

Study of influence of alkaline component type on pH value and properties of alkali activated concretes containing basalt rock

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Abstract. The paper discusses the questions of alkalinity changes in alkali activated cement-based materials at different stages of hardening. It was shown that use of alkali activated cement with dry alkaline component ("all-in-one system") in the presence of basalt rock leads to the immediate decrease of pH value and strength drop of the material. On the other hand, using alkaline component in the form of alkaline solution provides almost normal hardening of systems. Taking into account obtained results a methodology of changes in concrete mix design was proposed. In general, for different cement systems under study pH value varies from 8.5 to 12 reflecting on the materials structure formation processes and strength gain. Shrinkage of the systems was within the ranges 0.3...0.6 mm/m at 28 days age and 0.3...0.5 mm/m for different systems. Absence of expansion witnesses about compensation of ASR results and normal structure developments processes in the material comparing to OPC.

1 Introduction

Nowadays a question of widening of raw materials base became very sharp [1-2], including materials for cement and concrete production [3-5]. Permanent rising of prices on traditional high-quality aggregates leads to the search of cheaper ones, but at the same time the less qualified materials [6-8]. Between aggregates in this category there is a high possibility to have active grains (active silica) [9-11], able to participate in reaction with cement matrix and leading to the problems [12-14], some times – technogen accidents [15-17].

Problem of alkali-silica reaction (ASR) of aggregates in concretes is also critical from the point of view of absence of such tests in the quality assurance procedures of the most concrete and concrete constructions manufactures and determination of alkaline corrosion appearance is possible only at the stage of exploitation of construction, when it is too late to provide any actions.

Problem of losses in quality of aggregates and rise of their activity takes a top rate [18-20]. The point is that shift to the coal use in the portland cement clinker manufacture results in increasing of Na and K compounds in the cement [21-23]. That fact together with increasing of aggregates activity provides appearance of destructive processes in concrete directly in exploitation period [24-26]. In the body of concrete appears reactions «alkalis – active silica» (ASR) [27-29], leading to the formation in the hardened concrete pores new formations, exceeding volume of the pore [30-32]. These formations further lead to the internal stresses in the concrete and may lead to the increased leaching of the hardened stone and development of destructive processes [33-35].

Results of the studies [36-38] show that there are some ways to ASR development in concrete by using active mineral aggregates [39-41], which makes it possible to regulate and control ASR processes development, appearing because of this reaction and make it constructive [42-44]. The most effective systems in this case are alkaline cements [45-47], which make it possible to change ASR process mechanics from destructive to constructive, shifting in time moment of to the initial stages of structure formation and thus not creating possibility to have destructive processes [48-50].

At the same time, provided investigations studies mostly use of alkali activated cements by two component technology (alkaline component is in the liquid form). Such systems are effective enough, but application of this technology is complicated and far from the ideal [46, 47]. The modern technologies in construction require application of alkali activated cements in the dry state, «all-in-one» system), which are simply mixed with water.

The problem of such cement application together with active aggregates is that alkaline solutions (by the old technology) have much more alkalis in the system [49], and in the single component cement present alkalis content could be not enough for compensation of negative influence of active aggregate. Use of the extra alkalis on the other hand in dry cement system also is dangerous because of possibility of high leaching rates of material and development of destructive processes [51].

The aim of the study is to investigate changes in pH value for different cement systems and to study optimal alkaline component content in the cement system.

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2 Raw materials and methods

To investigate the processes of inner corrosion of the slag alkaline concrete the basalt rock has been selected as aggregates. Quartz sand was taken for reference. Chemical composition of the rocks is given in Table 1.

