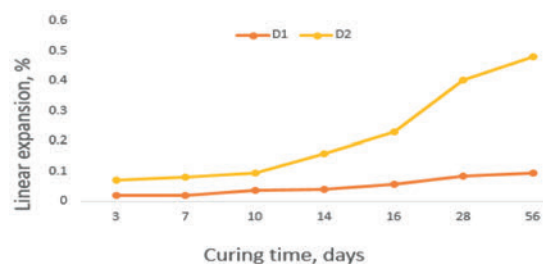


Fig. 4 Linear expansion values of (D) mixes cured in 1N NaOH up to 56 days  
4. ábra 56 napig 1N NaOH oldatban tárolt (D) keverékek lineáris tágulás értékei

From this area, two samples were chosen, with Sample D1 being the best because it included a good aggregate and had a lower proportion of porosity than the other sample. This resulted in the creation of a significant amount of calcium silicate hydrate and a reduction in the rate of expansion at the

age of 56 days. The limitations were actually strengthened by significantly expanding sample D3 due to the increased silica gel development, which has produced enough expanding force to crack concrete samples.

### 3.1.3 Products analysis (Scanning electron microscopy)

SEM micrographs of hardened specimens (D1) and (D2) after 56 days of curing are illustrated in Fig. 5. ASR gel appeared in cracks extending from reactive, fine-grained, dolomitic limestone coarse particles into the paste in plane-polarized transmitted light thin section electron microscopy. The ASR gel looked opaque in cross-polarized view, an unusual quality that typically sets it apart from other crystalline secondary deposits. The aggregates had a fine-grained calcite, clay, and quartz matrix with scattered, isolated dolomite rhombs as its main constituent. The dark gray, fine-grained dolomitic limestone coarse aggregates were the source of reactive silica for ASR. Si-rich phases in dolomitic limestone could exist locally in small regions and as extremely fine-grained, split silica particles intermingled with the rock matrix, in contrast to conventional alkali reactive siliceous rocks. Figure showed localized regions of cryptocrystalline quartz in the rock's matrix. As can be seen, silica occurred in two different ways: first, as relatively large particles, and second, as much smaller particles mixed up with the matrix. The finer particles were potentially reactive silica minerals, but the coarser ones were detrital, non-reactive quartz.

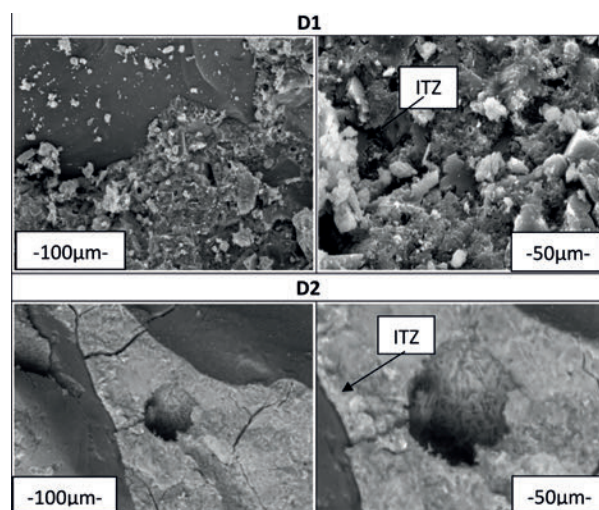


Fig. 5 SEM Photography of D1 and D2 immersed in 1N NaOH up to 56 days  
5. ábra 56 napig 1N NaOH oldatban tárolt D1 és D2 SEM felvételei

