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Mineral additive for lime dry building mixtures

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Abstract. The article deals with lime dry building mixtures for the restoration of historical buildings. Considering the low strength and water resistance of coatings based on lime compositions, the possibility of using mixed clay fired at low temperatures (450–500 °C) as mineral additives in dry building mixtures was considered. The chemical and mineralogical composition of clays was established. The spectrum of the distribution of adsorption centers according to the acid-base type, as well as the nature of its change in the process of firing clay at different temperatures, was investigated. The regularities of the structure formation of lime finishing compositions with the use of mineral additives was established. It is shown that lime composites based on compositions with a mineral additive fired at a temperature of 450–500 °C are characterized by a high content of zeolites, a decrease in Ca (OH)₂, and an increase in the amount of chemically bound lime by 31.8 %. It is shown that the introduction of clay fired at temperatures of 450–500 °C into the lime-sand composition contributes to a 2–2.6 times increase in compressive strength at the age of 28 days of air-dry hardening, depending on the firing temperature and the type of clay. The technological and operational properties of lime dry building mixtures and coatings based on them are given.

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1. Introduction

Lime compositions are widely used for the restoration of historical buildings [1–7]. Finishing layers based on lime compositions have high vapor permeability and biostability, but low strength and water resistance. One of the ways to prevent premature destruction of lime finish coatings is to introduce modifying additives into the formulation [8–11].

One of the modifying additives used for the production of dry building mixtures (DBM) is metakaolinite. Metakaolinite chemically interacts with lime, which helps to increase the strength of the composition [12–16].

In lime plaster mixes, metakaolinite, being a clay material, provides excellent plasticity to the compositions and the absence of stickiness, which makes it possible to produce high-quality dry mixes. The light color of the additive also allows it to be used in decorative mixtures. The rate of reaction with lime in metakaolinite is higher than in pozzolanic additives, which ensures reliable binding of slow-hardening lime already in the first days of its hardening.

The work found that when mixing lime, water and metakaolinite, fired at a temperature of 730 °C, the strength of the composition is 10–15 MPa. Hydration products are mainly C_2ASH_8 and CSH [17, 18]. In study [19], metakaolin was used to enhance the volume stability, mechanical properties and environmental erosion resistance of traditional plasters. Research results show that metakaolin reacts with Ca(OH)₂ via pozzolanic reactions. Additionally, the microstructure of mortars becomes more compact due to the formation of cross-linked microstructures.

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Despite the numerous positive properties of the metakaolinite additive, one cannot fail to note, firstly, significant energy costs for the thermal processing of clay (up to 800 °C), since today, the issues of energy conservation are still relevant. Secondly, Russia has limited resources of kaolin clays. In connection with the above, the problem arises of developing a mineral additive that is not inferior in properties to metakaolinite, but can be obtained using a less energy-intensive technology based on local clays with a predominance of kaolinite.

The territory of the Volga region, including the Penza region, has significant reserves of mineral raw materials, in particular, clays, which can be used in the production of mineral additives for dry building mixtures.

It is known that the clay surface is a set of Lewis and Bronsted centers of both acidic and basic types [20, 21]. Most of the processes occurring on the surface of clay particles are local in nature and are largely determined by the energy parameters of specific active centers. In this regard, it is of particular importance to study the spectrum of the distribution of adsorption centers according to the acid-base type, as well as the nature of its change during the firing of clay at different temperatures [22].

The clays of the Volga region, including the Penza region, are mainly polymineral in terms of their mineralogical composition. It is proposed to obtain active mineral additives from polymineral clays by firing them at low temperatures. According to [23–25], when clay is fired in the temperature range of 450–650 °C, single-water kaolinite $Al_2O_3.2SiO_2.H_2O$ (hydroxoaluminium salt of metasilicic acid) is formed, which is more reactive with respect to lime than metakaolinite.

The aim of the work was to develop a scientifically grounded technological solution for obtaining a mineral additive based on mixed clays for lime dry building mixtures. To reach this goal, it is necessary to solve the following tasks:

- to evaluate the possibility of obtaining a mineral supplement during low-temperature firing of clays;
- to establish patterns of structure formation of lime mixtures with mineral additives;

- to develop a formulation of a lime dry building mixture and to determine the operational properties of a finishing layer based on it.