Table 1. Chemical composition of rocks

| Oxide | Oxide content, % by mass, in aggregate |
|--------------------------------|--|
| | Basalt |
| SiO ₂ | 50.42 |
| Al ₂ O ₃ | 14.0 |
| Fe ₂ O ₃ | 6.14 |
| FeO | 8.37 |
| TiO ₂ | 2.66 |
| MnO | 0.243 |
| CaO | 8.04 |
| MgO | 5.56 |
| P ₂ O ₅ | 0.316 |
| K ₂ O | 0.71 |
| Na ₂ O | 2.27 |
| SO ₃ | 0.07 |
| Σ, % | 99.57 |

For a complex estimation of durability of the slag alkaline concretes with alkali-reactive aggregates, the strength kinetics was investigated, for which a standard procedure was used.

Ground granulated blast furnace slag with a specific surface 450 m²/kg (by Blaine) was used in the experiments. Chemical composition of the slag is shown in Table 2.

The graded aggregates were in used in the experiments. The quantities of aggregate retained on the No 2.5 mm, 1.25 mm, 0.63 mm, 0.315 mm and 0.14 mm-sieves were 20 % by weight of each.

The beam specimens (25×25×254 mm) made of slag alkaline concretes of equal consistency with a flow of 170±5 mm, according to procedure specified by the DSTU B V.2.7-186, were prepared using a vibrating table. The cement to aggregate ratio was taken as 1: 2. Used as alkaline component were the Na₂CO₃ in dry state and soluble glass with density 1250 kg/ cub m.

Alkalinity of the hardening systems (pH) was determined after 1, 2, 3, 4, 5 i 24 hours of hardening by measuring properties of 10% water solution at pH-meter PL-700al (pH/ORP/Conductivity/TDS/Salt/DO/Temp).

As a reference material was taken OPC Grade 42.5 with a specific surface 320 m²/kg by Blaine.

Also for reducing alkaline corrosion value of concrete the active mineral admixture was taken (represented by metakaolin) in the quantity 10% (MK).

Length of the specimens was measured using a pointer-type indicator with a scale factor of 0.01 mm.

Between 2 to 4 hr. before the next measurement the specimens were removed from the thermostat and allowed to cool in the environment with T=20±2 °C.

Table 2. Chemical composition of blast furnace slag

| Oxides | Oxide content, % by mass | |
|--------------------------------|--------------------------|--|
| | No 1 | |
| SiO ₂ | 38.6 | |
| Al ₂ O ₃ | 6.50 | |
| CaO | 48.50 | |
| MgO | 5.40 | |
| MnO | - | |
| FeO | 0.36 | |
| SO ₃ | 1.99 | |
| TiO ₂ | - | |
| P ₂ O ₅ | - | |
| K ₂ O | - | |
| Na ₂ O | - | |
| Σ, % | 101.35 | |

Linear deformations of the slag alkaline concretes were compared with those of the reference specimens made with sand. The linear deformations of the slag alkaline concretes made with alkali- reactive aggregates were studied using a basic blastfurnace slag as the most typical representative reflecting peculiarities of the processes of hardening [51].

3 Results and discussion

Traditionally during mix design of alkali activated cements alkaline solutions of sodium silicates and carbonates are using as an alkaline component. In our case, to determine limit points of alkali activated cements were studied of the cements using soluble glass (SS) with density 1250 kg/m³ with silicate modulus 2.6.

Variation of pH value for different cement compositions in paste are shown in Table 3 and at the Figure 1.

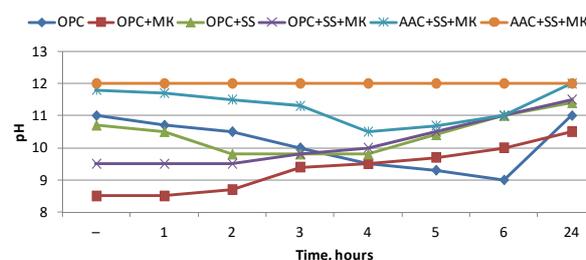


Fig. 1. Variation of pH value depending from content of the alkaline component, represented by alkaline solution.

Analysis of the results shows that at initial stages the highest pH value have alkali activated cement using soluble glass, regardless metakaolin admixture.

