Restriction of Cl⁻ and SO₄²⁻ Ions Transport in Alkali Activated Slag Cement Concrete in Seawater

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**Abstract.** The relevance of alkali activated slag cement (AASC) concretes for structures operated in seawater is due to their enhanced resistance in aggressive environments. The application of high consistency fresh concretes is accompanied by negative changes in their structure with higher penetration of aggressive environments. Thus, the methods to prevent corrosion of steel reinforcement in plasticized AASC concrete are actual for investigations. It is shown, that modification of plasticized AASC concrete (consistency class S4) by the complex «alumina cement - portland cement - clinoptilolite - trisodium phosphate (Na₃PO₄·12H₂O)» restrict the transport of aggressive Cl⁻ and SO₄²⁻ ions. The results of DTA, XRD, electron microscopy, microzond analysis show that mentioned complex limits transport of the mentioned aggressive ions due to their binding by AFm phases in hydration products, exchange with OH⁻ ions in the structure of clinoptilolite, formation of hydrated products of apatite group Ca₃(PO₄)₂(OH, Cl). This was confirmed by qualitative reaction on Cl⁻ and SO₄²⁻ ions in concrete structure, as well as by assessing of surface and mass loss of steel bars embedded in AASC concrete after 9 months in seawater. It was ensured the advanced crystallization with densification of microstructure, which increases corrosion resistance of artificial stone.

1. **Introduction**

Current trends in construction engineering predetermine application of binder materials alternative to portland cement. It’s well-known, that production of 1 ton of portland cement requires 1.5 tons of raw materials and is accompanied by 900 kg of CO₂ emission [1]. World cement production causes 6…8 % of total CO₂ emission and consumes of 12…15 % of total industrial energy [2]. Thus, production of low-emission cements, obtained by partial replacement of OPC clinker with additives, is demanded [3-6].

Alkali-activated slag cement (further, AASC) is the most perspective one in view of effective consumption of raw resources and energy as well as responsible attitude to environment. The ecological benefits of AASC are caused not only by reduction of CO₂ emission due to application of by-products as well as waste products [7, 8, 9], but also by possibility to utilize manufacturing waters in safety building materials [10]. AASC materials are characterized by advanced strength [11], heat resistance [12], corrosion resistance [13, 14] and freeze-thaw resistance [15] in comparison with analogues based on traditional clinker cements.

The benefits of AASC cause the effectiveness of concrete constructions which are exploited in under the influence of seawater (foundations, berths, piers, coast-protecting structures, dams etc.). Durability of AASC concrete in such conditions was confirmed [16, 17].

Peculiarities of AASC hydration products cause increased protective properties of AASC concretes to steel reinforcement in comparison with portland cement. It’s well-known, that the aggressive ions can be chemically adsorbed by hydrosilicates C–S–H and hydroaluminosilicate C–A–S–H gel phases at the early stages of AASC hydration [18, 19]. High content of gel-like low-calcium hydrosilicates causes advanced initial binding if compare with portland cement [20, 21]. Moreover, occlusion of Cl⁻ and SO₄²⁻ ions by alkaline aluminosilicates, which are analogues of natural zeolites, takes place [7]. Mentioned minerals form at late stages of structure formation [7, 22] and can be characterized both as cationites and anionites [23].
Advanced protection of AASC concrete to steel reinforcement is also caused by presence of alkaline component which acts as cathode corrosion inhibitor [24, 25].

Durability of AASC concretes, including reinforced concretes, which are obtained from harsh (low consistency) fresh mixes, was already proved by long-term exploiting experience [7, 8, 9]. However, the modern requirements to high consistency fresh concretes are governed by practice. This way, the increasing of porosity and, consequently, permeability can be caused by changes in hardened concrete structure. This causes the increasing transport of aggressive ions in AASC concrete and risk of disturbance of reinforcement passive state.

Thus, corrosion of steel reinforcement under action of chlorides and sulfates due to their penetration in concrete structure is the main problem for structures, which are exploited in seawater. There are two main processes during corrosion action on steel reinforcement: carbonation and pitting corrosion, caused by chloride-ions [26]. The passive state of steel reinforcement is provided at molar ratio of Cl-/OH ≤ 0.6 in pore solution [27, 28]. In one's turn, sulfate-ions don’t cause immediate depassivation of steel reinforcement, but determine formation of hydrogen sulfide (H₂S) and catalyze oxidation (carbonation) of hydrate phases.