2. Methods

In the development of the mineral additive, the clays of the Vorobyevsky, Kameshkirsky, Belinsky, as well as the Issinsky deposits of the Penza region were used. The chemical composition of clays is presented in Table 1.

Chemical	The content of chemical compounds depending on from clay deposit, (%)			
compound	Belinsky	Kameshkirsky	Vorobyevsky	Issinsky
SiO ₂	59.56	75.41	72.76	65.50
A12O3	11.85	11.04	11.57	9.54
Fe ₂ O ₃	4.54	6.61	7.39	7.72
Other	24.05	6.94	7.72	15.46

Table 1. Chemical composition of clays.

The indicator method of adsorption of indicators with different pKa values was used to evaluate the acid–base sites on the surface of clay particles. The quantitative determination of the centers of adsorption was carried out photometrically. Optical density was measured on a KFK-3KM photocolorimeter. The change in optical density as a result of the process of adsorption of the indicator (ΔD) on the sample surface was calculated by the formula

$$\Delta D = (D_0 - D_1) |\pm| (D_0 - D_2). \tag{1}$$

Number of active centers was calculated by the formula

$$q_{pKa} = \frac{N_{ind}V_{ind}}{D_0} \left(\left| \frac{|D_0 - D_1|}{a_1} \pm \frac{|D_0 - D_2|}{a_2} \right| \right), \tag{2}$$

where N_{ind} is concentration of the indicator solution, mg-mol/ml; V_{ind} is the volume of the indicator solution taken for analysis, ml; D_0 is optical density of the indicator before sorption; D_1 is optical density of the indicator after sorption; D_2 is the optical density of the "blank" solution.

The "-" sign corresponds to a unidirectional change D_1 and D_2 relative D_0 , i.e. D_1 and $D_2 < D_0$ or D_1 and $D_2 > D_0$. The "+" sign corresponds to a multidirectional change D_1 and D_2 relative D_0 , i.e. $D_1 > D_0$, $D_2 < D_0$ or $D_1 < D_0$, $D_2 > D_0$.

Three series of solutions were prepared for photometry.

Series 1 (working solution). The required volumes of indicators with a certain pKa value were poured into centrifuge tubes with a capacity of 10 ml, diluted to the mark with water, mixed, and after 30 minutes weighed portions ($a \approx 0.04$ g) of clay were poured out. After the adsorption equilibrium was established, the optical density value D_1 was measured at a wavelength (λ_{max}) corresponding to each indicator. Before measuring, the tubes with the material were centrifuged for 3-5 minutes.

Series 2 ("blank experience"). Weighed portions of the sample $(a_2 \approx a_1)$ were placed in dry centrifuge tubes and filled with 6 ml of water. After 1 hour, the solution was decanted into another tube with preliminary centrifugation. The required volumes of indicators were added to the solution, water was brought to the mark, the suspension was stirred, held for 30 minutes, and the value of optical density (D_2) was measured, taking into account $\pm \Delta D_2$ due to a change in the pH of the medium and dissolution of the sample.

Series 3 (indicator solution). The required volumes of indicators were poured into test tubes with a capacity of 10 ml, diluted to the mark with water, mixed, and after 30 minutes the optical density value (D_0) was measured.

Based on the data obtained, a curve of the distribution of acid-base centers on the surface of diatomite in coordinates was constructed $q_{pKa} = F(pKa)$.

During the tests, the following indicators were used: brilliant green (pKa = + 1.5), fuchsin (pKa = + 2.1), methyl orange (pKa = + 3.46), bramphenol blue (pKa = + 4.1), bromocresol purple (pKa = + 6.4), bromothymol blue (pKa = + 7.3), thymol blue (pKa = + 8.8), Nile blue (pKa = + 10.5) and ethylene glycol (pKa = +14.2). The determination of the cationic absorption capacity was carried out by the adsorption method based on the adsorption of an organic dye. Methylene blue solution was used as an organic dye. The research was carried out in the following sequence.

1. A 0.3 % solution of methylene blue was prepared and kept in a dark container and in a dark place for 3 days.

2. 10 g of clay was dried at a temperature of 105–110 °C and crushed until it completely passed through a sieve with 2500 holes/cm².