Use of OPC also provides high alkalinity of media (pH=11). However, it has regularity to rapidly decrease in time and at the age of 6 hours it is pH=9. Then at 1 day age is returns to pH=11. Such behavior could be explained in that way: high alkalinity at initial stage provided by calcium hydroxide further decreasing via reaction with active SiO₂, which is leaching from the aggregate. Then, reaching limit point in the system, content of soluble silica in the media is reducing and content of portlandite is

rising because of continuation of clinker minerals reaction continuation. This creates proper conditions for hardening and strength gain and this is explaining absence of negative influence of active aggregate on material exploitation properties at initial stages of hardening and exploitation.

Table 3. Variation of pH value in alkali activated cements using alkaline solutions as alkaline component and referenced materials.

| Time, Hrs | pH | | | | | |
|-----------|------|--------|--------|-----------|-----------|-----------|
| | AAC | AAC+MK | AAC+SS | AAC+SS+MK | AAC+SS+MK | AAC+SS+MK |
| – | 11.0 | 8.5 | 10.7 | 9.5 | 11.8 | 12.0 |
| 1 | 10.7 | 8.5 | 10.5 | 9.5 | 11.7 | 12.0 |
| 2 | 10.5 | 8.7 | 9.8 | 9.5 | 11.5 | 12.0 |
| 3 | 10.0 | 9.4 | 9.8 | 9.8 | 11.3 | 12.0 |
| 4 | 9.5 | 9.5 | 9.8 | 10.0 | 10.5 | 12.0 |
| 5 | 9.3 | 9.7 | 10.4 | 10.5 | 10.69 | 12.0 |
| 6 | 9.0 | 10.0 | 11.0 | 11.0 | 11.0 | 12.0 |
| 24 | 11.0 | 10.5 | 11.4 | 11.5 | 12.0 | 12.0 |

Introduction of metakaolin admixture to the OPC system provides decreasing of total alkalinity of the system at initial stage to pH=8.5. Further pH is rising slowly up to 10.5 at 6 hours age. Such peculiarities of hydration and structure formation processes provide low strength gain of OPC systems with active mineral admixtures using active aggregates, limiting application areas for such materials.

Introduction of metakaolin into the alkali activated cement composition using soluble glass as an alkaline component also decreases a while total alkalinity of the system at initial stage (pH reduces to 11.8 comparing to 12). Further alkalinity reduces for 4 hours, reaching pH=10.5. After that alkalinity rises to pH=11 at the age of 6 hours and reaches pH=12 at 24 hours age. Such high pH value in alkali activated cement using soluble glass at different stages provides possibility to use active aggregate without loses in strength properties.

Alkali activated OPC has low pH value at initial stage – 10.7. Further alkalinity of the system is rising and reaches pH=11 at 6 hours age and pH=11.4 at 24 ours age. Such behavior and differences from processes in OPC could explain by shifting of alkalinity source from calcium hydroxide to sodium compounds because of decreasing of total basicity of new formations (hybrid compounds). This makes it possible to predict slow strength gain at initial stages and further rapid increasing of strength after 1 days of hardening.

Introduction of metakaolin provides decreasing of initial pH to 9.5 with stability during 2 hours and rising rapidly to 11 at 6 hours age and 11.5 at 24 hours age. Such behavior could be explained by presence of high alkaline ions source in the system and bonding of active silica from the basalt rock composition at initial stages. This makes it possible to predict a little bit slow hardening at initial

stages and reaching of normative properties at 28 days age.

Thus a way, from the point of view of high alkalinity of media at initial stages the best systems are alkali activated OPC with and without metakaolin admixture. They have high alkalinity rates as at initial stages (pH=12 and 11.8 respectively), so as at the age of 1 day (pH=12 for both systems).

