Adhesive dry mix using an amorphous aluminosilicates

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Abstract. In the paper, it is proposed to use synthesized amorphous aluminosilicates as a modifying supplement in cement adhesive dry mixes. The results of the analysis of the chemical composition and microstructure of the developed supplement based on amorphous aluminosilicates used as a modifying supplement in the recipe of adhesive dry mixes are given. A method for evaluating the shear strength of a solution layer using the device GT 2.2.3 is proposed. The methods of testing and instrumentation for evaluating the bond strength of the solution layer are described. In the paper, the bond strength of the developed composition of tile adhesive based on cement with substrates made of gypsum, polystyrene foam (with reinforcing mesh) and foam concrete was evaluated. The results of the evaluation of the shear strength of a solution adhesive layer based on the developed recipe and the prototype relative to the substrate obtained using the developed method and the device GT 2.2.3 are presented. The obtained results were compared with the values of DIN EN 12004. It is established that the adhesive joint based on the developed recipe of the adhesive dry mix is resistant to peeling.

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Клеевая сухая строительная смесь с применением аморфных алюмосиликатов

for increasing the physical and mechanical properties of cement compositions is the activation of the binder [7].

In this paper, an adhesive solution made using a supplement, which based on amorphous aluminosilicates is considered [8]. Previous studies have confirmed the feasibility of using amorphous aluminosilicates in cement systems [9–11].

The injection of amorphous aluminosilicates into the dry mixes recipe makes it possible to increase the strength of the solution, the adhesive strength and the water holding capacity [12]. When designing the recipes of adhesive dry mixes, special attention must be paid to the value of the adhesive strength [13–17]. In accordance with the current DIN EN standards, the existing method for determining the bond strength involves the separation of the solution layer from the substrate. At present, there are several methods for determining the bond strength: a pull off test, cross-cut method, a peeling method, a method of parallel incisions, etc. However, during operation, the adhesive layer undergoes not only adhesive but also shear loads [18]. Therefore, when choosing a tile adhesive, it is necessary to take into account the value of its shear load depending on deformation factors (warm floors, new buildings, vibrations, impact loads, temperature deformations, etc.) that affect the coating. The literature review shows that today the method of determining the shear strength of solution layers based on dry mixes is essentially absent.

The goal of the paper is to determine the bond shear strength of the coating based on the developed composition with the base. To achieve the goal, it is necessary to solve the following tasks:

- to evaluate the bond strength of coatings based on the developed mix with the base (substrate);
- to determine the shear strength of the solution layer based on the adhesive dry mix with the base;
- to develop a recipe for a lime dry mix, coatings based on it have increased operational resistance;
- to establish technological and operational properties of cement dry mixes and coatings based on it.

2. Methods

The supplement based on amorphous aluminosilicates was obtained by thorough mixing of soluble glass and aluminum sulfate $\text{Al}_2(\text{SO}_4)_3$. The supplement is a white powder with a specific surface area determined by the BET method, that equals to $S_{\text{spec}} = (68.6\pm3.5)$ m$^2$/g. After drying at a temperature $(105\pm5)$ °C, the true density of the supplement is 2140 kg/m$^3$, the apparent density is 568.2 kg/m$^3$.

The structure of the supplement is mainly represented by particles of rounded shape measuring 5.208 μm to 5.704 μm, but there are particles of a flaky shape with a size of 7.13 μm to 8.56 μm (Figure 1). The chemical composition of the supplement is represented by chemical elements such as – O, Si, Na, S and Al - with contents of 48.71 %, 19.59 %, 16.42 %, 9.67 % and 4.7 %, respectively.

Figure 1. Microstructure of supplement

The developed adhesive dry mix includes the Portland cement M400 produced by the Volsky Cement Plant, the sand of the Ukhtinka field in the ratio of fractions 0.63...0.315:0.315...0.14, respectively 80:20 % and apparent density 1538.2 kg/m$^3$, the plasticizer “Kratasol”, redispersible powder “Neolith P 4400” and supplement based on amorphous aluminosilicates [19].

