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# Influence of Zeolite Additive on the Properties of Plaster Used for External Walls from Autoclaved Aerated Concrete

**Georgijus SEZEMANAS [1] *, Marijonas SINICA [1], Petro ZACHARČENKO [2], Natalija PIVENJ [2], Donatas MIKULSKIS [1], Modestas KLIGYS [1]**

[1] *Scientific Institute of Thermal Insulation, Vilnius Gediminas Technical University,*
*Linkmenų 28, LT-08217Vilnius, , Lithuania*
[2] *Kiev National University of Building and Architecture, Voduchoflotskij prospect 31, Kiev, UA 03680, Ukraine*

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Enclosures that had been built from elements of autoclaved aerated concrete (AAC) often are covered with plasters. Some adhesion problems between plasters and surface exist during the mechanized covering of surfaces of the mentioned enclosures with plaster. Some dispersible additives (vinyl acetate polymer or derivatives of acrylic acid esters) are used to improve the adhesion. Another problem is the increased water absorption of AAC elements, therefore water retarding additives (cellulose esters) are used in the mixture of plasters. Due to insufficient amount of water in the mixture of plasters, the hydration of cement minerals slows down.

Influence of the natural zeolite – clinoptilolite on the properties of plaster is analyzed in this work. It was found that this additive effectively absorbs water and thus, ensures adequate hydration of cement minerals. When in the mixture of plaster the sand is changed by clinoptilolite in amount of 15 % the structure slightly compacts, the compressive and flexural strength of hardened plaster increase by 47 % and 12 % respectively and the adhesion to a surface of the AAC element increases by 44 %. Drying shrinkage, water vapor resistance factor and coefficient of capillary absorption remain unchanged.

*Keywords*: plaster, clinoptilolite, autoclaved aerated concrete, compressive and flexural strength, adhesion, drying shrinkage, water vapor permeability, capillary absorption.

## INTRODUCTION

It is known that owing to high degree of porosity (60 % ÷ 90 %) in aerated autoclaved concrete (AAC) samples soaked in water, the moisture reaches up to 76 % mass of product [1 – 3]. It was established [4, 5] that even a low content of adsorbed water (up to some per cent) decreases noticeably the properties of AAC products (lower mechanical strength and freeze resistance, higher heat conductivity). Therefore, AAC small blocks used for building of enclosures should be protected from the atmospheric impact (rain) by special sheets or plasters [4, 6 – 8]. Plasters acquire specific properties from polymeric additives, hydrophobity from salts of fatty acids (e.g. calcium or zinc stearate) [9, 10], while good adhesion is ensured by so-called redispersive additives (mostly vinyl acetate polymeric formations or esthers of acrylic acid) [11, 12]. Fibrous additives decrease drying shrinkage deformations, i. e. they protect plaster from cracking [13 – 14]. Air-entraining additives (AEA) reduce density and vapor resistance coefficient of plaster and improve adhesion to base and increase freeze resistance [15, 16]. The porous structure of AAC stimulates migration of water from the applied layer of plaster mix inside AAC blocks, causing slowed-down hydration of Portland cement minerals contained in plaster, what affects the hardening of plaster and its mechanical strength [17]. To the aim of improving of the process, the water-retaining additives are added to plasters (the main formations of methylcellulose) [18, 19]. Such a role can be also played by natural zeolite additives, which are widely used in other Portland cement-based products and concretes [20 – 23]. Thank to their unique hollow structure, zeolites are distinguished for good sorption properties and can accumulate in themselves a sufficient amount of water required for full hydration of cement minerals [24, 25]. On the other hand, zeolites can be characterized by increased adhesion to various surfaces of building materials [26, 27] and can replace (partially or fully) redispersive polymeric additives used for this purpose.

The references [27, 28] present the data on effectiveness of zeolite additives in the production of dry mixes; nevertheless, no data are available on their impact on plasters meant for coating of AAC products.

The purpose of this study is to investigate the impact of natural zeolite (clinoptilolite) on operating properties of plaster meant for coating of AAC walls from outside and to correct the recipes of dry plaster mixes with zeolite additives.

## MATERIALS AND TEST METHODS

For the work, dry plaster mixes prepared under laboratory conditions were used. To this aim, the following materials were used:

– sand from the Matuizų quarry Giraitė (granulometric composition: fraction (1 ÷ 2) mm – 2.1 %, (0.5 ÷ 1) mm – 7.9 %, (0.25 ÷ 0.5) mm – 34.5 %, (0.125 ÷ 0.25) mm – 45.5 %, (0.063 ÷ 0.125) mm – 7.6 %, (<0.063) mm – 2.4 %), corresponding to [29];

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[*] Corresponding author. Tel.: +370-682-37630; fax.: +370-5-275-2642.
E-mail address: *georgijus.sezemanas@vgtu.lt* (G. Sezemanas)

– Portland cement of mark CEMII/A-L 42.5N, corresponding to the requirements of [30];

– ground slacked lime from the joint stock company “Naujasis kalcitas”, according to the requirements of [31].

