Cement compositions with modified hydrosodalite

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Kaunas University of Technology, Studentų St. 48, LT-51367 Kaunas, Lithuania Hydrosodalite has been synthesized at low-temperature (105 °C) in the unstirred suspension at isothermal curing from NaOH, Al(OH)₃, amorphous SiO₂ × nH₂O and H₂O. By the process of modification of hydrosodalite ions exchange was carried out by reduction of Na⁺ and increasing Ca²⁺. Through the high puzzolanic activity of hydrosodalite and formation of hydroaluminate phases it has the positive effect on the hardened cement paste compressive strength, especially at the early state of cement hydration. This modified hydrosodalite and cement can undergo puzzolanic reaction which results in less void content and higher final compressive strength, depending on the amount of synthetic modified zeolite. The amount of the additive has various influences on the strength of the hardened cement paste, however, with the increase of the additive amount from 5 to 20%, the compressive strength slightly improves, especially at the early state of cement hydration. The highest compressive strength was observed in samples with 15% of the modified hydrosodalite additive. The increase of the strength is related to the impact of SiO₂ and Al₂O₃ presented in modified hydrosodalite.

Key words: modified hydrosodalite, cement hydration

INTRODUCTION

Zeolites belong to the group of minerals that includes alkaline and alkaline-earth metal aluminosilicate hydrates, household economy and environment protection [1, 2]. Easy chemical modification of zeolites provides wide opportunities to perform controlled changes in their structure and properties. These parameters may be modified during synthesis, by ion exchange and other methods [3–6].

High amount of investigations of hydration zeolites in cement and concrete matrix and as additives in advanced building materials have been made in last years. The effect of modified zeolite on the alkali-silica reaction expansion was analyzed [7], and comparison between other mineral admixtures such as fly ash and ground blast furnace slag was made. The resistance to acidic and sulfate attack of Portlandpozzolan cement containing 35% by weight of zeolite was compared with that of unamended Portland cement [8]. This confirms that zeolitic cement can replace Portland cement in many applications with the advantage of higher resistance to acidic and sulfate attack [9].

The examined zeolite [10] consists mainly of heulandite type II and is a pozzolanic material that contributes to the strength development of zeolite-cement mixtures, the consumption of $Ca(OH)_2$ formed during the hydration of Portland cement and the formation of cement-like hydrated products.

Hydration and strength development of high alumina cement (HAC) containing sodium sulfate and different zeolites have been investigated and the zeolites obtained from different sources including different types containing clinoptilolite, chabazite, stilbite and natrolite are used. Chabazite was the most effective zeolite in promoting stratlingite formation in the HAC paste [11].

The hydration reaction of Ca A-type zeolite and calcium hydroxide has been investigated and the results revealed that

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the structural material of hydrocalumite (C-A-H) is generated at the early stage of hydration, and stratlingite – vertumnite develops afterwards [12].

F. Canpolat has presented a detailed study about the use of zeolite additives in cement production. According to the test results, mortars were produced by replacing cement with 5%–35% of zeolite [13].

Zeolite formation in alkali-activated cementation systems (AAC) was investigated and the introduction of sufficient alkali promotes the growth of crystalline zeolites in tobermorite matrix during autoclave curing [14].

Depositions of cesium and cobalt sorbet on zeolite in matrices of blast furnace slag were investigated. Cs and Co were sorbet from model solutions and waste water on chemically treated granular zeolite. The zeolite was incorporated into cement slurries based on blast furnace slag [15].

The cement-bentonite interface is a highly dynamic region in engineered barrier systems designed to isolate radioactive wastes. The formation of phillipsite-Na in these experiments is in agreement with the available thermodynamic data on zeolites [16]. A granular material was formulated by mixing zeolite by-product with Portland cement, and this material was tested for its efficiency for heavy metal removal from aqueous solutions [17].

In the work [18] the use of spent zeolite which eliminated lead ions from industrial spent water in the constructional practice is an alternative to make it safe without the second environmental pollution – superficial and underground waters and soils.

A calorimetric method for determining partial and integral heat of hydration reactions in zeolites is presented. Results of this method agree well with the values previously determined by other calorimetric techniques and regression of phase equilibrium data in the case of chabazite and other minerals [19].

The patent about lightweight concrete products, in which zeolite represents the major component, material and bubble-generating agent is presented, also binding material, useful for hazardous waste stabilization, comprising zeolite as a major component, is proposed [20]. In the Patent [21] it is suggested to use the concrete with some zeolites cement in order to attain the objective of shortening the time of cement hydration.