### 3.2 Alkali Carbonate Reaction (ACR)

#### 3.2.1 Limestone aggregate from El-Alameen area (L)

Limestone aggregates for concrete were collected from the El-Alameen region (L). Because of the interaction between the silica in limestones and the alkali components in OPC, an alkali silica gel has been created (Mustansar Naeem et al., 2019). The silica gel and calcite particles mixed together, which led to the aggregates' expansion. The gel that formed in the limestone also differed greatly. The sample seems to be larger and covers a significant portion of the surface. The linear expansion

values of concrete containing limestone are listed in Table 6 and shown as a function of curing time in Fig. 6. In comparison to the other samples, Sample L1 started with a high expansion at a curing age of 7 days. Even so, the sample increased till the age of curing of 360 days, with convergence in the length of the sample expansion after reaching a similar expansion at the age of 180 days. Without a certainty, the sample L2 showed that it had expanded gradually up to the age of 360 days. Although the later sample performed significantly better at age 7 days, the expansion was higher than that of sample L1. Despite being a reactive aggregate, sample L3 exhibited the same behavior as the other samples. The expansion of L3 became really clear at the age of 360 days of curing.

Mix notation Curing time, days	Linear expansion,%		
	L1	L2	L3
7	0.009	0.001	0.02
28	0.004	0.002	0.013
56	0.001	0.002	0.01
90	0.005	0.004	0.01
180	0.007	0.013	0.014
270	0.025	0.026	0.017
360	0.038	0.069	0.12

Table 6 Linear expansion values of (L) mixes cured over water up to 360 days  
6. táblázat 360 napig víz alatt tárolt (L) keverékek lineáris tágulás értékei

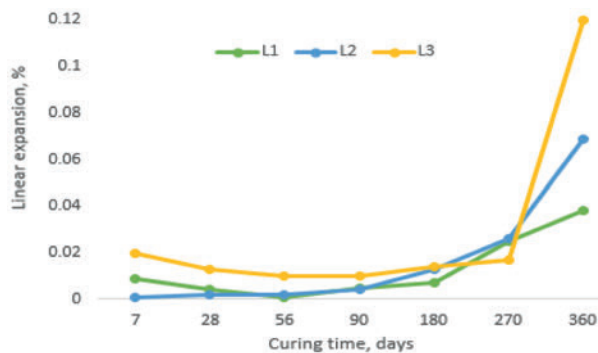


Fig. 6 Linear expansion values of (L) mixes cured over water up to 360 days  
6. ábra 360 napig víz alatt tárolt (L) keverékek lineáris tágulás értékei

### 3.2.2 Dolomitic stone aggregate from Attaqa area (D)

Dolomite aggregate for concrete was extracted from the Attaqa region (A). When the silica in the dolomite concrete mix reacted with the alkali component in Portland cement, an alkali-silica gel was created. As the gel expanded and absorbed water, the calcite particles were compressed together. Once the gel had stretched the calcite crystals together, they had become clearly visible (M. Ahmed et al., 2018). As a result, the concrete and aggregate particles both increased. The gel that formed in the aggregates was quite different. The sample seemed to be bigger and covered a substantial portion of the surface. Table 7 showed the linear expansion values of concrete containing dolomite aggregate, which are graphed as a function of curing time in Fig. 7. Sample D1 expanded more over the duration of 7 days to 56 days before decreasing at 90 days of curing and then expanding once more over the next 360 days till it reached the critical zone. After the age reached 360 days, the sample D2 expansion's length didn't stop expanding, indicating

that it was still active. The sample's porosity increased as a result of its expansion, increasing water absorption and lowering the sample's mechanical characteristics.

Mix notation Curing time, days	Linear expansion,%	
	D1	D2
3	0.018	0.072
7	0.021	0.079
10	0.038	0.093
14	0.041	0.158
16	0.056	0.228
28	0.083	0.401
56	0.094	0.479

Table 7 Linear expansion values of (D) mixes cured over water up to 360 days  
7. táblázat 360 napig víz alatt tárolt (D) keverékek lineáris tágulás értékei

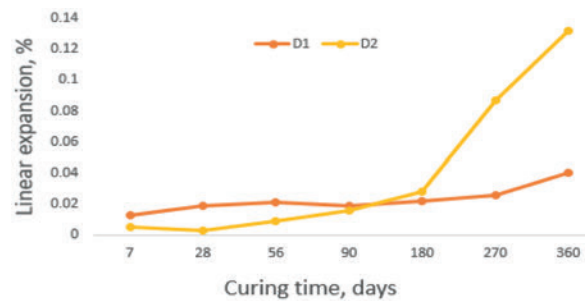


Fig. 7 Linear expansion values of (D) mixes cured over water up to 360 days  
7. ábra 360 napig víz alatt tárolt (D) keverékek lineáris tágulás értékei

### 3.2.3 Products analysis (Scanning electron microscopy)

Polarizing microscopy revealed that ACR produced a micrographic texture within the reaction ring that was made up of brucite and calcite spots.

