Alkali-aggregate reaction in alkali-activated cement concretes

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Abstract. The paper discloses the results of a study on structure formation processes in the interfacial transition zone “cement stone – aggregate” of the alkali-activated cement concretes. The results indicate the importance of the content of Al₂O₃ in the cement and aggregate. Together with components that are able to interact actively with alkalis in the presence of reactive SiO₂, the processes taking place during the alkali-aggregate reaction can have weather destructive or constructive effect. The constructive effect is caused by incorporating the corrosion products into alkaline aluminosilicate compounds, thus retarding the internal corrosion of the concrete and providing the higher durability even in case of alkali-activated cements and alkali-susceptible aggregates with relatively high alkali content.

1. Introduction

Internal corrosion of concrete resulted from alkali-aggregate reaction (AAR) is one of the less studied and “hidden” reasons of reduction of durability. The present study was dedicated to this issue with a final goal to develop methods of regulation of the reaction “alkali–silicic acid” and technological recommendations on how to prevent the concrete from this corrosion in order to meet the norm-specified requirements as to durability of buildings and structures. Corrosion of concrete as a result of reaction between alkalis of the cement and silica of the aggregate, is, as a rule, a long-term process. Sometimes, in the process of inspection of the structures this type of corrosion is identified in a wrong way, since its external signs (features) are similar to those from the processes of frost destruction and corrosion induced by aggressive environment.

The most widely known explanation of the reasons for AAR-induced corrosion is an interaction of the alkali metal compounds (of sodium and potassium) with active silica of the aggregate [1]–[4]. An alkali can be brought into a concrete with the cements containing increased quantities of sodium and potassium oxides (up to 1...2% by mass calculated as Na₂O-equivalent). A quantity of the alkalis brought into the concrete can be increased as a result of the use of chemical, mineral and organo-mineral admixtures and additives containing alkalis [5]–[7]. The alkalis also can come from outside, for example, with sea water or deicing reagents [8], [9]. Alkali-activated cements (AAC) like slag alkali-activated cements, alkali-activated portland-, slag portland-, pozzolana cements, geocement-based bonds, etc., become more common in construction industry while the content of alkalis (expressed as Na₂O) could reach between 1.5 and 5% by mass [10]–[14]. As a result of interaction of alkalis with active silica of the aggregates the hydration products causing deformations, strength
decline and deterioration of concrete are formed on the surface of the interfacial transition zone (ITZ) “aggregate – cement stone” [15]. In these cases, a network of cracks appears on the surface of the concrete followed by full destruction of the concrete [16], [17].

The compressive strength of the concrete does not guarantee durability of concrete by itself [18]–[20]. High durability is guaranteed, first of all, by high compaction of the concrete as well as by composition of the hydration products, both the cement stone itself, as well as hydration products in the ITZ “aggregate – cement stone” [21]–[23].

In Figure 1 is a classical schematic representation of the AAR in case of alkali-susceptible aggregates (ASA) with damage of the ITZ “aggregate – cement stone”. A gel-like shell, due to accumulation in it of the reaction products, tends to increase in volume, initiating the occurrence in the concrete of the expansion deformations and in certain conditions can result in damage of the concrete structures. Moreover, the gel-like CSH- shell acting as a semi-permeable membrane can allow for the water and alkali to penetrate and reach the aggregate grain. As a result of this, the osmotic pressure is produced in the ITZ of the aggregate with the cement stone which can reach rather high values. Since, for example, the osmotic pressure of the sodium silicate gel may reach 20 MPa and tensile strength of the concrete is only 3-5 MPa, this may result in the occurrence of micro cracks and gradual deterioration of the concrete [24]–[26].

As a result of the wider use in the practice of construction of the AAC, theoretical background of which has been developed by V.D.Glukhovsky and which are being developed by his followers [27], durability of concrete in case of the ASA is of first priority and concern.

