

## EFFECT OF ZEOLITE NANOSTRUCTURE ADDITIVE ON MORTAR HARDENING PROCESS

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**Abstract:** For many years lime mortar has been an indispensable building material used in all countries of the world. The position of lime mortar for restoration work has especially increased. Since one of the main factors of durability of historical and cultural monuments is the quality of mortar, it is necessary to select such components of mortar composition that determine its quality and durability. One of the most common methods of modifying the structure of composite building materials is the introduction of highly active micro- and nano-additives into their composition. Natural zeolites are used as additives to cement. Zeolites have attracted the attention of modern builders because of their unique mineralogical, chemical, and macroscopic properties. We have carried out a study of the influence of mineral additives (natural zeolite-clinoptilolite from different deposits) on the strength of lime mortar. Its composition, structure, and mechanical properties were determined using physicochemical methods of research.

**Keywords:** Physico-chemical research methods, zeolite clinoptilolite, lime, lime mortar, sand

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### 1. Introduction

The care and protection of cultural and historical architectural monuments and their preservation for future generations is an obligatory, priority, and responsible task of every civilized nation. A scientific approach and research of the causes of damage to monuments and their subsequent removal and restoration require great carefulness and precision.

Lime mortar is the simplest three-component building material that has been used for centuries in almost all countries of the civilized world. It was the main binding material in construction, on the quality of which the longevity of the monument largely depended.

Lime is mainly obtained by burning calcium and magnesium carbonate rocks (limestone) until the carbonaceous part is completely removed [1-3].

Depending on the degree of processing,

lime can be of two types: quicklime or slaked lime (hydrated). Products made from quicklime are 4–5 times stronger than those made from slaked lime [1].

The second most important component of lime mortar is sand, which is a mixture of silicates and aluminosilicates [4-6].

The class of silicates, which includes about 800 minerals and constitutes 75-90% of the Earth's crust, contains mainly not very many elements, namely Si, Al, Na, K, Ca, Mg, and Fe and in smaller amounts Li, Ti, Cu, and Zn. Anions of simple and complex groups ([BO<sub>3</sub>], [O], [OH], [F], [Cl]) as well as crystallization, adsorption, and interlayer waters also play an important role in the structure of silicates [7].

Aluminum has a special role in the structure of silicates, as it can be cationic, anionic, or play both roles simultaneously [7].

The fundamental structural unit of silicates is the silicon–oxygen tetrahedron,  $[\text{SiO}_4]^-$ . These tetrahedra are linked together through shared oxygen atoms, leading to polymerization and the formation of anionic frameworks of varying complexity. The nature of these connections forms the basis for the modern classification of silicates.

The hardness of silicates depends significantly on the ionic radius of the main cations and the type of chemical bond. Silicates built with cations with small ionic radii have high strengths [8].

After adding an appropriate amount of the third component—water—to the lime and sand mixture, the mortar hardening process proceeds in several directions. The first is the evaporation of chemically unbound water; the second is the interaction of calcium hydrate with carbon dioxide in the air, in which calcium hydrate gradually loses chemically bound water and calcium carbonate is formed – a solid salt of limestone. Absorption of carbon dioxide occurs rapidly, and since this process occurs mainly on the surface of the mortar, a crust is formed that

prevents the reaction from continuing in this direction and the complete evaporation of chemically bound water. Therefore, this process takes decades [5]. In addition to the carbonation process, lime-free particles interact with clay substances, actively decompose them, and form gel-like hydrosilicates, which gradually lose moisture and act as binders for various particles. For different aluminosilicates, this reaction proceeds at different rates and depends on the reaction medium. If the medium does not conform to the corresponding norm, the physical property of the mortar may change and it will not harden [5]. Therefore, in addition to the quality of lime, the chemical and phase composition of sand is of great importance in the mortar hardening process [4-6].

The aim of this research is to develop a high-strength, high-quality mortar suitable for both construction and restoration applications. To achieve this, we investigated mortar components with diverse chemical compositions and examined several mineral additives chosen for their distinctive structural characteristics.

## 2. Experimental part

**2.1 Material and methods.** In order to determine the optimal composition of the building mortar, sands of different phases and chemical compositions were investigated, and the best one was selected—it was yellow sand from the Sachkhere deposit. Lime was also selected taking into account its chemical composition.