Formation of AFm phases (Al₄O₇·Fe₂O₃-mono) was proposed to enhance protective properties of AASC concrete, which is obtained from high consistency fresh mix, to steel reinforcement. It’s well-known, that these phases can chemically bind anions (Cl⁻, SO₄²⁻, CO₃²⁻, OH⁻ etc.) [30]. The effectiveness of mentioned mean was confirmed by work [31]. The AFm phases form at early hydration and are characterized by greater stability comparing to AFt phases (ettringite) while increasing alkalinity of hydration medium [32].

Thus, application of calcium aluminate cement and portland cement as additives is reasonable for formation of highly-calcium hydroaluminates 3CaO·Al₂O₃·10H₂O [33], which ensures binding of Cl⁻ and SO₄²⁻ ions in AFm phases, known as Friedel’s salt 3CaO·Al₂O₃·CaCl₂(SO₄)·10H₂O [30, 34] and Kuzel’s salt 3CaO·Al₂O₃·0,5CaCl₂·0,5SO₄·10H₂O [35] etc.

Application of aluminosilicate ionite such as clinoptilolite (zeolite) in AASC concrete can restrict transport of aggressive ions. Admixture of clinoptilolite enhances occlusion function of alkaline hydroaluminosilicates (analouges of natural zeolites), formed during AASC hydration [23].

Furthermore, application of trisodium phosphate Na₃PO₄·12H₂O (further, TSP) was proposed to enhance the inhibitory action of AASC alkaline component. TSP is the most widespread mixed corrosion inhibitor [36, 37]. Inhibitory effect of TSP is caused by dense protective film presented by iron phosphates FePO₄ and FePO₄·2H₂O on steel surface [38], increasing polarization resistance of steel [39] as well as due to formation of chemical stable products like hydroxyapatite Ca₁₀(PO₄)₆(OH)₄ [40]. Formation of crystal hydroxyapatite causes densification of structure due to filling of micropores microcracks in AASC concrete to decelerate diffusion of aggressive Cl⁻ and SO₄²⁻ ions [38]. At that, hydroxyapatite can bind Cl⁻ ions in chlorapatite Ca₆(PO₄)₃Cl to decrease their content in porous solution, also while their transport from aggressive medium [41]. Effectiveness of TSP in complex with surfactants (sodium lignosulphonate, sodium gluconate) as well as calcium-containing additive (OPC clinker) for prevention of steel corrosion was shown [42]. Complex of additives (further, CA) based on salts of strong acids ensures mutil-effects: reduction of water content, acceleration of crystallization, alterations in morphology of hydrated phases [43, 44].

It can be predicted advanced protective properties of AASC concrete, which is obtained from high consistency mixes and exploited in seawater, to steel reinforcement, due to application of CA which ensures chemical adsorption of Cl⁻ and SO₄²⁻ ions by gel-like hydration products (C-S-H and C–A–S–H), chemical binding (AFm phases abd hydroxyapatite) as well as occlusion (zeolite-containing components and zeolite-like alkaline hydroaluminosilicates). Proposed CA acts as corrosion inhibitor as well.

Thus, the aim of this research was to investigate the possibility of restriction Cl⁻ and SO₄²⁻ ions transport in plasticized AASC concrete, which is exploited in seawater, due to modification by CA including portland cement, calcium aluminate cement, TSP and zeolite.
2. Raw materials and testing techniques

Ground-granulated blast furnace slag (further, GBFS) (CaO – 47.30 %; SiO₂ – 39.00 %; Al₂O₃ – 5.90 %; Fe₂O₃ – 0.30 %; MgO – 5.82 %; SO₃ – 1.50 %; TiO₂ – 0.31 %), basicity modulus= 1.11, content of glass phase= 84.0 %, specific surface= 450 m²/kg (by Blaine), was used as aluminosilicate component of AASC.

Polyorganohydridosiloxane (liquid 136-41) was used for milling intensification of GBFS and stabilization of AASC properties. Content of admixture was 0.1 by mass of GBFS.