There were also used the special additives to improve properties, such as calcium stearate (hydrophobizer); methylcellulose (water-retaining additive); VINNAPAS RE5011 L (vinylacetate and ethylene copolymer powder, the redispersive additive for improvement of adhesion to base). As an air-entraining additive, powder UFAPORE CC85 (content of active materials 85 %, pH of 1.0 % solution 10.5; ground natural zeolite rock from the Transcarpathian Zeolite Factory (specific surface 4500 $m^2$/kg), 77 % clinoptilolite content (certificate ISO 9001:2000 for this product is granted to the enterprise).

The chemical composition of aggregates and binding materials is provided in Table 1.

**Table 1.** Chemical composition of raw materials

| Composition | Raw materials, % | | | |
|---|---|---|---|---|
| | Portland cement | Lime | Sand | Zeolite rock |
| $SiO_2$ | 22.41 | 4.04 | 90.40 | 71.50* |
| $Al_2O_3$ | 4.20 | 2.22 | 4.02 | 13.12 |
| $Fe_2O_3$ | 4.76 | 0.87 | 0.65 | 0.94 |
| $TiO_2$ | – | – | – | 0.25 |
| CaO | 63.0 | 89.23 | 2.05 | 2.10 |
| MgO | 2.5 | 2.37 | 0.49 | – |
| MnO | – | – | – | 1.07 |
| $K_2O+Na_2O$ | – | 0.36 | 1.34 | 5.0 |
| $SO_3$ | 1.65 | – | – | – |
| LOI, % | 1.33 | 0.86 | 1.02 | 6.01 |

*Note: Content of reactive $SiO_2$ in zeolite rock 55.21 %.

Before preparation of dry mixtures, lime was slaked by isothermal treatment for 0.5 h in the laboratory autoclave with capacity of 100 liters (saturated water vapor pressure 0.4 MPa). When lime was cooled down to room temperature, all dry components were mixed for 10 min by VARI mixer BEAR in the laboratory mixer of 3.0 liters (speed 60 rpm). The composition of mixes, as provided in Table 2, were selected based on the references [12, 16, 17].

During preparation of plaster grouts, the dry mixes were mixed with water for 5 min. The speed of mixing during first two minutes was 60 rpm, and during remaining three minutes 120 rpm. The water amount was taken considering the flowability of grout determined by means of flow table [33] until the diameter of sample reached 160 mm ±5 mm. The hardening and conditioning of molded grouts proceeded in the following mode: 24 hours in moulds and following 48 h in demolded state at relative air humidity of 95 % ±3 % and temperature of 20 °C ±3 °C, then starting from the 4th day to completion of test, at relative air humidity of 65 % ±5 % and same temperature. The density of hardened plasters, as well as their compressive and flexural strengths and capillary absorption coefficient were determined according to the standard methods [34 – 36].

The compressive and flexural strengths were determined after 7, 28, 60 and 90 day-exposure by press Tinius Olsen H200kU at load speed of 200 N/s and load measurement accuracy ±0.5 % of applied load from 0.2 % to 100 % capacity. For determination of force of adhesion to base of plaster, freshly prepared plaster grout was applied to surface of AAC blocks (density 500 kg/m³, compressive strength class 2.5) treated by priming (4.0 % polyvinylacetate emulsion solution). The applied plaster grout layer was levelled up to ~8 mm thick, then after 2 h was covered by polyethylene film and left for hardening during 7, 28, 60 or 90 days at temperature of 20 °C ±3 °C. The force of adhesion to AAC base was measured by device CONTROLS according to standard methods [37] (Fig. 1).

**Table 2.** Compositions of plaster mixes

| Components | Compositions, % | | | | |
|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5* |
| Portland cement | 20.0 | 20.0 | 20.0 | 20.0 | 16.0 |
| Slaked lime | 7.2 | 7.2 | 7.2 | 7.2 | 7.2 |
| Sand | 68.53 | 68.53 | 58.53 | 53.53 | 60.57 |
| Zeolite | 0.0 | 5.0 | 10.0 | 15.0 | 15.0 |
| Vinnapas RE 5011L | 4.0 | 4.0 | 4.0 | 4.0 | 1.0 |
| Methylcellulose | 0.05 | 0.05 | 0.05 | 0.05 | 0.0 |
| AEA | 0.02 | 0.02 | 0.02 | 0.02 | 0.03 |
| Calcium stearate | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |

*Note: Composition 5 was selected upon evaluation of properties of other four hardened plasters according to the standard [32].