The preparation of the desired fraction of the samples was carried out on the devices: laboratory jaw crusher (RAM 35, Rantek Brand, Turkey); electromagnetic vibrator-shaker (BIOBASE BK-TS200, China). Methods of chemical, X-ray diffractometric, IR spectroscopy, and petrographic analysis were used to study the chemical and mineralogical composition and structure of the objects of study.

Chemical analysis was carried out on a Spectroscout XEP-04 (Germany); X-ray diffractometry analysis was carried out on a Dron-4 device (Russia). The X-ray diffractometer is connected to a personal computer via the USB port multimeter AX-18B

and the corresponding PC software PC-Link, which allows the processing of experimental data in Excel format; infrared spectrometric analysis was carried out on an Agilent Cary 630 FTIR Spectrometer in the range of  $350\text{--}5000\text{ cm}^{-1}$  (USA). Samples were prepared in KBr tablets. Petrographic analysis was carried out on the device Optica B383 polarizing microscope.

To carry out physical and mechanical tests of lime mortar, construction samples were made, which were pre-sieved through a sieve of  $N_{2.5}$  mm. Water was added in portions. The mixture was heating up due to the heat generated during the reaction, and a delay was necessary. Then the samples were formed by vibration as cubes of  $2 \times 2 \times 2$  cm. Forms containing samples were stored at room temperature ( $20 \pm 2$ ) °C and 65% humidity. After two days and nights, the forms were opened and kept under the same conditions.

Physical and mechanical compression tests of the samples were carried out 28 days after molding. The tests were carried out on a 5-tonne laboratory hydraulic press.

Lime mortar was mainly manufactured according to European and Russian standards: European Standard - EN459-1:2015 Building Lime – Part 1: Definitions, specifications and conformity criteria; Russian standards – 1) GOST 28013-98 Construction solutions, 2) GOST 9179-2018 Construction lime.

Zeolites, like quartz and feldspars, belong to the subclass of framework silicates and have a similar structure [9-11]. They are crystalline hydrated framework aluminosilicates whose large-cell frameworks consist of [Si, Al]<sub>2</sub>O<sub>2</sub> tetrahedrons. Their crystal structure contains voids occupied by ions and water molecules, which can move freely. A certain freedom of movement enables ion exchange and reversible dehydration. They have the ability of molecular sieves and can actively participate in ion exchange reactions. A dehydrated zeolite is a microporous crystalline “cloud” in which the pore volume is 50% of the volume of the zeolite framework. The diameter of the incoming channels included in such a “cloud” ranges from

0.3 to 1.0 nm, which is due to their high molecular sieve, substitution, sorption and catalytic properties [9-11].

All over the world, zeolites have been used in construction since prehistoric times as a facing material and are still used today. At present, in many countries, natural zeolites are used as additives to cement. Of particular interest is their use in pozzolanic cements, because zeolite tuffs have pozzolanic properties [12-14].

Zeolites have attracted the attention of modern builders because of their unique mineralogical, chemical, and macroscopic properties, not just as zeolite-containing tuffs. Geylandite-clinoptilolite minerals are the most common zeolites in nature [9, 10].

For the purpose of structural modification of lime mortar, we used zeolite clinoptilolite from three different locations (Zemo Khandaki – Kaspi Municipality; Khekordzula – Mtskheta Municipality; Aspindza – Aspindza Municipality).

### 3. Results and its discussion

**3.1. The results of chemical analysis of samples.** Chemical analyses of the samples of

sand, lime, and zeolites used as additives are given in Table 1.

**Table 1.** Data from chemical analysis of the samples.

Constituent elements in the form of oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	MnO	TiO <sub>2</sub>	SO <sub>3</sub>
1	2	3	4	5	6	7	8	9	10	11
Sand light yellow Location of Sachkhere (Georgia)	62.66	10.26	1.23	0.68	3.09	0.29	1.98	0.03	0.34	0.41
Lime	0.32	0.09	56.60	0.14	-	0.14	0.09	0.01	0.01	-
Clinololite location of Khekordzula (Georgia)	55.28	10.17	3.53	1.15	1.46	0.14	1.52	0.01	0.01	0.35
Clinoptilolite location of Khandaki (Georgia)	46.84	9.78	7.08	1.54	1.99	0.05	3.97	0.04	0.56	0.24
Clinoptilolite location of Aspindza (Georgia)	54.79	11.26	1.85	2.26	0.78	2.25	2.92	0.01	0.43	0.23