3. In three volumetric flasks with a capacity of 100 cm³ were placed 0.3 g of prepared clay.

- 4. Using a pipette, 50 cm³ of a 0.3 % aqueous solution of organic dye were poured into the flasks.
- 5. The contents of the flasks were shaken for 20 min and quickly filtered through a Blue Ribbon filter.

6. Measurement of the concentration of the methylene blue solution after treatment with a sample of clay was carried out on a KFK-3KM spectrophotocolorimeter.

7. The calculation of the cationic absorption capacity of the material was carried out according to the formula:

$$A = abd \, 100/c \cdot 100 \cdot e,\tag{3}$$

where *a* is the difference in the percentage of methylene blue in the solution before and after clay treatment; *b* is the amount of methylene blue taken for analysis, b = 50 ml; *d* is the percentage of the

dye, d = 0.3 %; c is sample of the test substance, taking into account hygroscopic water, c = 0.3 g; e is equivalent weight of methylene blue dye, e = 319.9.

The adsorption of the indicator under the conditions of analysis occurs both on the Bronsted centers and on the Lewis centers in accordance with its pKa value. The quantitative determination as a result gives the total content of the Lewis and Bronsted centers of the corresponding force on the surface of the studied sample.

The studies were carried out in the field of Bronsted acid (pKa from 0 to 7) and basic (pKa from 7 to 13) centers, and Lewis acidic (pKa > 13) centers. Centers of neutral character correspond to pKa = +7. An increase in pKa values corresponds to an increase in the donor properties of metal atoms, and a decrease in acceptor properties.

To study structure formation, the kinetics of binding of calcium oxide CaO was determined. The content of active CaO in lime was determined by titrating a weighed portion of lime with hydrochloric acid. For this, a 1 g sample of lime was placed in a 250 ml conical flask, 150 ml of distilled water was added, covered with a watch glass and heated for 5–7 minutes. After cooling, 2–3 drops of a 1 % alcohol solution of phenolphthalein were added and titrated with 1 n hydrochloric acid until the contents were completely discolored. The amount of active calcium oxide was determined by the formula

$$\% CaO = \frac{V \cdot 2.804 \cdot K}{m},\tag{4}$$

where %CaO is the content of active CaO, %; *V* is the amount of 1 N hydrochloric acid solution consumed for titration, ml; 2.804 is the amount of CaO corresponding to 1 ml of 1 N hydrochloric acid solution, multiplied by 100; *K* is correction to the titer of 1 N hydrochloric acid solution; *m* is weight of lime, g.

The rheological properties of lime-sand compositions were assessed by the indicator of plastic strength, which was determined using a KP-3 conical plastometer.

3. Results and Discussion

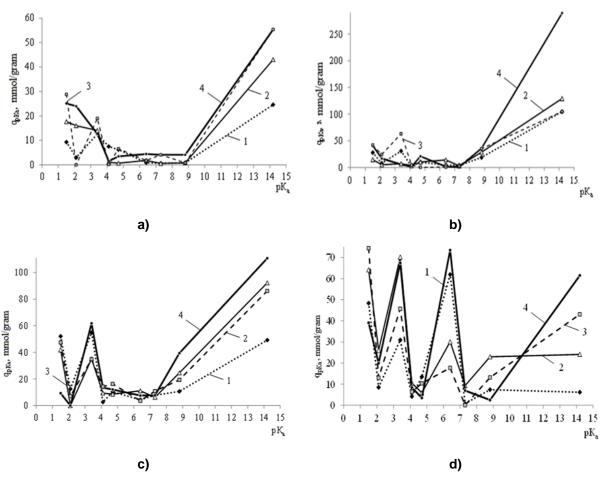
Analysis of clays in terms of cationic absorption capacity allows us to assert that, in terms of mineralogical composition, the clay of the Vorobyevsky deposit belongs to the kaolinite type of clays, the Belinsky deposit – to the hydromica type, Kameshkirsky and Issinsky deposits – to the montmorillonite type (Table 2).

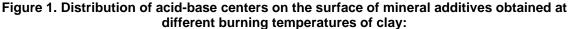
Clay deposit	Cationic absorption capacity value, E , mEq		
Vorobyevsky	11.79		
Belinsky	20.48		
Kameshkirsky	42.50		
Issinsky	39.73		

Table 2. Cationic absorption capacity of the studied clays.

In Fig. 1 shows the distribution curves of adsorption centers on the surface of clays.