As a result of the mechanism of hydration and hardening of the AAC which are completely different from those of the OPC, the processes of interaction of the environment of the hardening AAC stone with reactive aggregates differ in principal from those taking place in case of OPC [26], [28]. In particular, an effect of “encapsulation” of the grains of the ASA with a film composed from hydration products, which retard, though not always completely exclude, the corrosion process.

The present research study resistance of the aggregates from alkali-susceptible rocks in the environment of the cement stone using different types of cements, including AAC, and to assess the degree of influence of the cement matrix and different additives on the rate of reaction between the ASA and the alkali of the cement matrix as well as to reveal how it may be reflected on service

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**Figure 1.** Schematic representation of the alkali-silicic acid reactions in the OPC stone [6]: 1 – formation of alkali-metal hydroxides; 2 – diffusion of ions to place of reaction; 3 – ion-exchange and ion reaction; 4 – water absorption and swelling.
properties and durability of the concrete structures. The following factors which affect the “internal corrosion” of the concrete have been taken into account: aggregate type, aggregate particle size, quantity of alkali and type of alkaline component, cement type, type of modifying active additive and its quantity. The influence of each factor taken separately and of combination of factors was studied.

2. Materials and Methods
The following cements were used in the research on behavior of ASA:
- OPC CEM II 42.5N A-S (Na₂O-equivalent = 0.22% by mass);
- OPC CEM II 42.5N A-S with the increased content of Na₂O = 1.3% by mass;
- slag alkaline (alkali activated) cement (Type LCEM I, strength Grade 42.5 [11]) with Na₂O-content = 2.5% by mass;
- alkaline (alkali activated) slag portland cement “OPC clinker:slag”= 40:60 (Type LCEM IV, strength Grade 42.5 [11]) with the Na₂O-content = 2.5% by mass.
Anhydrous soda ash was used as Na₂O equivalent. The soda ash was introduced in a dry state in the process of joint grinding of cement components. In order to compare influence of alkaline component on cement characteristics, in particular crystallites content, the cement to aggregate ratio was 1:3. The potentially susceptible aggregates examined were aggregates from natural rocks with characteristics given in Table 1. Quartz crystallites content was set according to complex (quantitative and qualitative) XRD analysis.

| Origin      | Rock type   | SiO₂ | Al₂O₃ | TiO₂ | Fe₂O₃ | FeO | CaO | MgO | Na₂O | K₂O | Other | Acidity modulus | Glassy phase wt.% |
|-------------|-------------|------|-------|------|-------|-----|-----|-----|------|-----|-------|-----------------|-------------------|
| Austria     | Olivine rock| 44.99| 15.56 | 2.25 | 10.53 | 10.42| 8.98| 2.42| 2.4  | 0.10| 3.06  | 5-7              |                    |
| Ukraine     | Basalt rock | 50.42| 12.47 | 2.48 | 9.45  | 8.5 | 9.34| 5.74| 2.22 | 0.92| –     | 17-19            |                    |
| Ukraine     | Andesite rock| 54.16| 18.03 | 0.43 | 9.39  | 8.57| 4.65| 1.06| 2.06 | –   | 5.46  | 39-41            |                    |

The ASA were used as fine gravel of polyfractional composition with the particle size varying between 0.5 and 8 mm. A river quartz sand with a grading factor M=1.2 was taken as aggregate in the preparation of the reference specimens. The cement to aggregate ratio was 1:3. W/C ratio was in range from 0.39…0.4. In order to suppress destructive processes in the ITZ the metakaolin was added in a quantity of 15% by mass of the cement.

Prismatic specimens 4×4×16 cm and 2.5×2.5×28.5 cm with copper benches (for control of deformations) were prepared for flexural and compressive strength test. After 2 days the specimens were taken from the molds and placed for long-term steam treatment for 28 days (T=70°C, RH=100%) – accelerated procedure [25], [28] with test ages at 14 and 28 days. In compliance with the accelerated procedure used in the present study the aggregates can be classified after 28 days of continuous steam treatment with regard to suitability (conformance) for the use in concretes as the following:

a) expansion less than 1.5 mm/m – an aggregate is not alkali-susceptible one (no need for further testing);

b) expansion between 1.5 and 2.0 mm/m – an aggregate is alkali-susceptible one (preventive measures should be taken to reduce effect of reactive silica);

c) expansion higher than 2.0 mm/m – an aggregate is alkali-susceptible one and is not allowed for the use in concrete (alternative aggregates should be recommended).