Although pure zeolites are widely used in concrete technologies, the reviewed references did not contain any data of the use of modified zeolites with Na⁺ replaced by Ca²⁺ ions in concrete technologies.

The present work is aimed at the possibilities of using modified hydrosodalite as a hydraulic admixture and its influence on cement paste hydration processes. Also, in this work the hydraulic activity of pure and modified hydrosodalite was investigated. The samples of hardening cement paste were tested by X-ray, thermal analysis, IR spectra, scanning electron microscopy (SEM) and compression strength.

EXPERIMENTAL

Pure hydrosodalite was synthesized from reagents: NaOH, Al(OH)₃, amorphous SiO₂ \cdot *n*H₂O (loss on ignition was 23.5%). The molar ratio of the starting material $Na_2O : Al_2O_3 : SiO_2 : H_2O_3$ was 2:1:2:10. Low-temperature (105 °C) zeolite synthesis was performed in the unstirred suspension at the isothermal curing duration of 2 hours. The final product of synthesis was filtered, dried at about 60 °C temperature and sieved through a 0.5 mm-mesh sieve. Ions exchange reactions were performed in the unstirred suspension: saturated CaCl, solution obtained from CaCl, and water was filled up of zeolite. Ions exchange investigations at the temperature of 85 °C lasted about 5 minutes. The chemical composition of pure and modified hydrosodalite was evaluated from EDS paterns. It was confirmed by the chemical composition that modified hydrosodalite contains a significant content of CaO and a decreased content of Na₂O. The content of SiO₂ and Al₂O₃ in both pure and modified hydrosodalite are almost the same (Table).

A commercial Akmenes cementas Portland cement type CEM I 42.5 R for the tests was used. The chemical composition of Portland cement is shown in Table.

Table. The chemical composition of Portland cement clinker, pure and modified hydrosodalites

| Chemical composition | Portland cement clinker | Pure hydrosodalite | Modified hydrosodalite |
|------------------------------------|-------------------------|--------------------|------------------------|
| SiO ₂ , % | 20.54 | 34.58 | 34.07 |
| Al ₂ O ₃ , % | 5.49 | 37.16 | 37.32 |
| Fe ₂ O ₃ , % | 3.52 | _ | _ |
| CaO, % | 63.16 | - | 7.78 |
| SO ₃ , % | 0.95 | _ | _ |
| Cl ⁻ , % | 0.001 | _ | _ |
| MgO, % | 4.31 | _ | _ |
| CaO free, % | 0.52 | - | _ |
| Na ₂ O, % | _ | 23.88 | 16.55 |
| C ₃ S, % | 56.38 | _ | _ |
| C ₂ S, % | 16.34 | _ | _ |
| C ₃ A, % | 8.60 | _ | _ |
| C ₄ AF, % | 10.72 | _ | _ |

In all test sample compositions the cement and water ratio (W/C) was stable (W/C = 0.55). The compositions differed by the contents of the modified hydrosodalite – 0; 5; 10; 15 and 20%. The formed test samples, $2 \times 2 \times 2$ cm sized cubes, were continuous process of hardeneng for 28 days under conditions prescribed by EN 196-1. The compressive strength of hardened cement paste was determened by EN 196-1. A ToniTechnik 2020 press was used to evaluate the compressive strength of hardened cement paste.

The X-ray powder diffraction data were collected with a DRON-6 X-ray diffractometer with Bragg-Brentano geometry using Ni-filtered Cu K α radiation and a graphite monochromator, operating with the voltage of 30 kV and emission current of 20 mA. The step-scan covered the angular range 2–70° (2 θ) in steps of 2 θ = 0.02°.

Simultaneous thermal analysis (STA: differential scanning calorimetry-DSC and thermogravimetry-TG) was also employed for measuring the thermal stability and phase transformation of products at a heating rate of 15 °C/min, the temperature ranged from 30 °C up to 1 000 °C under the air atmosphere. The test was carried out on a Netzsch instrument STA 409 PC Luxx. The ceramic sample handlers and crucibles of Pt-Rh were used there.

IR spectra were recorded with a Perkin Elmer FT-IR System spectrometer. For the IR analysis, 1 mg of the substance was mixed with 200 mg of KBr and compressed in a forming press under vacuum.

The structure of hardened cement paste was studied by scanning electronic microscopy. A high resolution scanning electron microscope FEI Quanta 200 FEG with a Schottky field emission gun (FEG). Chemical compositions of hydrosodalite and modified hydrosodalite were investigated by an energy-dispersive X-ray spectrometer (EDS) with a silicon type drift droplet detector.