The chemical composition of clinoptilolites used as additives is different. The SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio for clinoptilolite from the Khekordzula location is the highest – 5.44; for clinoptilolite from the Aspindza location, it is 4.87, and for clinoptilolite from the Khandaki location, it is 4.79. The cation composition of these zeolites is also different. The content of

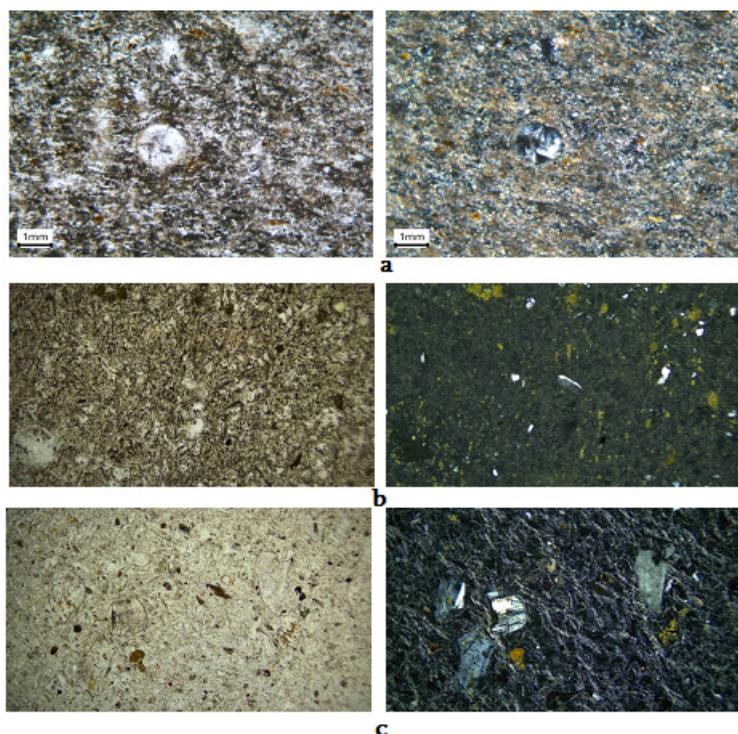
CaO and Fe<sub>2</sub>O<sub>3</sub> in the sample from the Khandaki deposit is high: 7.08 and 3.97%, respectively. The amount of CaO in the sample from the Khekordzula locality is 3.53%, and the amount of other cations is also low. The concentration of Na and Mg cations in clinoptilolite from Aspindza is 2.25 and 2.26%,

respectively, while the concentration of Ca is lower than in other clinoptilolite – 1.85%.

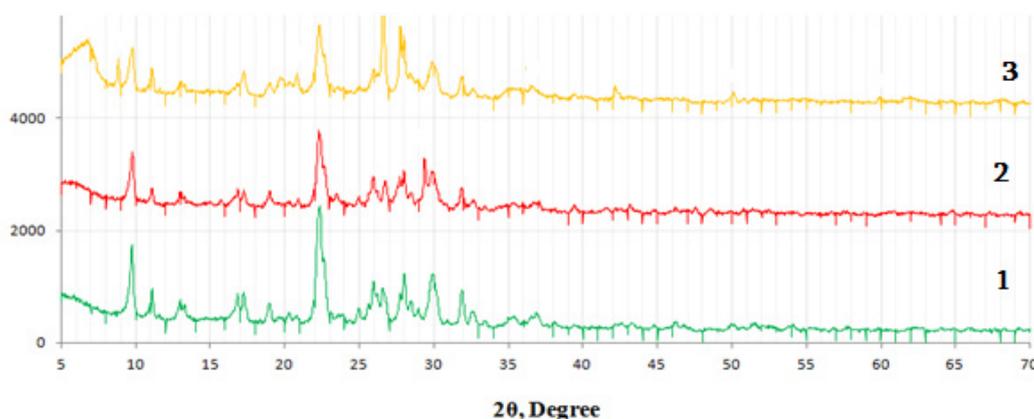
The humidity in the clinoptilolite of the Khekordzula location was 13%, and the loss of ignition was 12%; the humidity in the clinoptilolite of the Aspindza location was 14%, and the loss of ignition was 9%; the humidity in the clinoptilolite of the Khandaki location was 12%, and the loss of ignition was 14%. The humidity in the sand of the Sachkhere location was 10%, and the loss of ignition was 8%; the

humidity in the lime was 8%, and the loss of ignition was 33%.