Alkalinity of OPC systems is a little bit lower (pH=11) at initial stage, decreasing at initial stages of hardening and than stabilizing at the level pH=11 at the age of 1 day providing source for normal hardening and structure formation. Introduction of metakaolin decreases total alkalinity at initial stage to 8.5 with slow rising to 10 at the age of 6 hours and 10.5 at 28 days age. Such process of structure formation is potentially problematic for strength properties.

The worse system from the high pH value at initial stage is alkaline OPC. Low pH of the system without metakaolin (10.7) with following stabilization at pH=11 (at 1 day age) provides slow strength gain, and introduction of metakaolin decreases pH to 9.5, storing for 2 hours and rising to 11 at 6 hours age.

Also it should be mentioned that for alkali activated cement system with soluble glass the pH value is 12 at all stages regardless of metakaolin introduction.

On the other hand, the modern world needs single-component premixed cements. Alkaline component of such alkali activated cement was represented by sodium carbonate (soda ash).

To determine optimal content of alkaline component alkali activated cement (AAC) type I according to DSTU B V.2.7-181 was used with additionally alkalis (sodium carbonate) in the quantity 2, 4, 6, 8 i 10% by mass. Initial content of alkaline component in the cement was 4.5% (2.5% calculated on Na₂O). Thus, maximal alkalis content in the cement system was 7.54% by mass calculating on Na₂O. Such significant increasing of alkalis content in the system was directed for compensation of negative influence of active aggregate on structure and service characteristics of material. Test results are given in Table 4 and shown on Figure 2.

Table 4. Influence of alkaline component content on total alkalinity of cement paste media.

| Time, hrs | pH | | | | | |
|-----------|------|------------------|------------------|------------------|------------------|-------------------|
| | AAC | AAC +2% soda ash | AAC +4% soda ash | AAC +6% soda ash | AAC +8% soda ash | AAC +10% soda ash |
| – | 10.0 | 10.0 | 11.0 | 11.5 | 11.7 | 11.7 |
| 1 | 9.0 | 10.0 | 11.0 | 11.5 | 11.5 | 11.5 |
| 2 | 9.0 | 9.5 | 10.5 | 11.0 | 11.5 | 11.5 |
| 3 | 9.0 | 9.2 | 10.5 | 11.0 | 11.2 | 11.2 |
| 4 | 8.7 | 9.0 | 10.2 | 11.0 | 11.2 | 11.2 |
| 5 | 8.7 | 9.0 | 10.2 | 11.0 | 11.0 | 11.2 |
| 6 | 8.5 | 8.7 | 10.2 | 11.0 | 11.0 | 11.0 |
| 24 | 7.5 | 7.8 | 7.8 | 11.0 | 11.0 | 11.0 |

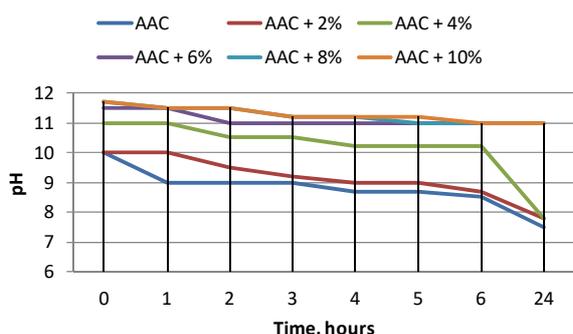


Fig. 2. Variation of pH value depending from content of the alkaline component, represented by dry salt.

Analysis of obtained results shows that using active aggregates alkalinity of the system if decreasing even at initial stage. Without additional alkaline component at initial stage pH is 10 and rapidly dropping. From the point of view of total; alkalinity of the system optimal content of additional alkaline component is 6% of soda ash that provides totally 5.4% of alkalis in the system calculation on Na₂O.

Obtained results confirms predictions that using active aggregates with dry alkali activated cement mix the traditional alkalis content is not enough. This could be explained by reaction between silica acid, leaching from the basalt rock, and alkaline component, resulting in sodium hydro silicates formation. Such process is not destructive. However, decreasing of total content of free alkalis in the media and pH value leads to slowing of structure formation processes in alkali activated cement of stop it in general. That is why for storing total alkalinity of the system the additional alkaline component content 6-6% by mass of the cement is required.