Alkaline components of AASC were presented by:
- soda ash (Na₂CO₃), dry state;
- five-water sodium metasilicate (Na₂SiO₃·5H₂O), dry state.

Two reference compositions of AASC were used:
- based on soda ash (GBFS – 93.50 %, soda ash – 6.50 % (3.80 % by Na₂O));
- based on sodium metasilicate (GBFS – 88.50 %, sodium metasilicate – 11.50 % (3.36 % by Na₂O)).

The AASC was also modified by CA, which components were presented by:
- portland cement CEM I 42.5 R (PJSC Ivano-Frankivskcement, Ukraine);
- cement aluminite cement ISTRA 40 (HeidelbergCement, Germany);
- TSP (Na₃PO₄·12H₂O) according to CAS № 7601-54-9
- natural zeolite (clinoptilolite) powder (by mass, %: SiO₂ – 72.5, Al₂O₃ – 13.1, Fe₂O₃ – 0.9, TiO₂ – 0.2, CaO – 2.1, MgO – 1.07, P₂O₅ – 0.003, K₂O+N₂O – 5.03), fr. 0 - 0.1 mm, content of clinoptilolite ≤ 93.0 %, porosity 54.0 % (JSC Zeolite-Bio, Ukraine).

Content of CA was 15.00 % by mass of AASC. Contents of CA components, %: (portland cement + calcium aluminite cement) – 5.00, clinoptilolite – 5.00, TSP – 5.00. The ratio between portland cement and calcium aluminite cement was like 2.17:1.00 to form 3CaO·Al₂O₃·10H₂O.

Surfactant was presented by sodium lignosulphonate according to CAS 8061-51-6 (pH ≥ 8.5), dosage 0.8 by mass of AASC.

The standard quartz sand according to EN 196-1 was used in AASC fine aggregate concretes (ratio AASC to sand = 1:3).

Aqueous solution of salts, which simulates 99.90 % of ones in seawater, was used in investigations. Composition of aqueous solution, % by mass of mixture: NaCl – 78.70, MgCl₂ – 9.80, MgSO₄ – 5.76, CaSO₄ – 3.75, KCl – 1.73, CaCO₃ – 0.29. Total concentration of the salts was 35 g/l.

Normal consistency AASC pastes were mixed with seawater for simulation of chemical binding of Cl⁻ and SO₄²⁻ ions by AASC hydrate phases, as it takes place in contact zone of concrete surface with aggressive medium. Cement pastes were prepared in Hobart mixer. Consistency of AASC pastes was determined according to the national standard of Ukraine DSTU B V.2.7-185:2009. Monitoring was carried out by X-ray diffraction (XRD), differential-thermal analysis (DTA) and electronic microscope with microanalyzer.

Fresh concretes were prepared in mixer «Raimondi Iperbet» (Italy). Consistency (workability) was determined by cone slump according to the national standard of Ukraine DSTU B V.2.7-114:2002.

Performances of AASC concrete were determined on specimens 40x40x160 mm. Strength of AASC concrete was determined according to the national standard of Ukraine DSTU B V.2.7-187:2009. Transport properties of AASC concrete, regarding penetration of Cl⁻ and SO₄²⁻ ions, were evaluate after 28 d of hardening in normal conditions (t=±20 °C, R.H.=95±5 %) and further 270 d of storing in seawater.

Penetration depth (transport) of aggressive ions was determined by qualitative reactions due to spraying of aqueous solution of corresponding salts on surface of AASC concrete specimens right away after their breaking in half. Penetration depth of chlorine-ions was determined by white sediment of silver chloride, which was formed as a result of spraying of AgNO₃ solution (concentration 0.1 N). Penetration depth of sulfate-ions was determined by white sediment of lead sulfate, which was formed as a result of spraying of Pb(CH₃COO)₂ solution (concentration 0.5 N).

Corrosion resistance coefficient (K_cr) of AASC concrete was determined as ratio of tensile strength in bending of specimens after storing in seawater to tensile strength in bending of specimens after storing in fresh water. According to DSTU B V.2.7-288:2011 concretes can be classified as unresistant
(K_cr < 0.3), low-resistant (0.3 < K_cr < 0.5), resistant (0.5 < K_cr < 0.8) and high-resistant (K_cr > 0.8) ones in corrosion mediums.