The influence of the temperature factor led to a change in the energy state of the particles surface in the studied clays. Comparison of the distribution of adsorption centers on the surface of materials that have practically the same chemical, mineralogical, and granulometric compositions indicates that the number of Bronsted and Lewis acid centers on the surface of thermally treated clays exceeds the number of the same centers on the surface of unburnt clay.





a – from the Vorobievskoye deposit: 1 – without burning; 2 – at 500 °C; 3 – at 600 °C; 4 – at 450 °C; b- from the Kameshkirsky deposit: 1 – without burning; 2 – at 450 °C; 3 – at 600 °C; 4 – at 400 °C; c – from the Belinsky deposit: 1 – without burning; 2 – at 600 °C; 3 – at 450 °C; 4 – at 500 °C; d – from the Issinsky deposit: 1 – without burning; 2 – at 400 °C; 3 – at 600 °C; 4 – at 500 °C.

Dehydration of the surface of layered aluminosilicates leads to the formation of a large number of Lewis centers. The number of active centers in the region pKa > 13 on the surface of Kameshkir clay before firing was 104.57 mmol/g, after firing at a temperature of 400 °C – 413.65 mmol/g. It is the Lewis centers on the surface of the clay that give it a greater reactivity with the binder. Not only the quantity but also the total content of adsorption centers differs.

To study the process of dehydration of clay from the Vorobyovskoye deposit, its differential thermal analysis (DTA) was carried out (Fig. 2) [26–28]. When analyzing the obtained thermogram, it was found that 6.1 % of bound water is present in Vorobievskaya clay, which is gradually removed to a temperature of 1000 °C with the formation of new minerals. When burning clay at a temperature of 400 °C, 2.2 % of water is removed, at 500 °C – 3.4 %, at 550 °C – 3.7 %, at 600 °C – 4.0 %. It has been established that half of the bound water in the amount of 2.9 % in Vorobievskaya clay is removed at a temperature of 450 °C, at which monohydric kaolinite is formed, which gives the mineral additive the greatest activity to lime.

The hardening process of lime compositions consists in carbonization, crystallization of lime, and additionally in the formation of calcium hydrosilicates, hydroaluminates and calcium hydroaluminosilicates. This helps to increase the strength of the finishing compositions. However, metakaolinite Al_2O_3 · 2SiO₂ can react with lime only with the dissolution of the initial substance, while monohydrate caolinite Al_2O_3 · 2SiO₂·H₂O can interact with Ca(OH) ₂ topochemically [29, 30].

To determine the content of the mineral additive in the composition of the DBM, studies were carried out to select the optimal concentration of fired clay, at which the highest value of the ultimate strength in compression of the lime composite will be achieved. Clays of the Vorobyevsky, Kameshkirsky, Belinsky and Issinsky deposits after burning at a temperature of 500 °C were used as a mineral additive, and Ukhtinsky sand was used as a fine aggregate.

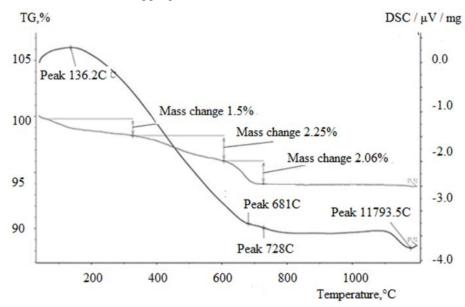


Figure 2. Derivatogram of the Vorobyovskoe deposit clay.

It was found that the highest compressive strength is achieved when a mineral additive is added to the composition of the DBM in an amount of 10 % of the lime mass. So, when Vorobyov's clay was added to the DBM formulation after burning at a temperature of 500 °C in an amount of 10 %, the compressive strength R_{cs} was 1.55 MPa, in an amount of 15 % – 1.38 MPa, in an amount of 20 % – 1.32 MPa. A similar pattern is also observed with the introduction of mineral additives based on Belinskaya, Issinskaya and Kameshkirskaya clays. This is probably due to the fact that with an increase in the amount of the mineral additive over 10 %, an excess of the finely dispersed fraction is obtained, which leads to a lack of mixing water.