A measurement was taken preliminary on the specimens intended for control of deformations. The measurements of deformations were done using a digital device with a value of the scale division of 0.01 mm. The ITZ was studied using a digital microscope on thin sections of the specimens.
3. Results and Discussion
Initially, the size of the quartz crystallites present in the aggregates was measured to determine aggregate reactivity. Determination was done using three characteristic peaks of quartz: \(d=0.426(8); 0.334(10); 0.182(6)\) nm. The results are given in Figure 2. An evident fact is the less size of crystallites, the higher solubility, hence the reactivity, may be expected [5], [12]. Thus, coming from the size of quartz crystals in the rocks under study, in reactivity the rocks can be arranged as the following: andesite rock (65.7 nm) > basalt rock (114.1 nm) > olivine rock (124.8 nm).

![Figure 2](image)

**Figure 2.** The size of the quartz crystals in different rocks.

3.1. OPC-based concretes
The presence of alkali (\(\text{Na}_2\text{O} \leq 0.6\%\) by mass) in the cement composition is not dangerous as no expansion was found in all test specimens (Figure 3). Thus, there is no need to apply technological measures or use additives or admixtures. As it follows from Figures 3b and 4a, the increase in the \(\text{Na}_2\text{O}\) content in the OPC up to 1.3% by mass accelerates the destructive processes in the ITZ and in case of the andesite rock results in critical expansion exceeding 2.0 mm/m (+2.43 mm/m). The signs of corrosion are clearly seen in the microphotos of the ITZ (Figure 4a). The expansion of other compositions is found to fall within the admissible limits.

The introduction of the metakaolin additive is found to allow the use of the andesite rock as well (Figure 3b), the ITZ being clear and free from the products of corrosion (Figure 4b).

![Figure 3](image)

**Figure 3.** a) Deformations of the concretes made with ASA: a) OPC CEM II 42.5N (\(\text{Na}_2\text{O} = 0.22\%\) by mass). b) OPC with the increased content of alkali (\(\text{Na}_2\text{O} = 1.3\%\) by mass).
Figure 4. The ITZ “andesite aggregate-cement stone” in case of OPC (Na₂O=1.3% by mass); a) without the metakaolin; b) with the metakaolin; 1 – cement stone; 2 – ITZ; 3 – alkali-susceptible aggregates.

3.2. Slag alkali-activated cement-based concrete and alkali-activated slag portland cement-based concrete

In case of the slag alkali-activated cement all specimens without the additive (with the exception of the reference one) showed the expansion from +0.12 to +0.316 mm/m, what is not a critical one (Figures 5 and 6). The metakaolin additive allowed to reduce further the expansion (−0.14...+0.01) mm/m (Figure 7). No any products of corrosion induced by AAR were detected in the ITZ (Figure 7, a, b).

Figure 5. Deformations of the compositions made with ASA: a) slag alkali-activated cement (Na₂O = 2.5% by mass). b) alkali-activated slag portland cement (Na₂O=2.5% by mass) Type LCEM IV “OPC clinker:slag”= 40:60.
Figure 6. Interfacial transition zone “andesite – cement stone” (slag alkali-activated cement (Na$_2$O=2.5% by mass): а) without the metakaolin; b) with the metakaolin; 1 – cement stone; 2 – ITZ; 3 – alkali-susceptible aggregate.

A behavior of the ASA in the concretes based on alkali-activated slag portland cement (Figures 6b and 7) is somewhat similar to that of these aggregates in the presence of OPC with the increased content of Na$_2$O (Figures 3 and 4), but with the higher values of expansion. The largest expansion was reported of the specimen with the andesite aggregate without the additive ±2.91 mm/m, thus much exceeding the maximal admissible limit. The specimens prepared using other aggregates under study showed the expansion within the range from +0.36 to 0.947 mm/m.