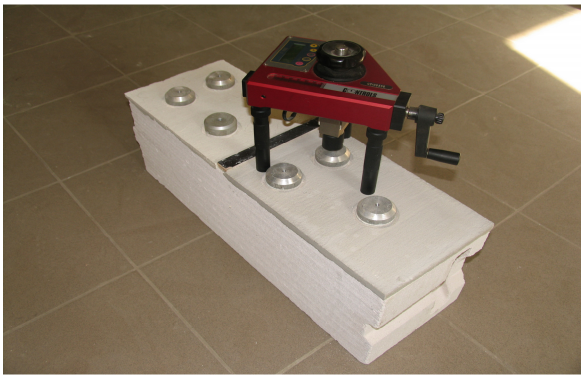

**Fig. 1.** Device CONTROLS meant for testing of adhesion of plaster to AAC base [37]

The drying shrinkage deformations were measured by device MITUTYO ID-C112B based on standard requirements [38]. The granulometric composition of sand was determined by device HAVER EML 200 DIGITAL T according to [39]. The water vapor permeability was determined according to the standard [40] after testing of 3 samples of mix of each composition, sized 18 cm in diameter and 2.0 cm thick. The climatic conditions of temperature mode 23-50/95 (i. e. environment temperature 23 °C, relative air humidity over sample 50 %; relative air humidity under sample 95 %).

For analysis of surface of hardened plasters and contact zone with base, the computerized optical microscope MOTIC with digital camera (magnification up to ×100) was used.

The X-ray structural testing was performed by diffractometer DRON-7 with copper anticathode, nickel filter, anode voltage of 30 kV, anode current of 12 mA, goniometric slots (0.5; 1; 1.5) mm. For decoding of peaks, ICDD database was used.

## RESULTS AND DISCUSSION

While mixing of dry mixes with water (the compositions are provided in Table 2), it was observed that along with increase in content of zeolite additive in the mix (at same consistence of mix), the water to solids ratio (W/S) is also increasing from 0.20 to 0.30. The density of hardened grout also grows by 7.6 % (Fig. 2).

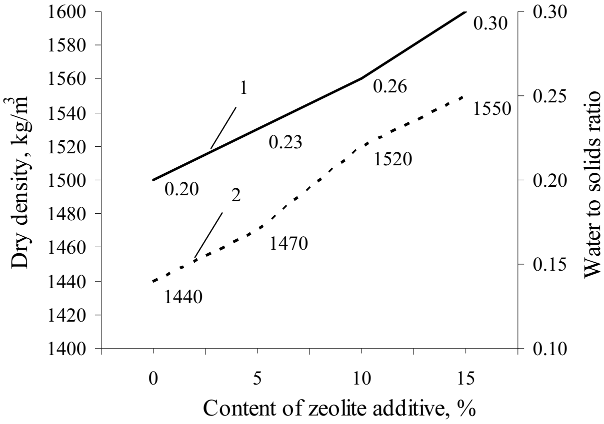

**Fig. 2.** Impact of zeolite additive on W/S (1) and on dry density of hardened plaster (2)

The increase in W/S is predetermined by hollow structure and excellent sorption ability of zeolites. Upon partial replacement of sand by a fine-dispersed pozzolanic additive, in our case clinoptilolite, the structure of hardened plaster is getting more compact. During the pozzolanic reaction between lime (portlandite, the evolved cement mineral, plus lime contained in composition of grout) and amorphous $SiO_2$ (which makes from 50 % to 60 % in clinoptilolite rock [41, 42]), gel C-S-H calcium hydrosilicate forms and this hydrosilicate increases the strength of plaster during the hardening of grout [27–28, 41].

The relationship between compressive strength of hardened plaster and content of clinoptilolite additive is provided in Fig. 3.

As we can see, the effect of the pozzolanic reaction manifests itself after 60 days and later. This can be explained by slow change of phases in gel calcium hydrosilicates. For instance, in the 4 sample of grout mix 4 where the content of zeolite additive is the highest (15 %), the compressive strength after 90 days was even by 52 % higher than that of reference sample (Fig. 3, curve 1, 4). After 28 days only the index of compression for grout 4 almost concided with that of reference sample (0.27 N/mm$^2$ and 0.26 N/mm$^2$, respectively), while the strength of other samples (with zeolite additive content 5 % and 10 %) was lower by 8 % and 16 % than that of reference sample. This coincides with the results of investigations of other scientists [27, 41].