**3.2. The results of petrographic analysis.** Petrographic and phase analysis of clinoptilolites showed that all three samples are mainly composed of isotropic zeolite mass, which contains quite a large number of carbonatized faunal grains, as well as rather large (up to 1 mm) plagioclase grains. There are also ore minerals (Fig. 1).



**Fig. 1.** Microphotograph of zeolites – in open and closed nicol, magnification 40x: **a** –Khandaki location; **b** – Khekordzula location; **c** – Aspindza location



**Fig. 2.** X-ray diffractograms of zeolite-clinoptilolite used as an additive: **1** – from Khekordzula location; **2** – from Khandaki location; **3** – from Aspindza location

The phase composition of clinoptilolites in the samples is as follows: Hecordzula deposit – clinoptilolite – 90%, plagioclase fragments – 5%, chlorite – 3%, ore minerals – 1%, etc.

Aspindza deposit – clinoptilolite – 75%, plagioclase clasts – 15%, biotite – 3%, chlorite – 1%, etc. Handaki deposit – clinoptilolite – 70%, plagioclase clasts – 10%, carbonates – 7%, chlorites - 3%, biotite – 1%, etc.

**3.3. The results of both X-ray diffractometric analysis and IR-spectroscopy.** X-ray diffraction studies of clinoptilolites used as additives showed that their diffractograms fully correspond to the literature data [10], although in some points there are low-intensity peaks characteristic of other impurity minerals (Fig. 2).

In addition to the peaks characteristic of clinoptilolite structure, the diffractograms also

show low-intensity quartz peaks, as well as calcite peaks and an X-ray amorphous phase, which indicates the presence of clay impurities in the zeolites.

The study of IR spectra of clinoptilolites from different locations used as mortar additives showed that all three spectra are practically identical. Of particular importance in these spectra are the presence of intertetrahedral vibration bands: valence vibrations in the range of 1205–1208  $\text{cm}^{-1}$ , and deformation vibrations in the range of 523–664  $\text{cm}^{-1}$ . Their presence characterizes the zeolite structure in aluminosilicates [15] (Table 2).

**Table 2.** Characterization of IR spectra of zeolite-clinoptilolite from different locations

Vibrational assignment	Clinoptilolite (Khekordzula location)	Clinoptilolite (Khandaki location)	Clinoptilolite (Aspindza location)
$\delta(\text{Si-O})$	469	457	465
Intertetrahedral $\delta[\text{O-Si(Al)}]$	523	523	520
	607	604	603
	664	664	668
$\nu(\text{Si-O})$ Symmetrical	747	712	797
$\nu$ Si-O-Si(Al) Symmetrical	1070	1046	1055
$\nu$ Si-O-Si(Al) Asymmetrical	1208	1205	1207
$\delta(\text{H-O-H})$	1644	1654	1636
$\nu(\text{OH}^-)$	3438, 3634	3437, 3630	3421, 3640

**3.4. Test results of lime mortar containing clinoptilolite.** To test zeolite-added mortar, 7 types of building mortar samples were prepared, consisting of ground quicklime (burnt at 1000°C) and sand from the Sachkhere deposit in the proportion of 150÷150 g, to which zeolite

was added in amounts of 30 and 60 g, respectively. The samples were formed into cubes, and after 28 days, their physical and mechanical compression tests were carried out. The results are presented in Table 3.