Such sensitivity of the andesite aggregate can be attributed to the presence in both cases of Ca$^{2+}$ ions in rather large quantities except for the Na$^+$ ions and to the higher content of active silica in the andesite aggregate – of around 40% by mass (Table 1).

Figure 7. The interfacial transition zone “andesite aggregate – cement stone” in case of alkali-activated slag portland cement (Na$_2$O = 2.5% by mass). a) without the metakaolin; b) with the metakaolin; 1 – cement stone; 2 – ITZ; 3 – alkali-susceptible aggregate.
A reason of such “favorable (positive)” behavior of the ASA in case of slag alkali-activated cement may be attributed to the presence in the finely ground slag side by side with amorphous SiO$_2$, active Al$_2$O$_3$ in sufficient quantities, thus creating conditions for synthesis of the alkaline zeolite-like hydro aluminosilicates represented by a formula – (Na$_2$,Ca)O·Al$_2$O$_3$·mSiO$_2$·nH$_2$O.

This, first of all, enables to bind/attract excessive quantities of alkali into insoluble or poorly insoluble hydration products, and secondly, promotes the formation around grains of the alkali-susceptible aggregates of almost impermeable and strong shell, which prevents the alkaline ions from diffusion to the aggregate and following development of destructive processes in the ITZ “cement stone – ASA”. This also enables to enhance adhesion between the cement stone and aggregate. The introduction of active alumina in a form of metakaolin gives further increase of this effect (Figure 3, 5).

3.3. Effect of size of grains of the aggregates and type of alkaline component

A sensitivity/ susceptibility of the aggregates in question to the AAR depending upon size of their grains were studied. The results are given in Figure 8a. A conclusion was made that the grains of size from 1.25 to 5 mm had the largest sensitivity to alkali.

![Figure 8](image)

**Figure 8.** Expansion deformations of the concrete made with alkaline slag portland cement; a) The effect of particle size of the andesite aggregate; b) the effect of type of alkaline component of expansion deformations of the concrete.

An influence of type of alkaline component on deformation of expansion is given in Figure 8b. A conclusion was made that the concretes in which sodium metasilicate was used showed somewhat low shrinkage values compared to those of the concretes made with the soda ash. Evidently, this may be attributed to the presence in the metasilicate of active SiO$_2$, which is capable even at early stages of hydration to bind partially free calcium of the cement, thus resulting in the formation of not enough dense semi-permeable membrane composed of the CSH-gel and reduction of the quantity of the sodium silicate gel in the ITZ “cement stone – ASA”.


3.4. Effect of quantity of metakaolin additive
A full-factor experiment was used to assess effect of quantity of the metakaolin additive and alkali on the rate of reaction “alkali – silicic acid”. An adequate equation of regression was produced as a numeral model. The results are given in Figure 9. A conclusion can be drawn that increase in quantity of the alkali in the concrete and decrease in quantity of the metakaolin additive are proportional to the growth of expansion deformations, that is, development of degradation of the ITZ. If, coming from recommendations given in [26], an expansion value below 1.5 mm/m to accept as a safety value, then, with the typical content of alkalis in the alkali-activated cements of 2.5…4% by mass (as Na₂O), introduction of the metakaolin additive in a quantity from 7 to 10% by mass seems to be an adequate measure.

![Figure 9. Isolines of expansion deformations, mm/m, vs quantities of the metakaolin additive and alkali content of the cement. Cement – alkali-activated slag portland cement (OPC clinker:slag=40:60); aggregate – andesite rock.](image)

3.5. Mechanism of AAR-related corrosion
In case of the soda ash as an alkaline component, the reactions in the ITZ can evolve as the following:

\[ \text{Na}_2\text{CO}_3 + \text{Ca(OH)}_2 \rightarrow \text{CaCO}_3 + 2\text{NaOH} \quad \text{(Na}^+\text{, OH}^-) \quad (1) \]

\[ 2\text{NaOH} + \text{SiO}_2 + \text{H}_2\text{O} \rightarrow \text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot \text{nH}_2\text{O} \quad \text{(gel of alkali metal silicate)} \quad (2) \]