RESULTS AND DISCUSSIONS

This study was conducted on Portland cement hydration process and hardened cement properties with pure synthetic and modified hydrosodalite additives and confirmed the possibility of using these additives in concrete technology. It explains processes of hydration and the hydration products formed as a result of morphological changes and their impact on the physical-mechanical properties of samples.

Hydraulic activity and microstructure of hydrosodalite

Figure 1 presents pure and modified hydrosodalite X-ray diffraction analysis curves. The X-ray diffraction curves show that the diffraction peaks with interplanar distances of (*d*): 0.628; 0.363; 0.281; 0.256; 0.290; 0.174 nm are characteristic of hydrosodalite. Therefore, after modification of pure hydrosodalite $CaCl_2$, i. e. after the ion exchange, the crystal structure of this zeolite remained unchanged.

In the IR spectrum of the sample a strong absorption band is in 3 455, 3 463 cm⁻¹ and depicts the frequencies of

the hydroxi group that are bound with hydrogen bonds. An absorption band in 1653, 1663 cm⁻¹ shows deformation vibration water molecules. The IR spectrum in 988 cm⁻¹ displays a broad absorption band attributed to atomic frequencies of (Si, Al)–O bond in tetrahedron along lines that bind $[(Si, Al)O_4]^{4-}$ tetrahedron oxygen atom with the central Si or Al atom. The characteristic hydrosodalite absorption bands are as follows: in 988 cm⁻¹ of asymmetric atomic vibration, in 722, 662 cm⁻¹ of symmetrical atomic vibration, in 461, 462 cm⁻¹ of Si(Al)–O deformation vibration. The band of adsorption in 1424 cm⁻¹ of spectrum is characteristic of CaCO₂.

The hydraulic activity of pure and modified hydrosodalite was determined. Both tested hydrosodalites had similar CaO binding capacity: 184 mg/g in pure hydrosodalite and 193 mg/g in modified hydrosodalite during the test duration. During 24 hours, pure hydrosodalite binds 5.18 mg of CaO, while modified hydrosodalite binds slightly less, 5.02 mg of CaO. It was also determined that solid-phase volume (sediment volume) increases almost 4 times during chemosorption with pure hydrosodalite and about 3.5 times with modified hydrosodalite.



Fig. 1. The X-ray diffraction patterns (*a*) and IR spectra (*b*) of hydrosodalite: *1* – pure; *2* – modified. Notes: Hs – hydrosodalite; CC – calcium carbonate

The obtained data show that investigated hydrosodalite demonstrates a rather high hydraulic activity of 184 and 192 mg/g and therefore can be treated as hydraulic substance. We believe that the obtained data result from many factors such as active SiO_2 and Al_2O_3 present in zeolites, specific surface area, etc.

Cement hydration with modified hydrosodalite

Cement is a complicated system consisting of various minerals which during hydration react with water at different rate and form crystallization products of different composition and degree of crystallinity. Calcium hydroxide is one of the hydration products developing from a saturated solution with high pH. The pH of pure hydrated cement is 12–14; therefore, when zeolite admixture is present in the cement and water mixture, high pH of zeolite causes disintegration and depolimerization of the aluminosilicate frame:

$$n [\equiv Si-O-Si\equiv] + 3nOH^{-} \rightarrow [-O-Si-(OH)_{3}]_{n}, \qquad (1)$$

$$n = \text{Si-O-Al} + 7nOH^{-} \rightarrow$$

$$\rightarrow [-O-\text{Si-(OH)}_{3}]_{n} + [\text{Al-(OH)}_{4}]_{n}.$$
(2)

During the depolimerization $[SiO(OH)_3]^-$ and $[Al(OH)_4]^$ ions enter the solution and react with Ca²⁺ ions thus forming hydrated calcium silicates and calcium aluminates, very similar to those compounds which develop during the hydration of the ordinary cement. Consequently, the zeolite additive makes the cement paste microstructure thicker [22]. The presence of zeolite in the puzzolanic reaction increases the C–S–H content and significantly improves the microstructure of hardened cement paste. The remainders of the collapsed zeolite SiO₂ framework react with lime and form C–S–H type gel, the content of which is in direct proportion to the zeolite content. This raises the hydration rate not only of clinker minerals but of the liquid glass phase as well.