**Table 3.** Test results of lime mortar containing clinoptilolite

Mortar composition				Zeolite location	%	Amount of water added, %	Strength after 28 days, MPa
Scorched at 1000°C with a 2-hour delay	150g	Yellow sand	150g				
				Without zeolite additive	–	33.3	2.0
				Khandaki location	10	36.7	2.8
				Khandaki location	30	46.8	3.1
				Aspindza location	10	64.0	3.0
				Aspindza location	30	60.0	3.13
				Khekordzula location	10	48.0	3.55
				Khekordzula location	30	58.0	2.62

The test showed that the addition of zeolite nano-additives increases the mortar strength from 20% to 77%. It should be noted that the effectiveness of the additive strongly depends on the content of the zeolite phase. In particular, the zeolite phase content in the clinoptilolite of the Khekordzula locality is more than 90%, whereas in the case of the other two samples, the zeolite phase does not exceed 70%. Thus, in the first case, 10% additive

already gives optimum results, while in the other cases, 20% additive is required to achieve the best results.

The chemical composition of samples containing clinoptilolites from all three locations as an additive is shown in Table 4. The table shows that the content of silicon and aluminum cations (except calcium) in the mortar changed sharply.

**Table 4.** Data of chemical analysis of samples

#	Constituent elements in the form of oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SO <sub>3</sub>
1	Sand + lime (without additives)	12.96	1.34	41.75	0.36	0.50	0.13	0.74	0.03	1.40
2	Sand + lime + 30 g zeolite (Khekordzula locality – 10%)	13.05	1.58	39.19	0.32	0.51	0.13	0.74	0.05	1.32
3	Sand + lime + 60 g zeolite (Khekordzula locality – 30%)	14.65	1.78	36.28	0.42	0.53	0.13	0.79	0.03	1.33
4	Sand + lime + 30 g zeolite (Aspindza locality – 10%)	8.79	1.01	38.78	0.26	0.40	0.13	0.66	0.01	1.17
5	Sand + lime + 60 g zeolite (Aspindza locality – 30%)	8.79	1.16	36.73	0.22	0.42	0.13	0.65	0.005	1.10
6	Sand + lime + 30 g zeolite (Khandaki locality – 10%)	21.15	2.54	30.06	0.33	1.05	0.13	1.08	0.05	1.18
7	Sand + lime + 60 g zeolite (Khandaki locality – 30%)	16.98	2.13	34.46	0.34	0.74	0.13	1.08	0.07	1.22

According to the data in Table 4, the test parameters in the samples are: the humidity in sample #1 was 9%, and the loss of ignition was 23%; the humidity in sample #2 was 12%, and the loss of ignition was 31%; the humidity in sample #3 was 13%, and the loss of ignition was 33%; the humidity in sample #4 was 12%, and the loss of ignition was 35%; the humidity in sample #5 was 35%, and the loss of ignition was 33%; the humidity in sample #6 was 10%, and the loss of ignition was 29%; The humidity in sample #7 was 9%, and the loss of ignition was 32%;

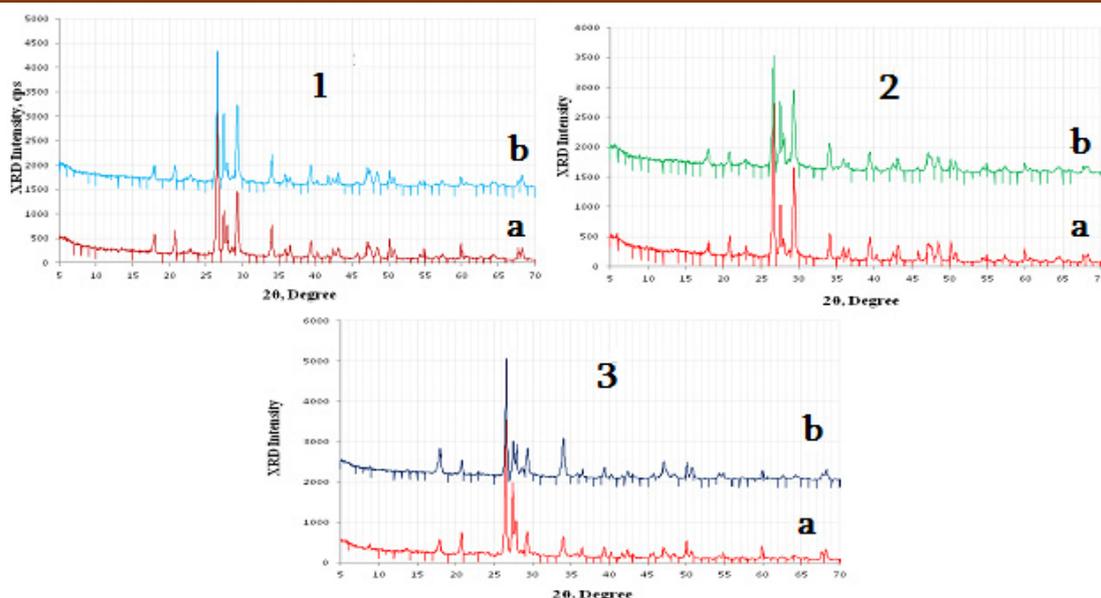
The X-ray diffraction analyses of samples containing clinoptilolites from all three locations as an additive are shown in Fig. 3.