To evaluate the bond strength with the base, the pull-stub pull off method (normal pull off) was used according to the European standard DIN EN 1348 [20]. The method is based on the determination of the peeling strength of the pull-stub from the surface by pull off. Samples were produced, which are representing a substrate onto which the studying composition of tile adhesive is applied of 5 mm thick and ceramic tile of size (10×10×5) mm. After 27 days of storage of samples under normal conditions, using epoxy pull-stubs of cylindrical shape and 20 mm in diameter were adhered to the tiles. After another 24 hours' storage of the samples under normal conditions, the adhesion strength of the test solution with the base was determined.

For this purpose, the load was applied to the pull-stub with a constant increase rate (250 ± 50) N/s. The bond strength of the finishing composition with the substrate was determined by the formula:

\[
R_{adg} = \frac{P}{F},
\]

where \( P \) – common tension force, N; \( F \) – gluing surface area, mm².

The ultimate compression strength of samples was determined in accordance with National standard of Russia 5802-86 "Mortars. Test methods". As equipment for testing the compression strength of samples, a test machine of the type "IR 5057-50" was used. Depending on the type of used power sensor of "IR 5057-50", the force measuring range was from 50 to 50000 N with an accuracy of 1 N (0.1 kgs). Built-in cross-bar speed controllers allow to set the speed of application of the load from 1 to 100 mm / min (in terms of displacement). The compression strength (MPa) of the samples was determined by the formula:

\[
R_{com} = \frac{P}{F},
\]

where \( P \) – destructive force, N; \( F \) – cross-sectional area of the sample before the test, m².

To evaluate the shear strength of the adhesive dry mixes the method for determining the shear resistance at shearing test of soils was used, which provides for the use of the device GT 2.2.3 produced by GEOTEK Ltd Company (Figure 2) [21].

**Figure 2. Appearance of device GT 2.2.3**

The principle of operation of the device GT 2.2.3 is to create in the apparatus a horizontal shearing load on the sample based on the test composition. The force created by the reducer is transferred to the movable carriage of the shear box and is measured by a force sensor.

The test sample is placed in the carriage of the device in such a way that the substrate is in a small shear ring, and the test composition is in a large shear ring. Manufacturing clearance of 1 mm forms the shear area. During the test, as the tangential load applied to the lower shear ring increases, the value of the shear strain of the test composition relative to the substrate is fixed.

This method and the device GT 2.2.3 were used to determine the shear strength of the solution layer relative to the substrate.

During the tests substrates were used which made of foam concrete of cylindrical shape and geometric sizes of 71.4 × 15 mm. The tile adhesive test composition of a 5 mm thick was applied on the substrate [22].

The samples were tested according to the scheme shown in Figure 3.

During the tests, the optimal shear rate of the solution layer based on tile cement adhesive was used, that equals to 0.2 mm/min. Foam concrete is chosen as a substrate. For shear tests of a solution layer based on dry mixes on calcareous and gypsum binders, the shear rate should be adjusted.

3. Results and Discussion

The kinetics of hardening of a cement-sand solution in the presence of a supplement based on amorphous aluminosilicates and a redispersible powder Neolith P 4400 was evaluated. Samples were produced using the sand of the Ukhtinka field with a cement-sand ratio of C:S, which is C:S = 1:2 and water-cement ratio W/C, which equals W/C = 0.47. The samples were hardened at air-dry conditions (temperature (18±2) °C, relative air humidity 60%-70%). The results of the studies are shown in Figure 4.

Analysis of the experimental data shown in Figure 4 revealed that the injection of redispersible powder Neolith P 4400 and a synthetic supplement into the solution recipe increases its compression strength. It has been established that the injection of a supplement based on amorphous aluminosilicates (20 % by mass of cement) to the recipe of a cement-sand solution increases the compressive strength of samples at 90 days of air-dry hardening by 37.3 % (Figure 4, curve 2) as compared with control samples (without supplement). The injection of a synthetic supplement (20 % by mass of cement) and Neolith P 4400 powder (0.5 % by mass of cement) into the recipe of a cement-sand solution increases the compressive strength of samples at the age of 90 days by 39.9 % (Figure 4, curve 3) compared to the control sample (no supplement). The use of a synthetic supplement (20 % by cement mass) and Neolith P 4400 powder (1 % by cement mass) in the recipe of cement-sand solution increases the compressive strength of samples at the age of 90 days by 49.4 % (Figure 4, curve 4) by compared with the control sample (no supplement).