X-ray and thermal analysis of hardened cement paste

Investigation of X-ray diagrams of hardened cement paste with hydrosodalite additive has revealed that with the increase of hydrosodalite admixture content from 0 to 20% the content of $Ca(OH)_2$ in mineral composition of cement paste reduces. Diffraction patterns in Figures 2 a) and b) show that the $Ca(OH)_2$ interplanar spacing of the peaks of 0.493 nm is more intensive when the hydrosodalite additive is not used;



Fig. 2. X-ray diffraction patterns of hardened cement paste after 3 days (*a*) and after 28 days (*b*). Hydrosodalite content accounts for 20% (1); 15% (2); 5% (3); 0% (4). Notes: CH – portlandite; A – alite; K – calcium silicate hydrate; D – belite; HS – hydrosodalite; E – ettringite; CC – calcite; G – gypsum, Z – calcium aluminium silicate hydrate



Fig. 3. DTA curves of hardened cement paste after 3 (*a*, *b*) and 28 (*c*, *d*) days. Hydrosodalite content is 0% (*a*, *c*) and 20% (*b*, *d*)

peak intensity drops down when 5, 15 or 20% of hydrosodalite is added.

With hydrosodalite content at 5% all its content reacts with cement hydration products during the first three days of hardening. When hydrosodalite content in cement paste is at 15 or 20%, X-ray patterns of hydration products show that part of hydrosodalite (d - 0.6318, 0.3638 nm) remains unbound (Fig. 2).

The influence of sample hardening time has shown that with longer hardening time bigger amounts of alite and belite hydrate forming calcium hydrosilicates and portlandite. Thus in X-ray patterns we observe more intensive portlandite (d - 0.491, 0.262, 0.193, 0.179 nm) peaks and less intensive alite and belite peaks. We may also state that hydrosodalite additive accelerates the cement hydration process because peaks in X-ray diagrams show more intensive peaks of calcium hydrosilicates and less intensive peaks of the minerals of cement when hydrosodalite additive is used. Peaks of ettringite (d - 0.967), 0.560 nm) and gypsum (*d* – 0.758 nm) become obviously less intensive with the increase of hydrosodalite additive content (Fig. 2). By using synthetic modified hydrsodalite additive calcium aluminium silicate hydrate forms in hardened cement paste.

In addition to X-ray analysis, thermal analysis of hardened cement paste samples was performed (Fig. 3). Thermal analysis data have confirmed the results of X-ray analysis. Endothermic peaks from 102 up to 107 °C show water loss from the phase of tobermorite gel. The endothermic peak at 154, 149 °C temperature is related to the dehydration of calcium (magnesium) aluminosilicate hydrates. It clearly increases with hydration time. Another intensive endothermic peak in 469-447 °C temperature range indicates the dissociation of Ca(OH)₂. Peaks in 695-723 °C temperature range are related to water withdrawal from calcium hydrosilicate (1.5-2.0) CaO · SiO, nH₂O of tobermorite pattern. The aforementioned temperature induces the formation of C₂S. Endothermic peaks in 745-747 °C temperature range indicate the thermal dissociation of CaCO₂.

According to TG curves we may state that $Ca(OH)_2$ content in the mineral composition of hardened cement paste with hydrosodalite additive reduces with addition of modified hydrosodalite. As seen in Fig. 3, the weight loss, associated with $Ca(OH)_2$, by adding 20% of the hydrosodalite, decreases from 12 to 9% after 3 days and from 15 to 8% after 28 days of



Fig. 4. SEM image of hardened cement paste after 28 days of hardening without the additive (*a*) and with modified hydrosodalite additive (*b*, *c*)

hydration. The loss of $CaCO_3$ content was also observed in samples where the admixture was added, while after 28 days of hydration $CaCO_3$ disappears completely (Fig. 3 d).

Microstructure of hardened cement paste

The SEM image of hardened cement paste without admixtures reveals rhombohedral-shaped $CaCO_3$ flat crystals and hexagonal laminated crystals of $Ca(OH)_2$ (Fig. 4 a). With the addition of modified hydrosodalite additive, the amount of $Ca(OH)_2$ and especially $CaCO_3$ crystals noticeably reduces. No traces of $CaCO_3$ are seen in the respective SEM image (Fig. 4b) while the amount of $Ca(OH)_2$ is much less. The last SEM image (Fig. 4c) shows that newly formed calcium hydrosilicate C–S–H compounds have small needle-shaped crystals and calcium aluminium silicate hydrate preparations give apparently single phase preparations with relatively coarse hexagonal plate morphology.

We may state that modified hydrosodalite additive stimulates the formation of hydrosilicates.

Properties of hardened cement paste

Zeolites, similar to modified hydrosodalite, feature a high content of active SiO_2 and Al_2O_3 . During the reaction of $Ca(OH)_2$ with modified hydrosodalite, the strength and density of the hardened cement paste is increased in the manner similar to other puzzolanic substances. The strength development in hardened cement paste samples is mainly dependent on the hydration rate of clinker, while in cement-modified hydrosodalite systems it depends on the combination of cement hydration and the pozzolanic activity of zeolite.