The same tendency is seen with flexural strength (Fig. 4). Tests results show that the increase in flexural strength of hardened samples subject to zeolite content is not so considerable as in the case of compressive strength. For instance, the flexural strength of grout mix sample 4 after 90 day exposure increased by 12 % versus that of reference sample (Fig. 3, curves 1, 4), while in other samples it increased less, by 2.2 % (Fig. 4, curve 2) and by 6.0 % (Fig. 4, curve 3).

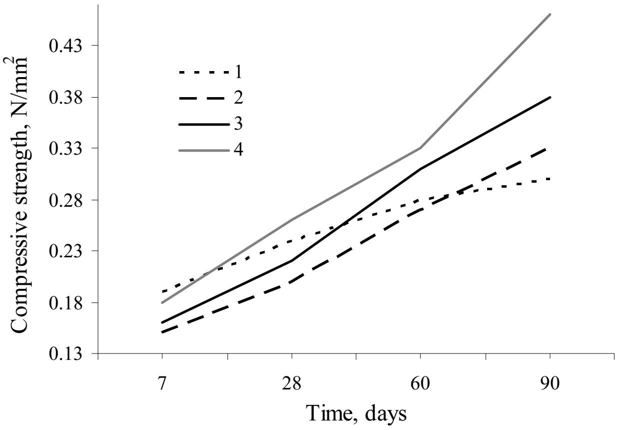

**Fig. 3.** Variation of compressive srength in plaster samples in view of exposure time (the marking of curves corresponds to that of compositions of mixes provided in Table 2)

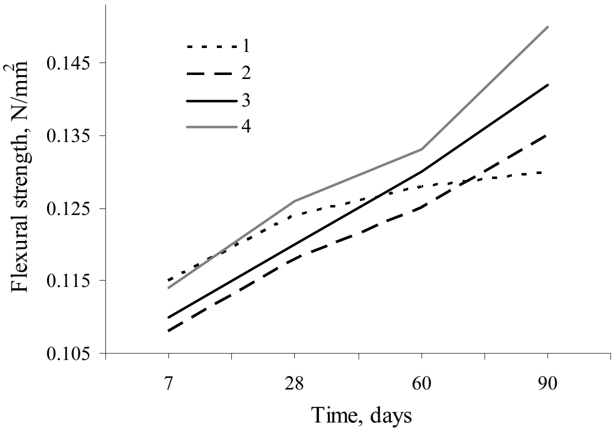

**Fig. 4.** Variation of flexural strength in plaster samples in view of exposure time (the marking of curves coresponds to that of compositions of mixes provided in Table 2)

Other very important factor for hardened plaster grouts is adhesion to base. The results of investigations show that along with increase in content of zeolite additives in mixes and exposure time, the adhesive force is increasing respectively (Fig. 5).

The data of Fig. 5 shows that the adhesive force increasing more rapidly in first 60 days of exposure of samples, afterwards the process is slowing down. The references [26, 28] stress that the zeolite additive increases the force of adhesion to base of grouts. However, the authors explain this process differently, i. e. the reference [28] stresses on the influence of montmorillonite admixtures contained in zeolite rock, while another reference [26] underlines the impact of vacuum effect, which appears after water migration from hollows of zeolite structure to hydrating cement minerals. Evidently, both factors are of importance to the force of adhesion to base, nevertheless, to our mind, a third variant is possible as well. It is related to gel C-S-H, which forms in the zeolite structure [41, 42] and the zone of adhesion of plaster to AAC base, and penetrates into surface pores of AAC structure and to its hardening.

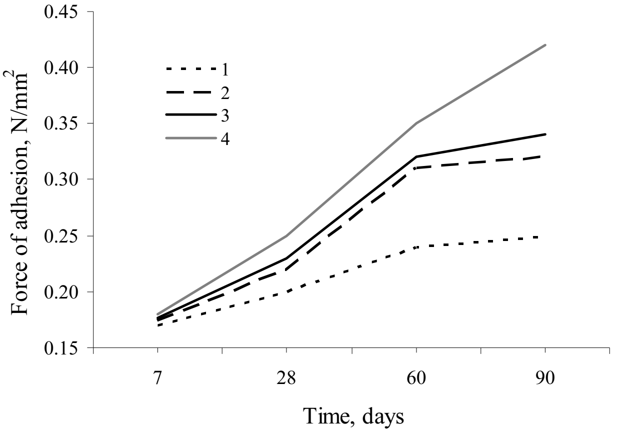

**Fig. 5.** Relationship between variation of force of adhesion to base in hardened plasters and exposure time (the marking of curves corresponds to that of compositions of mixes provided in Table 2)

Fig. 6 demonstrates the samples of AAC base and of plaster torn off it after 90 day exposure, as well as the zone of adhesion of plaster to base (section).