Figures 5 and 6 show curves of the kinetics of the cement stone strength gain by the approximation of the experimental data of determination of the strength of the test samples. Approximation of the experimental data was carried out with the help of software Curve Expert 1.3. The points in the figures indicate the experimental values of the ultimate compressive strength of cement composites.

Based on the results of approximation and experimental studies, it follows that the use of a supplement based on synthetic zeolite in a cement-sand solution recipe promotes a more intensive structure formation of the cement composite than the use of methylcellulose FMC 2094 in the recipe.

The curves shown in Figures 5 and 6 were described by the exponential equation:

$$y = a (1 - e^{-bx}),$$

where $a$ – constant, taking into account the highest possible compressive strength; $b$ – structure formation rate constant; $x$ – time of hardening.

Table 1 shows the values of the constants $a$ and $b$ for the established kinetics equation for the strength gain of a cement-sand composite in the presence of modifying supplements.

<table>
<thead>
<tr>
<th>Modifying supplement</th>
<th>Quantity, %</th>
<th>$a$</th>
<th>$b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Supplement based on synthetic zeolite</td>
<td>20</td>
<td>34.674</td>
<td>0.221</td>
</tr>
<tr>
<td>Methylcellulose FMC 2094</td>
<td>1</td>
<td>24.434</td>
<td>0.117</td>
</tr>
</tbody>
</table>

Thus, the studies made it possible to establish that a domestic supplement based on synthetic zeolites can replace an expensive import supplement methylcellulose FMC 2094 in the recipe of adhesive dry mix.

During the tests the cohesion of the shear of the ceramic tile was established, which is the $R_{\text{kog}} = 1.4$ MPa (Figure 7). The tile was not detached from the base.

Table 1. The values of the constants of the kinetics of a gain of strength

![Figure 5. The kinetics of the gain of strength of cement-sand composition with the use of methylcellulose FMC 2094 in the recipe (1 % by the cement mass)](image5)

![Figure 6. Kinetics of a gain of strength of cement-sand composition with the use of the synthetic supplement in 20 % by the binder mass](image6)

![Figure 7. The nature of the detachment of the washers during the test](image7)
Thus, it has been found that samples of cement-based adhesive have a bond strength higher than 1.4 MPa. The adhesion strength of the adhesive layer to the substrates made of gypsum, expanded polystyrene (using reinforcing mesh) and foam concrete was evaluated. The test results are shown in Table 2.

**Table 2. The adhesion strength of the adhesive layer**

<table>
<thead>
<tr>
<th>Type of the foundation</th>
<th>Strength of adhesion, $R_{adg}$, MPa</th>
<th>The character of separation</th>
<th>Composition based on the developed mix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gypsum</td>
<td>by the tile</td>
<td>more than 1.1</td>
<td></td>
</tr>
<tr>
<td>Expanded polystyrene, extruded</td>
<td>by the tile</td>
<td>more than 1.3</td>
<td></td>
</tr>
<tr>
<td>Styrofoam PSB S-35</td>
<td>by the tile</td>
<td>more than 1.2</td>
<td></td>
</tr>
<tr>
<td>Foam Concrete</td>
<td>by the tile</td>
<td>more than 1.3</td>
<td></td>
</tr>
<tr>
<td>Cement-sand</td>
<td>by the tile</td>
<td>more than 1.4</td>
<td></td>
</tr>
</tbody>
</table>

As can be seen from the results represented in Table 2, the tile adhesive, made on the basis of the developed composition with the use of supplement based on amorphous aluminosilicates, has sufficient adhesion strength with different types of surface, more than $R_{adg} > 1.1$ MPa. Comparing the obtained results with the requirements for the adhesion strength of the tile adhesive to the base and tiles specified in the DIN EN 12004 series, where $R_{adg} \geq 0.5$ MPa, we conclude that the adhesion strength of the tile adhesive made on the basis of the developed adhesive dry mix composition satisfies the conditions of the standard [23].