Obtained results make it possible to propose method of compensation of negative influence of active aggregates on concrete basing on single component alkali activated cements. This will provide necessary density of alkaline component in the cement media and create conditions for hardening.

Compressive strength of the concretes under study is shown in Table 5 and on Figure 3.

Table 5. Strength properties of fine grain concretes using basalt rock as an aggregate

| № | Composition | Compressive strength of fine-grain concrete, MPa, at the age, days | | | |
|---|-----------------------------|--|------|------|------|
| | | 2 | 7 | 28 | 90 |
| 1 | OPC+ Soluble glass | 18.8 | 28.4 | 46.8 | 58.4 |
| 2 | OPC+ Soluble glass + 10% MK | 13.8 | 28.4 | 70.8 | 81.5 |
| 3 | AAC type I | 10.8 | 29.4 | 41.8 | 48.4 |
| 4 | AAC type I +10% MK | 5.8 | 28.4 | 42.3 | 48.8 |
| 5 | OPC Grade 42.5 | 8.8 | 23.4 | 41.2 | 47.3 |
| 6 | OPC Grade 42.5 | 5.2 | 20.1 | 40.2 | 43.1 |

Analysis of obtained results shows that systems with metakaolin are a little bit weaker than systems without admixture. The best strength properties have systems using alkaline OPC (with soluble glass) regardless using metakaolin. This could be explained by presence of high

sodium silicate ions in the system because of soluble glass use.

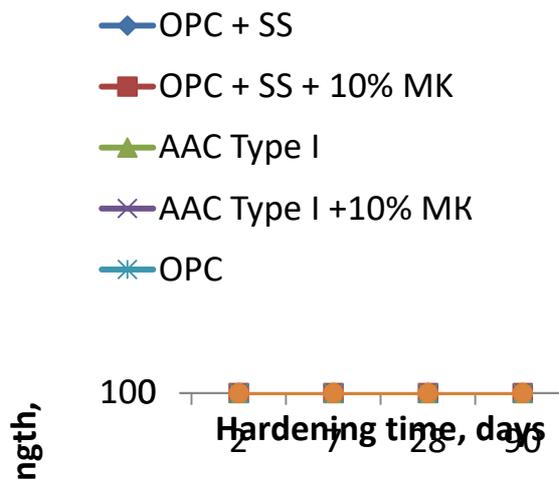


Fig. 3. Variation of compressive strength of fine grained concretes using basalt rock as an aggregate.

The systems mixed with water are characterized by almost equal strength at 28 days age and showing strength gain at further ages. This could witness about constructive development of structure formation processes and absence of huge destructive processes in the material at the stages under study.

Fine grained concretes on AAC type I basis are showing equal strength properties at 2 days age comparing to those on OPC basis. At the age of 7 days characteristics of alkaline concrete is higher than characteristics of OPC based concretes, becoming equal at 28 days age and a little bit higher at 90 days age. That is witnessing about enough potentials of properties development for the systems on AAC basis.

At the same time, strength properties are not expressing well development of destructive processes caused by ASR in concretes. The more informative are study of shrinkage (expansion) deformations of the systems under study. The basis characteristics were taken immediately after remolding, and then characteristics were fixed at 2, 7, 28, 90 and 180 days of hardening. Tests of deformation were performed by measuring tool with indicator IP-04 with accuracy 0.01 mm. Results of the study are shown in Table 6 and on Figure 4.