We can see that at maximal zeolite content in the mix (15 %), cracks are appearing on surface of plaster, however, irrespective of that, the adhesive force of plaster is maximal (0.42 N/mm$^2$, Fig. 5, curve 4). Such value of adhesion is conditioned by close contact of adhesive zone (Fig. 6, d), therefore, on surface of torn off plaster samples (Fig. 6, c) the odds and ends of AAC are visible. This is not seen in the photos a and b, Fig. 6 where the surface of torn off samples is clear, i. e. the tearing from the AAC base occurred in the contact zone. In this case the contact between plaster and AAC is not very close, therefore, the adhesive force value reaches only 0.25 N/mm$^2$.

The cracks in the layer of plaster (Fig. 6, c) appeared only in the samples with the highest content of zeolite additive (15 %) and only at the contact with AAC surface. No cracks were observed on samples of pure plaster meant for determination of other properties (e. g. prisms sized (160 × 40 × 40) mm for determination of mechanical strength).

In the standard for plaster grout [33], the determination of drying deformations of mortar is not regulated. However, to the aim of revealing of impact of zeolite additive on shrinkage of plaster, the measurements of variation in length of hardened plaster samples were carried out. The data are provided in Fig. 7.

The data of Fig. 7 shows that along with increase of content of zeolite additive in mixes, the drying deformation is growing as well, and its values stablize approximately after 65 ÷ 70 days. At the maximal content of zeolite additive in the mix (Fig. 7, curve 4), the drying shrinkage of samples becomes extremely evident and after 90 days makes even 4.2 mm/m and with 10 % additive the analogical index reaches the value of 2.5 mm/m only (Fig. 7, curve 3). The higher drying deformations of plaster samples with zeolite additive versus reference ones are preconditioned by higher drying shrinkage of gel C-S-H hydrosilicate, which in the case of insufficient content of aggregate (quartz sand) of suitable granulometric composition, affects the shrinkage of the whole sample.