X-ray diffractograms show a quartz (SiO<sub>2</sub>)

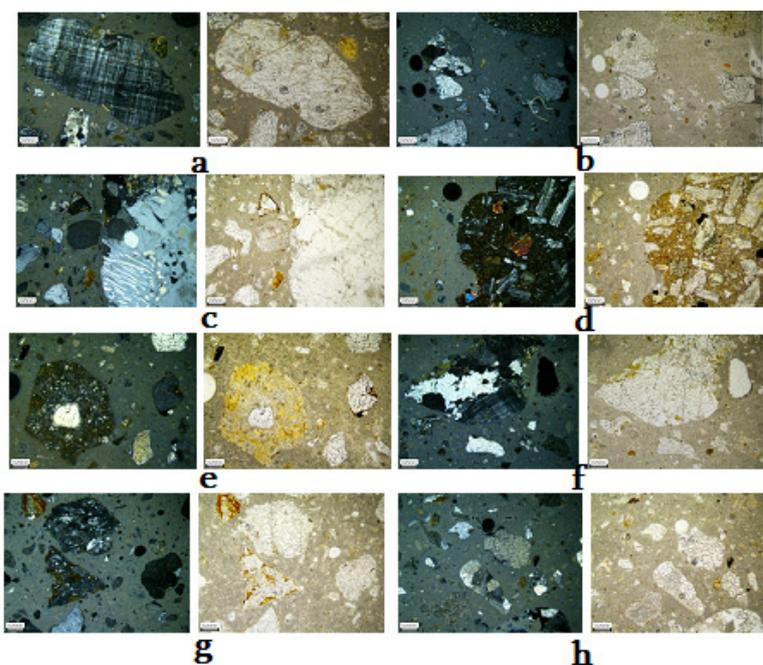
peak at 3.34 Å and a calcite peak at 3.036 Å. Their intensity decreases with time. In 28 days, concrete strength increases compared to 7 days, which indicates the reactivity of the substances contained in the mortar.

It should be noted that delaying the cubes for 90 days showed that their strength more than doubled compared to delaying them for 28 days. This again proves that the mortar hardening process is continuous and prolonged.

Petrographic studies of the mortars showed that their phase composition changes insignificantly. In all three cases, the mortar mainly consists of finely dispersed clay and carbonate masses, as well as fragments of quartz, plagioclase, microcline, and basalt (Fig. 4).



**Fig. 3.** X-ray diffractograms of the studied samples: **1** – clinoptilolite from the Khekordzula location (a – 10%; b – 30%); **2** – clinoptilolite from the Aspidza location (a – 10%; b – 30%); **3** – clinoptilolite from the Khandaki location (a – 10%; b – 30%)



**Fig. 4.** Petrography of samples: **a** – test mortar without zeolite additive, in the proportion of 100g lime + 150g sand; **b** – test mortar without zeolite additive, in the proportion of 150g lime + 150g sand; **c** – test mortar with 10% zeolite additive (clinoptilolite from Khandaki location); **d** – test mortar with 30% zeolite additive (clinoptilolite from Khandaki location); **e** – test mortar with 10% zeolite additive (clinoptilolite from Aspidza location); **f** – test mortar with 30% zeolite additive (clinoptilolite from Aspidza location); **g** – research mortar with 10% zeolite additive (clinoptilolite from Khekordzula location); **h** – research mortar with 30% zeolite additive (clinoptilolite from Khekordzula location)

IR spectroscopy study of samples obtained with clinoptilolite addition showed that for the mortar without zeolite addition, the intensity of Si-O-Si (Al) bond vibrations in the IR spectrum, both in the valence and

deformation regions, is much lower than in the spectrum of the mortar with zeolite addition. This is observed for clinoptilolite from all three deposits (Table 5). The bands of intertetrahedral deformation vibration at 594 and 695  $\text{cm}^{-1}$

characteristic of zeolite-clinoptilolite are also present in the mortar composition [15].