Since the tile adhesive in the process of utilization is subjected to shear loads relatively to the substrate, then a shear strength was determined. The results of testing of cement-based tile adhesive on the basis of the developed composition relatively a foam concrete substrate are shown in Figure 8.

**Figure 8. The shearing test of a cement based adhesive: a – is the value of the strain; b – shear stress**

**Figure 9. The shearing test of a cement based adhesive: a – the developed composition; b – a prototype**

The analysis of the data presented in Figure 8 and Figure 9 showed that the adhesion strength at the shear of the tile adhesive based on the developed composition of the adhesive dry mix using a supplement based on amorphous aluminosilicates is $R_{shf} = 0.88$ MPa, and based on the prototype is 0.55 MPa, which meets the requirements imposed on the adhesion strength of the adhesive layer [24].

Table 3 presents the technological and operational properties of the finishing composition and coatings based on it, which were based on the developed dry mix and the prototype composition.

**Table 3. Technological and operational properties of the finishing composition**

<table>
<thead>
<tr>
<th>Parameter name</th>
<th>Parameter value for developed composition</th>
<th>Prototype</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion strength, MPa</td>
<td>more 1.4</td>
<td>1.1</td>
</tr>
<tr>
<td>Mixing time, min</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>Adhesion strength at the shear, MPa</td>
<td>0.92</td>
<td>0.6</td>
</tr>
<tr>
<td>Frost resistance, cycles</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>Water-holding capacity, %</td>
<td>97.9</td>
<td>95</td>
</tr>
<tr>
<td>Recommended layer thickness, mm</td>
<td>3-5</td>
<td>before 10</td>
</tr>
<tr>
<td>Sliding tiles, mm</td>
<td>0.3</td>
<td>0.5</td>
</tr>
<tr>
<td>Shrinkage deformations, %</td>
<td>0.028</td>
<td>0.030</td>
</tr>
<tr>
<td>Water vapour permeation μ, mg/m²×t×Pa</td>
<td>1.43</td>
<td>2</td>
</tr>
<tr>
<td>Warranty period of storage, month</td>
<td>12</td>
<td>12</td>
</tr>
</tbody>
</table>

The technological and operational properties of the developed dry mix were compared with the properties of the cement mix "UNIS".

Thus, coatings based on the developed dry mix have higher operational properties such as compressive strength, adhesion strength, frost resistance, etc.

4. **Conclusions**

1. The bond strength of coatings based on the developed mix with the base (substrate) was evaluated. It has been established that samples of cement-based tile adhesive are characterized by high adhesion strength, which is more than $R_{\text{kg}} = 1.4$ MPa and $R_{\text{adg}} > 1.1$ MPa. The greatest contribution to the increase in the adhesion strength is made by the addition Neolith P 4400, which is dispersed at adding water and, as the moisture evaporates, forms high-strength films on the solid particles of the solution, increasing its adhesion to the cement-sand substrate.

2. The shear strength of the solution layer based on the adhesive dry mix with the base was determined. The carried out researches showed the possibility of using the proposed method and instrumentation to determine the adhesion strength to shear of the solution layer based on cement-based adhesive dry mix using a supplement based on amorphous aluminosilicates. The tests carried out proved that the adhesive joint based on the developed dry mix composition is resistant to exfoliation, since $R_{\text{shift}} < R_{\text{adg}} < R_{\text{kg}}$.

3. The recipe for a lime dry mix was developed; coatings based on it have increased operational resistance. The developed adhesive dry mix includes the Portland cement M400, the sand in the ratio of fractions 0.63...0.315:0.315...0.14, respectively 80:20%, the plasticizer "Kratasol", dispersible powder "Neolith P 4400" and supplement based on amorphous aluminosilicates.

4. Technological and operational properties of cement dry mixes and coatings based on them was established. Adhesive layer based on the developed dry mix is characterized by a mark on frost resistance F50, frost resistance of the contact zone Fcz50, water absorption due to capillary attack 1.43 kg/m²×h⁰.⁵.

**References**


**Литература**