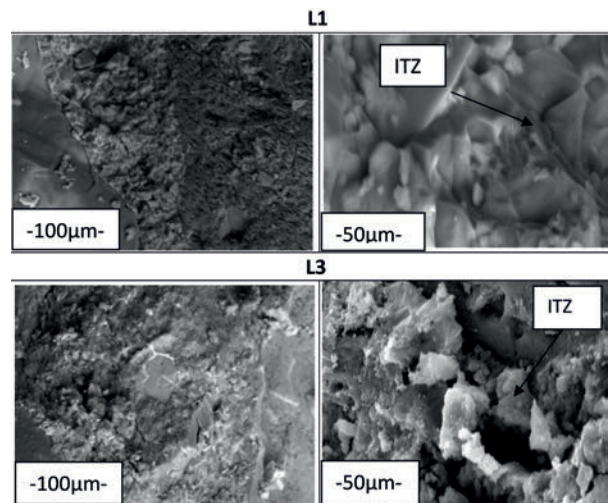


Fig. 8 SEM Photography of L1 and L3 cured over water up to 360 day  
8. ábra 360 napig víz alatt tárolt L1 és L3 SEM felvételei

As seen in Fig. 8, SEM micrographs of hardened specimens (L1) and (L3) after 360 days of curing are plotted, aggregates with limestone-rich patches and cracks after reaction was chosen for observation using SEM and element materials mapping analysis in order to further explore the reason of expansion stress generated by alkali-carbonate reaction and

showed a large number of rod-shaped crystals next to the calcite. Calcite and brucite were the byproducts of the reaction between dolomite and alkali in aggregate. Because some of the dolomite had not completely reacted during the dedolomitization process, it is important to note that unreacted dolomite is still present in aggregate particles. Calcite ( $\text{CaCO}_3$ ) was generated by ACR when the region was primarily made up of Ca, C, and O components. Moreover, when the region is primarily made up of Mg and O components, ACR will generate brucite ( $\text{Mg}(\text{OH})_2$ ). Expansion strain caused by the accumulation and development of brucite crystals, and the aggregate then went through an alkali-carbonate reaction to create expansion.

## 4. Conclusion

The goal of this study was that comparing between the concrete prism test (alkali carbonate reaction) over a 360-day period and the accelerated mortar bar test (alkali silica reaction) at 56 days. Five aggregate samples have been collected from the El-Alameen and Ataqaa areas. They were composed primarily of dolostone with some limestone. In comparison to the other samples, which were taken from the El Alameen region, Sample L1 demonstrated the least linear expansion of the accelerated mortar bar test (alkali silica reaction) at 56 days of curing and the least linear expansion of the concrete prism test (alkali carbonate reaction) at 360 days of curing. In every test, including the concrete prism test (alkali carbonate reaction) and the mortar bar test (alkali silica reaction), Sample D1 performed better than the other samples from the Ataqaa area. Mortar and concrete composed of limestone have an effect on the resistance of sodium hydroxide and reduce the production of silica gel than that composed of dolostone. The accelerated mortar bar test AMBT was time-efficient and comparable to the alkali carbonate reaction (ACR), which was shown to be more indicative of particular issues. The alkali carbonate reaction ACR succeeded in its objective, as shown by the accelerated mortar bar test AMBT. As was indicated before, the ages of 270 and 360 days in the concrete prism corresponded to the ages of 16, 28, and 56 days in AMBT. Because AMBT is utilized in the Egyptian Code for Laboratory Tests manual and is valid in all situations, it is assumed based on these earlier findings that AMBT fits and matches ACR.

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