The state of steel reinforcement in plasticized AASC concrete was estimated according to following method. The basic steel rebarS, length 120 mm and diameter from 4.1 mm to 4.3 mm, were embedded in specimens 40x40x160 mm of AASC concrete. These rebars were degreased by acetone and weighted with accuracy of ±0.001 g before embedding. After hardening of specimens in normal conditions (t=20±2 °C, R.H.=95±5 %) the basic rebars were reached from AASC concrete and etched during (25±5) min in 10 % solution of hydrochloric acid with adding of urotropine (1 % by acid mass) to remove rests of cement stone and products of corrosion. The reference rebar, which weren’t embedded in concrete, were weighted and etched simultaneously with basic rebarS. After etching the basic and reference rebarS were cleansed by distilled water and were immersed in saturated solution of sodium nitrate for 5 min. Then rebarS were wiped by filter paper, dried up and weighted. Mean mass loss of the basic as well as the reference rebars were calculated as ratio of mean differences in their mass before and after etching to surface area. Mass loss was calculated as a difference between mean loss of the basic and the reference rebars.

3. Results and discussions

Restriction of Cl^- and SO_4^{2-} ions from seawater in plasticized AASC concrete was considered in view of advancing both chemical binding and occlusion while application of modifiers.

3.1. Chemical binding of Cl^- and SO_4^{2-} ions

Effectiveness of CA components in AASC’s for chemical binding of Cl^- and SO_4^{2-} ions was investigated.

AASC based on soda-ash

According to XRD the low-calcium hydrosilicates such as CSH(B) (d= 0.307; 0.280; 0.183 nm) and gyrolite 2CaO·3SiO_2·2H_2O (d= 0.33; 0.268; 0.180 nm) were identified in hydration products of the reference AASC mixed with seawater after 270 d of hydration (Figure 1, curve 1). Calcite (d= 0.307; 0.191; 0.160; 0.152 nm) can be fixed as well. Specified phases are typical for AASC [45].

Clorine- and sulfate-binding zeolite-like minerals, which are similar to nosean Na_8(Al_2Si_6O_24)(SO_4)·H_2O, sodalite Na_8(Si_4Al_3)O_12Cl and cancrinite (Na,Ca)_8(Al_2Si_6O_24)(CO_3,SO_4)_2·2H_2O etc., formed due to occlusion of aggressive ions by zeolite-like alkaline aluminosilicates, can be also predicted [7, 25]. However, these hydrates were not identified because of their submicrocrystalline state in specified period.

DTA confirms formation of calcium hydrosilicates CSH(B) by endothermic effect at t= 175 (dehydration) and exothermic effect at t= 865 °C (recrystallization into wollastonite) (Figure 2, curve 1). The endothermic effect at t= 175 °C and 700 °C (stepped dehydration) and exothermic effect at t= 865 °C (recrystallization into wollastonite) are typical for gyrolite 2CaO·3SiO_2·2H_2O. The endothermic effect at t= 890 °C confirms presence of CaCO_3.

Chloride-sulfate AFm phases, known as Kuzel’s salt 3CaO·Al_2O_3·0.5CaCl_2·0.5SO_4·10H_2O (d= 0.83; 0.42; 0.23 nm) [30], were also identified in hydration products of AASC, modified by admixtures of portland cement and calcium aluminate cement, mixed with seawater (Figure 1, curve 2). Specified phases were formed due to chemical binding of Cl^- and SO_4^{2-} ions by calcium hydroaluminate 3CaO·Al_2O_3·10H_2O, formed by co-acting of portland cement and calcium aluminate cement in hydration process.

The presence of Kuzel’s salt in hydration products of modified AASC, mixed with seawater, was confirmed by endothermic effects at t= 330 °C (dehydration) and 480 °C (departure of chloride) as well as by exothermic effect at t= 1000 °C (decomposition of sulfate) (Figure 2, curve 2). Relocation of effects to higher temperatures is evidence of CSH(B) and gyrolite with advanced crystallization.
Figure 1. XRD of alkali-activated slag cement, based on soda-ash and mixed with seawater, after 270 d of hydration: 1 – the reference; 2 – co-modified by portland cement and calcium aluminate cement; 3 – co-modified by portland cement, calcium aluminate cement and TSP.