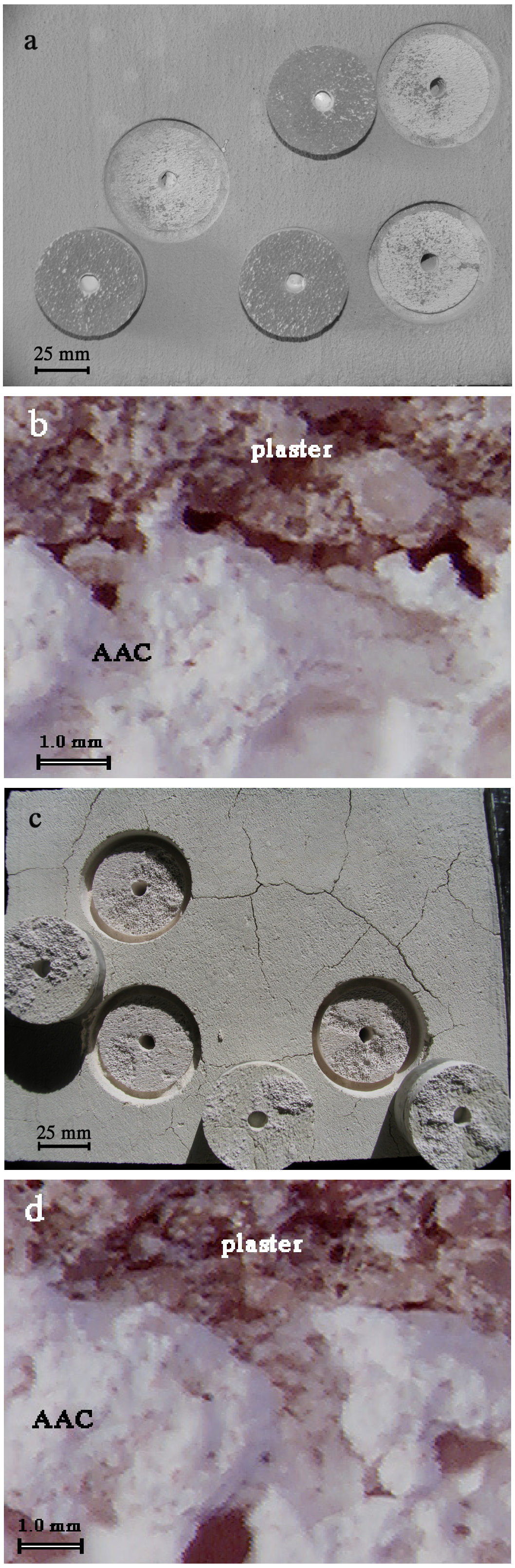

**Fig. 6.** Samples after measuring of force of adhesion to AAC base (a and c) and section of adhesion zone (b and d). a and b – reference samples, c and d – samples with 15 % zeolite additive

At 15 % zeolite additive, the content of sand in the samples is minimal (53.5 %, Table 2), therefore, if the structure has no firm carcass made of sand grains, then in the plaster layer on AAC surface cracks are appearing (Fig. 6, c).

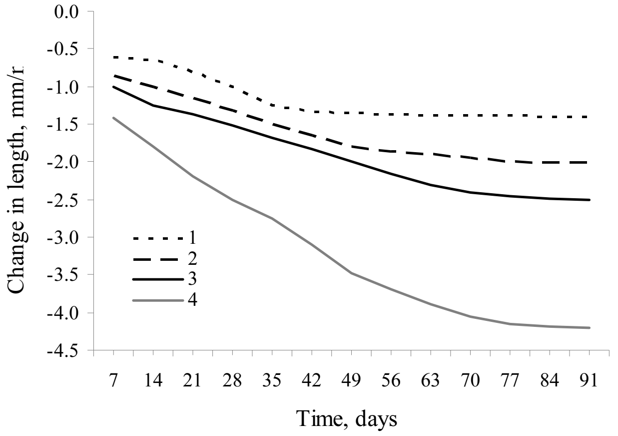

**Fig. 7.** Relationship between drying shrinkage variations and time in hardened plaster samples (the marking of curves corresponds to that of compositions of mixes provided in Table 2)

The received data correlates well with other scientists' results of investigations [43]. Because of this process, the water vapor permeability of hardened samples decreases as well. The gel C-S-H formations generated in the structure of plaster and in the hollows of zeolite additive due to the pozzolanic reaction prevent water vapor from migrating through the layer of plaster. The impact of zeolite additive on water vapor resistance factor is shown in Fig. 8.

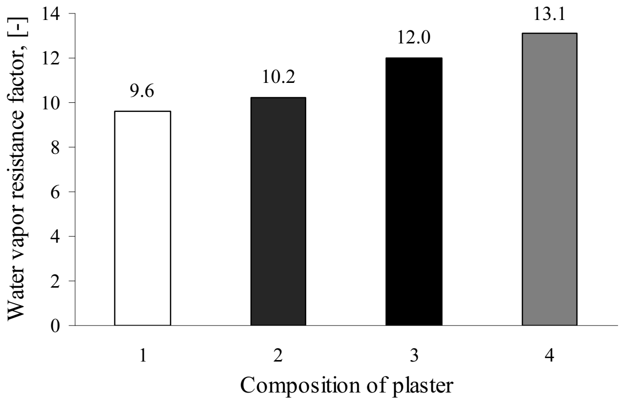

**Fig. 8.** Impact of zeolite additive on water vapor resistance factor of hardened plaster

The data of Fig. 8 shows that together with growth of content of zeolite additive in plaster mixes and with increased formation of gel C-S-H, the water vapor permeability decreases (the vapor resistance factor increases). At 15 % zeolite additive, this value is 13.1, while that of reference sample 9.6.

During testing of capillary water absorption of plaster samples, it was observed that the zeolite content has no effect on this index, since the low water absorption value is preconditioned by the hydrophobic additive, calcium stearate, contained in all samples (Fig. 9). We can see that after 90-minute contact with water, the coefficient of capillary absorption changes insignificantly, from 0.05 kg/m$^2$min$^{0.5}$ to 0.06 kg/m$^2$min$^{0.5}$.