**Table 5.** Characterization of the IR spectra of the studied mortars

Vibrational assignment	Test mortar without zeolite additive		Test mortar with zeolite additive clinoptilolite (Khandaki location)		Test mortar with zeolite additive clinoptilolite (Aspindza location)		Test mortar with zeolite additive clinoptilolite (Khekordzula location)	
	100 g sand + 100 g lime	150 g sand + 150 g lime	10%	30%	10%	30%	10%	30%
$\nu(\text{Me-O})$ ; $\delta(\text{Si-O-Si})$ ; $\nu(\text{Si-O-Me})$	-	396	-	397 372	599	384 393	-	375
$\delta(\text{O-Si-O})$ ; $\delta(\text{Si-O})$	424 458	425 461	426 463	467	463	426 472	464	467
Intertetrahedra 1 $\delta[\text{O-Si(Al)}]$	533 594	518 594	521 595	521 595	535 590	533 563	535 581	530 582
$\nu[\text{Si-O-Al(Si)}]$	-	695	649	649, 695	695	668	646, 694	646, 694
$\nu_3(\text{CO}_3^{2-})$	713	713	713	-	-	715	-	712
$\nu(\text{Si-O-Si})$ – Intratetrahedra 1 $\nu(\text{Si-O-Si})$ – Intertetrahedra 1	778	778 797	787	779 797	779 797	779 798	778 798	788 796
$\nu_2(\text{CO}_3^{2-})$	875	874	874	875	875	859, 875	875	858, 875
$\nu_1(\text{CO}_3^{2-})$ $\nu(\text{Si(H)-O})$ $\nu(\text{Si-O})$	1060 1105 1190	1030 1090 1162	1030 1080 1160	1055 1080 1185	1065 1084 1180	1053 1085 1160	1037 1083	1024 1052 1092
$\nu_3(\text{CO}_3^{2-})$	1458	1464	1457	1470	1478	1458	1458	1457
$\delta(\text{H-O-H})$	1645	1684	1627	1651	1643	1663	1627	1635
$\nu(\text{C-O})$	1790	1793 1890	1792	-	-	-	-	1792
$\nu(\text{OH}^-)$	3495 3642	3453 3643	3490, 3513 3644	3442, 3573 3644	3409 3642	3457, 3470 3645	3469 3644	3435 3628

Of particular interest in the IR spectra is the decrease in the intensity of bands of water hydroxyl groups valence vibration (at 3495, 3513, and 3644  $\text{cm}^{-1}$ ). Apparently, after a certain time, water present in the zeolite structure participates to some extent in the

chemical processes occurring in the mortar composition. This is also evidenced by the decrease in the intensities of the Si–O–Si(Al) intertetrahedral vibration bands (at 633, 595, and 1190  $\text{cm}^{-1}$ ).

### Conclusion

Zeolites play a key role in zeolite-containing tuff rocks, serving as the principal binding component of this material. Acting as natural adsorbents, they help regulate humidity and temperature within buildings. However, zeolite tuff should not be regarded merely as a building material. Its effective application requires a comprehensive understanding of its mineralogical, chemical, and macroscopic properties, since its performance in construction results from the synergistic interaction of its constituent components. Our research has once again confirmed this interdependence.

The introduction of a certain amount of zeolite additive into building mortar had a pronounced effect on its strength. Interestingly, zeolites with similar structures but differing chemical and phase compositions exhibited

varying influences on the mortar's mechanical properties.

The addition of 10% zeolite–clinoptilolite increased the strength of the mortar as follows:

- clinoptilolite from the Khekordzula deposit — by 77%;
- clinoptilolite from the Aspindza deposit — by 55%;
- clinoptilolite from the Khandaki deposit — by 50%.

As noted above, the structure, chemical, and phase composition of zeolites have a significant impact on the hardening process of lime mortars. The presence of more reactive minerals and cations within the zeolite enhances the strength and durability of the resulting material.

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