Legend: H – calcium hydrosilicates CSH(B), G – gyrolite, C – calcite, K – Kuzel’s salt, ClA – chlorapatite.

Chlorapatite Ca$_5$(PO$_4$)$_3$Cl (d= 0.339; 0.285; 0.196 nm) was identified in hydration products of AASC mixed with seawater besides of Kuzel’s salt while additional application of TSP (Figure 1, curve 3). Specified mineral was formed due to chemical binding of Cl$^-$ ions by hydroxyapatite Ca$_{10}$(PO$_4$)$_6$(OH)$_$_2$.

The presence of chlorapatite in hydration products of AASC co-modified by portland cement, high aluminate cement and TSP, while mixing with seawater, was confirmed by endothermic effects at $t$= 80 °C (dehydration) as well as by exothermic effect at $t$= 360 °C (Figure 2, curve 3). Advanced crystallization of specified phases was confirmed by relocation of effects to higher temperatures.

According to electron microscopy, co-modification of AASC by mentioned additives ensures formation of hexagonal thin plates of Kuzel’s salt 3CaO·Al$_2$O$_3$·0.5CaCl$_2$·0.5SO$_4$·10H$_2$O (content in probe, %: CaO – 32.72, Al$_2$O$_3$ – 21.51, Cl – 10.27, SO$_3$ – 9.56) and long prismatic formations of chlorapatite (content in probe, %: CaO – 57.82, Al$_2$O$_3$ – 5.34, P$_2$O$_5$ – 47.82, Na$_2$O – 0.64) (Figure 3).

AASC based on sodium metasilicate

XRD testifies to formation of low-calcium hydrosilicates such as CSH(B) and gyrolite in hydration products of the reference AASC mixed with seawater after 270 d of hydration (Figure 4, curve 1). The presence of zeolite-like minerals (similar to nosean, sodalite, concrinite etc. by composition), which can bind Cl$^-$ and SO$_4^{2-}$ ions, may be assumed. The presence of mentioned hydrates was confirmed by DTA (Figure 5, curve 1).

According to XRD (Figure 4, curve 2) and DTA (Figure 5, curve 2) low-calcium hydrosilicates and Kuzel’s salt were fixed in hydration products of AASC, co-modified by portland cement and high aluminate cement, while mixing with seawater. Displacement of mentioned effects to higher temperatures ensures formation of CSH(B) and gyrolite with advanced crystallization.
The presence of chlorapatite in hydration products of AASC, co-modified by TSP and other mentioned additives (portland cement, high aluminate cement), while mixing with seawater, after 270 d of hydration was confirmed by XRD (Figure 4, curve 3) and DTA (Figure 5, curve 3). Microprobe analysis confirmed formation of hexagonal thin plates of Kuzel’s salt (content in probe, %: CaO – 32.72, Al₂O₃ – 21.51, Cl – 10.27, SO₃ – 9.56) and long prismatic formations of chlorapatite (content in probe, %: CaO – 57.82, Al₂O₃ – 5.34, P₂O₅ – 47.82, Na₂O – 0.64) (Figure 6).

Thus, co-modification of AASC by portland cement, high aluminate cement and TSP ensures binding of Cl and SO₄²⁻ ions by calcium hydroaluminate 3CaO·Al₂O₃·10H₂O and hydroxyapatite Ca₁₀(PO₄)₆(OH)₂ in Kuzel’s salt and chlorapatite agreeably. Specified structure formation of modified AASC ensures restriction of aggressive ions transport due to chemical binding in contact zone of plasticized AASC concrete with seawater.
Figure 6. SEM images of alkali-activated slag cement, based on sodium metasilicate and mixed with seawater, after 270 d of hydration.

3.2 Occlusion of Cl\(^-\) and SO\(_4^{2-}\) ions

Occlusive additive of zeolite was applied for additional restriction of aggressive ions transport in plasticized AASC concrete and enhancement of its protective properties to steel reinforcement.