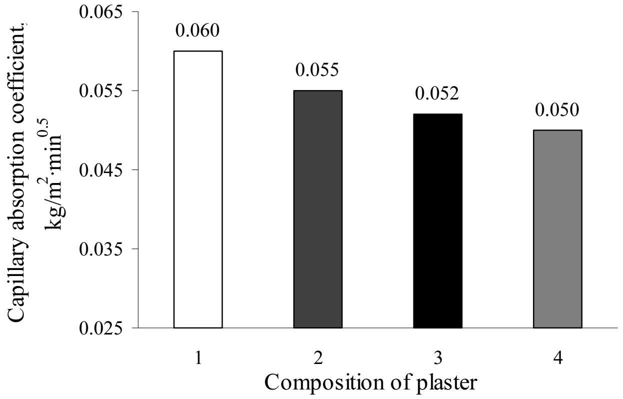

**Fig. 9.** Effect of zeolite additive on capillary absorption coefficient (the marking corresponds to that of compositions of mixes provided in Table 2)

Comparing the macrostructure of samples of hardened plasters (Fig. 10, a and b), it is obvious that the samples with zeolite additive are denser, i. e. the number and size of pores in them is lower.

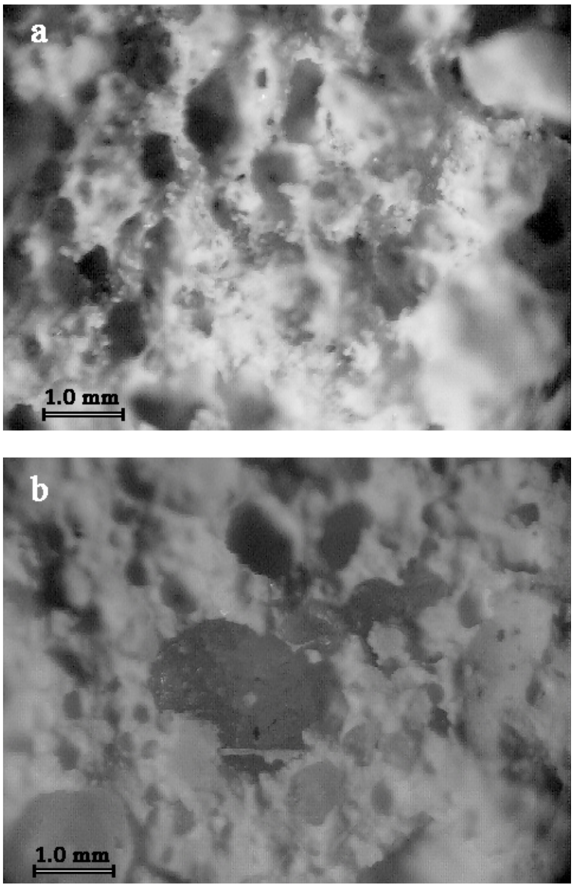

**Fig. 10.** Macrostructure of plaster: a – reference sample, b – sample with 15 % zeolite additive

The X-ray structural tests of samples after 90-day hardening showed that in the plaster sample with 15 % zeolite additive the content of portlandite is lower, due to the pozzolanic reaction proceeding more intensely (Fig. 11, b) than in the reference sample (Fig. 11, a).

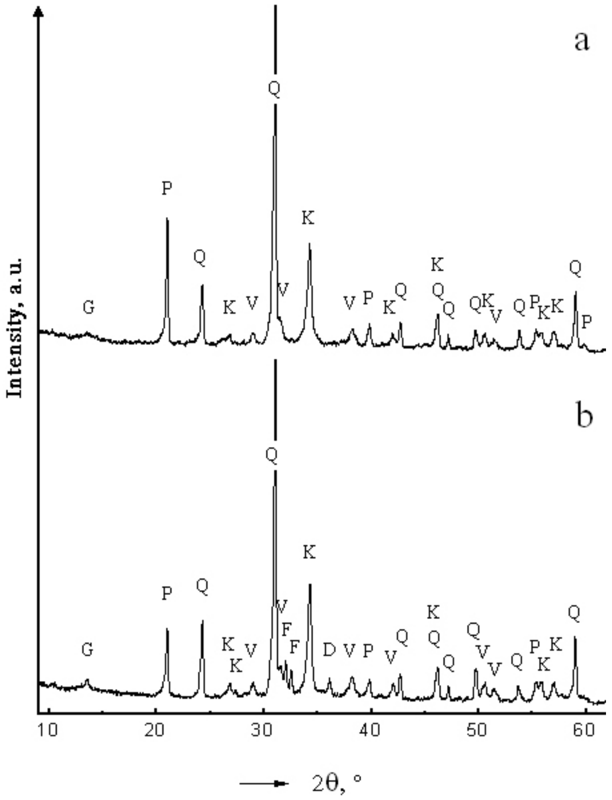

**Fig. 11.** X-rays of plaster samples, which hardened for 90 days: a – reference sample, b – sample with 15 % zeolite additive. G – gypsum, Q – quartz, V – vaterite, K – calcite, P – portlandite, D – dolomite, F – feldspar