Additionally, to assess the structure formation of lime-sand compositions with the addition of a mineral additive, the kinetics of lime binding was studied. Analysis of experimental data indicates that over time, there is a decrease in the amount of free lime. So, at the age of 7 days, the amount of free lime in the lime-sand composition is 84.5 %, and at the age of 28 days it is 78.6 %. A lower content of the amount of free lime in the compositions with the use of clay after firing at a temperature of 500 °C was established. So, in the samples with the addition of Belinskaya and Kameshkirskaya clays, fired at this temperature, the content of free lime on the 28th day of hardening decreased to 68.21 % and 70.83 %, respectively.

The introduction of fired clay into the lime-sand composition increases the compressive strength at the age of 28 days of air-dry hardening, depending on the firing temperature and the type of clay, by 2.0–2.6 times. With the introduction of Kameshkirsky clay, the highest value of the compressive strength at the age of 28 days of hardening is achieved with the introduction of an additive fired at a temperature of 400 °C into the composition, and is $R_{cs} = 1.75$ MPa, while for the composition without the additive it is 0.84 MPa. With the introduction of Vorobyovskoy clay, fired at a temperature of 450 °C, the strength increases to 1.67 MPa. The introduction of Belinsky and Issinsky clays after firing at a temperature of 500 °C helps to increase the strength of the compositions to 2.2 and 1.68 MPa, respectively. The results of the conducted studies confirm that the addition of Iow-fired clay to the lime-sand composition contributes to an increase in the strength of lime composites by 6–26 % compared to metakaolinite due to the monohydrate kaolinite formed at a temperature of 400–500 °C, which decomposes in an aqueous alkaline medium to ions Al(OH)²⁺ and SiO₃²⁻. The infinite nanoradical [SiO₃] ∞^{2-} has increased activity in an aqueous medium towards calcium oxide and hydrate, while infinite layers of the radical [Si₂O₅] ∞^{2-} kaolinite and the final radical [Si₂O₇] ⁶⁻ in metakaolinite have little activity towards lime.

In Fig. 3 shows the curves of the kinetics of the strength gain of lime compositions based on the results of the approximation of experimental data for determining the compressive strength of the test samples. The data were approximated using the CurveExpert 1.3 program. The dots in the figures indicate the experimental values of the ultimate compressive strength of lime samples. Based on the results of approximation and experimental studies (Fig. 3), it follows that clays after firing at temperatures of 400–500 °C (curve 2) contribute to a more intense structure formation of lime-sand samples than clays fired at a temperature of 600 °C (curve 1).

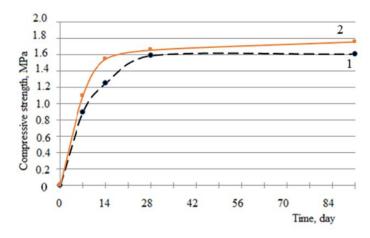


Figure 3. Kinetics of strength gain of lime-sand compositions with the addition of clay from the Vorobievskoye deposit: $1 - after firing at t = 600 \degree$ C; $2 - after firing at t = 450 \degree$ C.

The curves shown in Fig. 3 are described by the exponential equation

$$y = a\left(1 - e^{-bx}\right),\tag{5}$$

where a is a constant that takes into account the maximum possible compressive strength; b is rate constant of structure formation; x is the hardening time.

The values of the rate constant of structure formation b are given in Table 3.

Clay deposit	Clay firing temperature, °C	а	b
Vorobievskoe	450	1.744	0.146
	600	1.611	0.123

The calculation results show that at low-temperature firing of clay, the value of the rate constant of structure formation is higher in comparison with firing clay at a temperature of 600 °C. Thus, the rate constant of structure formation b for lime compositions with the addition of Vorobiev's clay after firing at 600 °C was 0.123 MPa/day, while for compositions with clay fired at 450 °C it was 0.146 MPa/day (Table 3). A similar law is observed with the introduction of mineral additives based on Belinskaya, Kameshkirskaya and Issinskaya clays.

It was found that mineral additives increase the plastic strength in the early stages of hardening (Fig. 4). The fastest increase in plastic strength was observed for the composition with the addition of clay after firing at 500 °C (Fig. 4, curve 4), the plastic strength after 67 hours from the moment of mixing was 0.064 MPa, while for the composition with the addition of clay fired at temperature of 600 °C, - 0.054 MPa.