Effectiveness of CA including portland cement, calcium aluminate cement, TSP and clinoptilolite, was tested while consistency class S4 (slump 160 - 210 mm) for AASC fresh concrete was provided. While mixing with seawater W/C ratios were 0.46 and 0.41 for cases of soda-ash and sodium metasilicate as alkaline components. Compressive strength of AASC concrete based on soda-ash and metasilicate corresponds to values of 40.3 and 48.7 MPa after 28 d of hardening and of 38.2 and 47.5 MPa after 270 d storing in seawater agreeably. Thus, corrosion resistance coefficient of plasticized AASC concrete based on soda-ash and sodium metasilicate corresponds to values of 0.95 and 0.97 agreeably. Dense macrostructure of AASC concrete prevents penetration of Cl\(^-\) and SO\(_4^{2-}\) ions even after 270 d in seawater.

The absence of Cl\(^-\) and SO\(_4^{2-}\) ions transport in plasticized AASC concrete was confirmed by qualitative reactions by solutions of silver nitrate (AgNO\(_3\)) and lead acetate (Pb(CH\(_3\)COO)\(_2\)) agreeably. The absence of white sediment on the surfaces of specimens confirmed this fact (Figure 7).

![Figure 7](image)

Figure 7. Sections of half specimens of plasticized AASC concrete, based on sodium metasilicate and stored 270 d in seawater, after spraying of silver nitrate (a) and lead acetate (b).

3.3 The state of steel reinforcement

The effect of CA on protective properties of plasticized AASC concrete was evaluated by mass loss tests of steel rebars. Mass losses of steel rebars, reached from plasticized AASC concrete (the reference as well as modified by CA) after 270 d of storing in seawater, were fixed (Table 1).

Generally, mass loss of steel rebars was in compliance with mandatory requirements (no more than 10 g/m\(^2\)) according to DSTU B V.2.6-181 that ensures protective properties of plasticized AASC concrete to steel reinforcement. In case of AASC concrete, based on soda ash, application of CA caused 3.7 times lesser mass loss than for the reference one. For AASC concrete, based on sodium metasilicate, application of CA resulted in 3.4 times smaller mass loss. The obtained results correlate with foregoing ones, concerning enhanced activity of AASC based on sodium metasilicate, if compare with soda-ash, that was caused by enhanced density of AASC concrete structure with advanced protective properties to steel reinforcement.
### 4. Conclusions

1. The enhancement of steel reinforcement protection in AASC concrete, which is obtained from high consistency mix and exploited in seawater, is possible due to restriction of Cl⁻ and SO₄²⁻ ions transport in structure while application of complex of additives including portland cement, calcium aluminate cement, trisodium phosphate and zeolite.

2. Restriction of aggressive ions transport from seawater in plasticized AASC concrete can be provided due to their chemical adsorption by gel-like phases, chemical binding in Kuzel’s salt 3CaO·Al₂O₃·0.5CaCl₂·0.5SO₄·10H₂O and chlorapatite Ca₅(PO₄)₃Cl as well as their occluding in structure of zeolite-containing admixture and hydrates presented by alkaline hydrosilicates. Advanced crystallization with densification of artificial stone restricts mentioned ions transport from seawater.

3. The 3.4…3.7 times lesser mass loss of steel rebars embedded in AASC concrete in comparison with non-modified ones, depending on anion of alkaline component, confirms advanced protective properties of plasticized AASC concrete, modified by proposed complex of additives after influence of seawater during 9 months.

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**Table 1.** The state of steel rebars embedded in plasticized AASC concrete.

| Type of AASC concrete                                      | Loss mass, g/m² |
|-----------------------------------------------------------|-----------------|
| reference AASC concrete based on soda-ash                 | 4.95            |
| reference AASC concrete based on sodium metasilicate      | 4.32            |
| AASC concrete based on soda-ash and modified by the complex of additives | 1.34            |
| AASC concrete based on sodium metasilicate and modified by the complex of additives | 1.27            |
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Acknowledgments

Authors would like to acknowledge the contribution of the COST Action CA15202 «Self-Healing concrete: the path to sustainable construction» of the European Union’s framework program HORIZON2020, http://www.cost.eu/COST_Actions/ca/CA15202. The authors also express their gratitude to the Ministry of Education and Science of Ukraine for financial support of this research that is carried out within the budgetary financing of topic with registration No 1020U001010 and implementation period 2020 - 2022.