The modified zeolite additive added at 5% slightly reduces the compressive strength of hardened cement paste samples; however, with the increase of the additive content from 5 to 20%, the compressive strength slightly increases. These changes were observed after 3,7 and 28 days of hardening. The highest compressive strength was observed in samples with 15% of modified hydrosodalite additive. When 15% of modified hydrosodalite additive is added to the cement paste, the early compressive strength (after 3 and 7 days of hardening) becomes higher, whereas the compressive strength without the admixture is lower compared to the strength observed with zeolite admixture (Fig. 5). Characteristics of sample density are similar to strength characteristics. The density of hardened cement paste slightly decreases with addition of hydrosodalite to 5% after 3, 7 and 28 days of hardening (from 1924 to 1830 kg/m³) and remains stable or slightly increases with addition of more (10, 15 and 20%) hydrosodalite (1858-1873 kg/m³). It depends on cement hydration process and morphology of hydration products. A small content of hydrosodalite in the cement system reduces the density of hardened cement paste. Therefore, the higher content of



Fig. 5. Influence of the additive content on the density (*a*) and compressive strength (*b*) of hardened cement paste

hydrosodalite forms a second hydration product in pores and capillars of hardened cement paste, according to hydraulic activity.

The modified hydrosodalite possessed great surface and water absorption ability and the early strength of hardened cement paste might be decreased when zeolite percentage increases. These results were confirmed by other investigations of zeolite usage in cement pastes [9]. The higher content (more than 5%) of modified zeolites increases the strength of hardened Portlad cement paste for the high hydraulic activity of hydrosodalite. The changes in compressing strength are closely related with changes in the density of hardened cement paste.

CONCLUSIONS

After modification of pure hydrosodalite with CaCl₂ and the exchange of ions, the crystal structure of this zeolite remained unchanged.

Pure and modified hydrosodalite demonstrates similar hydraulic activity: the hydraulic activity of pure hydrosodalite was 184 mg/g and the hydraulic activity of modified hydrosodalite was 193 mg/g throughout the testing time. On the average, pure hydrosodalite is capable to bind 5.18 mg of CaO during 24 hours, while modified hydrosodalite demonstrates a slightly lower CaO binding capacity of 5.02 mg. X-ray and thermal analyses have demonstrated that the increasing content of the hydrosodalite additive from 0 to 20% in the mineral composition of the hardened cement paste reduces the content of non-carbonated $Ca(OH)_2$ from 12 to 9% after 3 days of hydration and from 15 to 8% after 28 days.

Due to the presence of zeolitic water in hydration process, the additive of modified zeolite added at 5% slightly reduces the compressive strength of hardened cement paste samples, whereas the increased content of the admixture from 5 to 20% slightly increases the compressive strength in the early hardening stage (3 days). The highest compressive strength was observed in samples with 15% of modified hydrosodalite additive. The increase of strength is related to the impact of SiO₂ and Al₂O₃ presented in modified hydrosodalite.

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CEMENTO KOMPOZICIJOS SU MODIFIKUOTU HIDROSODALITU

Santrauka

Hidrosodalitas susintetintas žemos temperatūros sintezės būdu (105 °C) nemaišomose suspensijose iš pradinių medžiagų: NaOH, Al(OH), amorfinio SiO, · nH,O ir H,O. Atlikus minėto hidrosodalito modifikavimą jonų mainų būdu, jo sudėtyje Na+ jonų sumažėjo, o Ca2+ jonų padaugėjo. Dėl didelio hidrosodalito puculaninio aktyvumo ir hidroaliuminatinių fazių susidarymo cementiniame akmenyje jis turi teigiamą efektą cementinio akmens stipriui gniuždant, ypač cemento hidratacijos pradžioje. Tarp šio modifikuoto hidrosodalto ir cemento galėjo vykti puculaninė reakcija, dėl kurios esant mažesniam kiekiui cementinių bandinių stipris šiek tiek sumažėjo. Priedo kiekis turėjo skirtingą įtaką cementinio akmens stiprumui. Modifikuoto ceolito 5 % priedas menkai sumažina cementinio akmens bandinių stiprį gniuždant, tačiau naudojant minėto priedo daugiau (nuo 5 iki 20 %), stipris šiek tiek padidėja. Didžiausią stiprį gniuždant turėjo bandiniai su 15 % modifikuoto hidrosodalito priedu. Bandinių stiprio padidėjimas gali būti susijęs su aktyviu SiO, ir Al₂O₃, esančiu modifikuotame hidrosodalite.