Table 6. Shrinkage deformation of fine grained concretes using basalt rock as an aggregate

| № | Composition | Shrinkage deformations, mm/m, at the age, days | | | | |
|---|-----------------------------|--|-------|-------|-------|-------|
| | | 2 | 7 | 28 | 90 | 180 |
| 1 | OPC+ Soluble glass | -0.23 | -0.45 | -0.63 | -0.55 | -0.51 |
| 2 | OPC+ Soluble glass + 10% MK | -0.30 | -0.49 | -0.61 | -0.49 | -0.36 |
| 3 | AAC type I | -0.21 | -0.33 | -0.44 | -0.43 | -0.40 |
| 4 | AAC type I +10% MK | -0.15 | -0.26 | -0.31 | -0.33 | -0.32 |
| 5 | OPC Grade 42.5 | -0.25 | -0.41 | -0.50 | -0.44 | -0.40 |
| 6 | OPC Grade 42.5 | -0.19 | -0.27 | -0.41 | -0.30 | -0.23 |

Analysis of the results shows that all materials under study are characterized by shrinkage deformations in all

test area. It should be mentioned that highest shrinkage for sure have compositions using soluble glass, and the lowest – alkali activated cement AAC type I. That could be explained by higher gel phases in the systems with soluble glass, which are much lower represented in the systems with dry alkaline components.

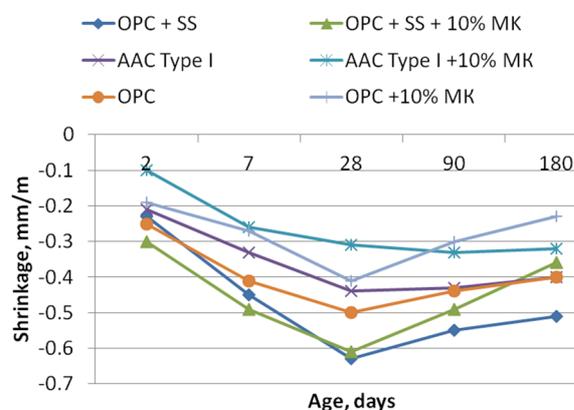


Fig. 4. Shrinkage deformation of fine grain concrete using basalt rock as an aggregate.

It has to mention that for all systems under study shrinkage is reducing with time, witnessing about development of expansion processes via ASR.

The best systems were alkali activated cement with and without metakaolin admixture, which are characterized by spline shrinkage deformation development and insignificant function changes by aggregates corrosion processes gain and expansion.

The present results are expressing specimens hardening in normal conditions. The further studies will be focused on the use of express methods of shrinkage investigation.

Conclusion

1. It is shown that the best cement composition from the high pH value at the initial stages of hardening are alkaline OPC using soluble glass as an alkaline component as in initial state, so as using metakaolin admixture (pH=12 and 11.8 respectively), keeping the rate at the age of 1 day (pH=12 for both systems). Alkalinity of OPC systems is lower (pH=11) at the initial stage, reducing at the initial terms and stabilizing at pH=11 at the age of 1 day, providing normal condition for hardening. Introducing a metakaolin decreases pH to 8.5 with rising to 10 at the age of 6 hours and 10.5 at the age of 1 day. The worth system is alkaline OPC. Low initial alkalinity (pH=10.7) with following reducing to the 10 at the age of 1 day provides low strength gain, and introduction of metakaolin admixture reduces pH to 9.5, storing it for 2 hours and further rising to 11 at the age of 6 hours.
2. Use of active aggregates could complicate of stop structure formation processes in alkali activated cements with dry alkaline component. It is shown that traditional alkalis content in the cement is not enough for normal structure formation processes,

and total alkalinity of the system drops at the initial stages and keep reducing in time. Optimal content of alkalis in the system is 6-8%, providing pH 11.5-11.7, and also pH storage in time (up to 11 within 4-24 hours). From the strength point of view the best systems are alkaline OPC as it is, so as with metakaolin admixture (46.8 and 70.8 MPa at the age of 28 days respectively).

3. From the shrinkage point of view the best systems are alkali activated cements with and without metakaolin admixture (-0.44 and -0.31 mm/m respectively at the age 28 days), showing also lower shrinkage at further ages. That could witness about slowing of alkaline corrosion of aggregate processes in the material.

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