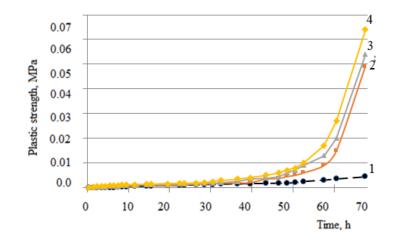


Figure 4. The effect of the clay firing temperature on the kinetics of changes in the plastic strength of the mixture: 1 – control composition (without additive); 2 – clay content 10 % by weight of lime (without firing); 3 – clay content 10 % by weight of lime (clay fired at a temperature of 600 °C); 4 – clay content 10 % by weight of lime (clay fired at a temperature of 500 °C).

The water-holding capacity of composites is 96.5–98 %. The results obtained agree with the data of the authors [31, 32]. The authors proposed, when developing a composition for restoration, to use a mixture of calcium hydroxide, pozzolan, volcanic ash and quartz sand as a filler. Using X-ray analysis and high-resolution SEM images of newly applied plaster, the transformation of portlandite to calcite and the pozzolanic effect of kaolinite are established.

We recommend using the proposed dry mortar for the restoration of historical buildings. The mortar mixture is well applied to the surface and is combined with previously finished surfaces.

Table 4 shows the compared indicators of technological and operational properties of finishing compositions based on developed dry mixes and a prototype composition. The technological and operational properties of the developed dry mixes were compared with the properties of the finishing classical Runit plaster based on air lime, produced by the company OOO "Azhioproekt" [33]. Plaster "Runit" is used for the restoration of buildings.

	Value of indicator of Finishing compositions	
Name of indicator	developed	prototype
Average density DBM, ρ _a , kg/m ³	1290–1304	1575
Viability at stored in open capacities, hour	8–10	≥ 2
Workability	good	good
Recommended thickness of one layer, mm	before 20	Before 20
Consumption of finishing composition at 1 layer is applied of thickness 10 mm, kg/m ²	12–14	15–17
Water-holding capacity,%	98.0–98.3	95.0–97.0
Time of drying at 20 °C to degree "5", min	no more 55	no more 45
Adhesive durability R_{ad} , MPa	0.60-0.76	0.40
Durability at compression, R_c , MPa	2.52-3.71	0.40
Shrinkable deformations, E, %	0.027-0.034	0.030-0.040
Vapor cofficient μ , mg/m·h·Pa	0.055–0.058	0.047
Cracks due to shrinkage	no	no
Temperature of application, °C	5–35	5–35

Table 4. Technoloc	gical and operational	properties of finishin	q compositions.
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It was found that the developed dry mix is not inferior to the prototype in terms of technological and operational properties. At the same time, the production of the proposed dry mixes is more economical due to the use of local raw materials and domestic additives.

4. Conclusions

1. The authors developed a technology and formulation of a mineral additive for lime dry building mixtures involving low-temperature firing of clays at a temperature of 400–500 °C. It was revealed that the effect of the temperature factor leads to a change in the energy state of the surface of clay particles, which consists in an increase in the number of Lewis centers (up to 4 times), as well as the total number of active centers (up to 2 times).

2. The study established regularities of the structure formation of lime finishing compositions with the addition of mineral additives. It was revealed by XRD and mechanical tests that samples based on compositions with a mineral additive fired at a temperature of 500 °C are characterized by a high content of zeolites, a decrease in portlandite, and an increase in the amount of chemically bound lime by 31.8 %. The optimal concentration of a mineral additive was selected, which is 10 % of lime mass. It is shown that the introduction of clay fired at temperatures of 450–500 °C into a lime-sand composition increases compressive strength at the age of 28 days of air-dry hardening, which, depending on the firing temperature and the type of clay, is 2–2.6 times.

3. The main technological and operational properties of the lime composition and coatings based on it were determined. The finishing layer based on the developed mixture is characterized by the following indicators: adhesion strength $R_{adg} = 0.60-0.76$ MPa, cohesive strength $R_{kog} = 0.35-0.36$ MPa, vapor permeability $\mu = 0.055-0.058$ mg/mhPa, conditional coefficient crack resistance $K_{tr} = 0.54-0.58$, water absorption by weight W_t = 12.29–13.62 %, softening coefficient K_p = 0.48–0.52, elastic modulus E_{el} = 40–50 MPa, deformation modulus E_{def} = 20.59–24.00 MPa, shrinkage deformations ε = 0.027–0.034 %.

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