Under condition of large quantities of calcium side by side with sodium (that is, in case of OPC and slag portland cement), the reaction can evolve further:

\[ \text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot \text{nH}_2\text{O} + \text{Ca(OH)}_2 \rightarrow 2\text{NaOH} + \text{CaO} \cdot \text{SiO}_2 \cdot \text{nH}_2\text{O} \quad \text{(CSH-gel)} \quad (3) \]

The gel of alkali metal silicate itself is not able to produce an expansion pressure. It only breaks the contact “cement stone – aggregate”, what is not good, but yet not critical. The presence of calcium enables to synthesize a dense gel-like hydro silicate of calcium (CSH), which is present in the outer
part of the reaction zone and acts as a semi-permeable membrane to permit access of new portions of water, ions of Na+ and OH– to the place of reaction, but not allowing removal of the reaction products and thus leading to the increase of pressure in the ITZ “cement stone – ASA” (see Figure 1). Besides, an alkali released in the end of reaction, takes part in the deterioration of the deeper layers of a grain of the ASA. That’s why, finally, the available quantities of Ca(OH)2 can be, to some extent, a restricting (limiting) factor for damping the reaction. This may explain the reasons of more breaking destructive effect in the presence of excessive quantities of not only the Na+-ions, but of the Ca2+ -ions compared to action of solely Na+-ions.

Thus, a conclusion can be drawn that formation in the ITZ of the silicate gel of alkali metal or alkali-earth metal taken each separately is not able to produce a breaking pressure and result in degradation of the ITZ. Of danger is a combination of these reaction products and their combined action.

The metakaolin additive allowed to reduce an expansion of the specimens made with the andesite aggregate from a dangerous (+2.91 mm/m) to admissible level (+0.719 mm/m) (Figure 5). The expansion of other specimens with the metakaolin additive was found to be not appreciable one (under the limits from +0.063 to +0.193 mm/m).

A scheme of reaction “alkali – silicic acid” with so-called “positive effect of corrosion” in AAC stone is given in Figure 10. An active alumina can get into the AAC composition together with active mineral substances such as granulated blast-furnace slag, fly ash, and metakaolin. A gel of the alkali metal (Na2O·SiO2·nH2O-gel), formed as a result of reaction between the alkali and ASA, is an intermediate product and a supplier for synthesis of the zeolite-like (Na2O·Al2O3·mSiO2·nH2O) dense, strong (solid) and impermeable shell around the aggregate which stops further development of the destructive reaction.

**Figure 10.** Schematic representation of reaction “alkali-silicic acid” with “positive” effect of corrosion in the alkali-activated cement stone: 1 – diffusion of ions from the alkaline activator solution to the place of reaction; 2 – metakaolin; 3 – ion-exchange and ionic reaction.

4. **Conclusions**

The results of study allowed to demonstrate a possibility to use the alkali-susceptible aggregates (ASA) in the concretes made from the alkali-activated cements (AAC). The introduction of the active alumina into the compositions of the AAC had a positive effect allowing to keep the structure formation processes in the interfacial transition zone “cement stone – ASA” under control by reducing
the expansion deformations down (below 1.5 mm/m) to admissible levels or even completely suppress them. To reach a safety value of expansion limit for the alkali-activated cements (with 2.5…4% Na₂O₉), it was found that introduction of the metakaolin additive in a quantity from 7 to 10% by mass seems to be an adequate measure. It was shown that use of soda ash is more dangerous for alkali-aggregate reactions comparing to the compositions with sodium metasilicate. Andesite rock seams to be the most active type of ASA and the most active fraction of aggregate is 0.315/5.0 mm. The following studies will be directed on developments and investigation of properties of concrete compositions using ASA.

Acknowledgement
The research leading to these results has received the funding from the State budget of Ukraine (project 4DB-2019).

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