Upon completion of investigations as to properties and structure of samples with clinoptilolite additive and evaluation of all nuances, both positive (increased mechanical strength and adhesion to AAC surface) and negative ones (increased drying shrinkage, lower water vapor permeability) related to impact of additive on properties of plaster, the correction of composition of plaster was performed (Table 2). To eliminate the negative impact of zeolite on drying shrinkage, the granulometric composition of sand was changed. Sand of fine fraction (<0.125 mm) was replaced by larger fraction (0.5 ÷ 1.0) mm. The content of replaced sand varied from 5 % to 10 %. At correlation of 5 % sand content, the shrinkage of plaster resulted in cracks on surface coated. The 10 % sand replacement eliminated cracking. At replacement of 10 % fine sand by larger fraction, the better strength indices for plaster samples were received as well.

The water vapor perrmeability was increased by addition of AEA from 0.02 % to 0.03 %. The AEA addition decreased the density of plaster samples from 1590 kg/m$^3$ to 1420 kg/m$^3$ what reduced the water vapor resistance factor from 13.1 to 9.7.

Parallel with the mentioned above, the strength indices of plaster were reduced, because the zeolite additive considerably increased the mechanical properties and plaster adhesion to AAC surface. For correction of composition of plaster mixes, we gradually decreased the content of methylcellulose, from 0.05 % to 0.0 %. It was established that 15 % zeolite additive can fully replace methylcellulose and reduce the content of additive VINNAPAS by 75 %.

Beside reduction of content of polymeric additives, the content of binder (Portland cement) was cut down as well, by from 10 % to 20 %. The 20 % reduction ensured the strength properties of plaster close to the reference one. The values of samples of this mix after 90-day hardening are provided in Table 3.

**Table 3.** Characteristics of hardened samples from reference and corrected plaster mixes

| Characteristics | Composition of plaster | |
|---|---|---|
| | 1 (reference) | 5 (corrected) |
| Density in kg/m$^3$ | 1440 | 1420 |
| Compressive strength in N/mm$^2$ | 0.3 | 0.44 |
| Flexural strength in N/mm$^2$ | 0.13 | 0.145 |
| Adhesion to base in N/mm$^2$ | 0.25 | 0.34 |
| Drying shrinkage in mm/m | 1.4 | 1.4 |
| Water vapor resistance factor [-] | 9.6 | 9.7 |
| Capillary absorption coefficient in kg/m$^2$min$^{05}$ | 0.06 | 0.06 |

Irrespective of the fact that the composition of corrected plaster mix contains less cement and additive VINNAPAS and no methylcellulose, the data of Table 3 show that the properties of corrected and reference plaster are similar and the values of strength and adhesion to AAC base are slightly better in the corrected mix.

## CONCLUSIONS

1. It was established that the clinoptilolite additive in the composition of plaster reacts with lime contained in the mix, as well as with portlandite, which evolves during hydration of cement minerals, and as doing so, it plays a role of a pozzolanic material. This is confirmed by the X-ray analysis of plaster mixes. In connection with the abovementioned, the strength properties of hardened plaster samples increase what allows reducing the content of Portland cement by one fifth (20 %).

2. The clinoptilolite additive is famous for good sorption properties, due to its unique structure, and can be used in dry mixes as a water-retaining additive (i. e. it accumulates water in itself and gradually supplies the hydrating cement minerals by it), thus enabling to give up fully traditionally used methylcellulose.

3. The clinoptilolite additive ensures good adhesion to AAC base, i. e. it helps to achieve a higher force of adhesion to rendered base and 75 % reduction of VINNAPAS used in the production of dry mixes.

4. The higher drying deformations in plasters with clinoptilolite can be reduced by correction of granulometry of quartz sand in the composition of plaster, by replacing 10 % sand of finer fraction (up to 0.125 mm) by sand of coarser fraction (0.5 ÷ 1.0) mm.

5. The additional content of gel C-S-H calcium hydrosilicate formed due to the pozzolanic reaction of zeolite with lime causes a more compact structure of plaster and worsens the water vapor permeability. This shortcoming can be avoided by increasing AEA content in plaster mixes, from 0.02 % to 0.03 %.

6. After investigation of the impact of clinoptilolite additive on the operating properties of plaster meant for

coating of AAC walls from outside, corrected composition of dry plaster mix was proposed (in mass %): Portland cement – 16, ground slacked lime – 7.2, sand – 60.57, clinoptilolite – 15, VINNAPAS – 1.0, AEA – 0.03 and calcium stearate – 0.2.

## *Acknowledgments*

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