Increasing the water resistance of the composite gypsum binder for arbolito concrete due to hydrofobization

O S Shynkevich, D S Linnik, I V Zaginaylo and G G Bondarenko

1 Department of processes and machine in the technology of building materials, Building and Technology Institute, Odessa State Academy of Civil Engineering and Architecture, Odessa, Ukraine
2 Department of metal, wood and plastic structures, Institute of Civil Engineering, Odessa State Academy of Civil Engineering and Architecture, Odessa, Ukraine
3 Department of Physics, Institute of Civil Engineering, Odessa State Academy of Civil Engineering and Architecture, Odessa, Ukraine
4 Nikolaev Construction College KNUSA, Nikolaev, Ukraine
5 fgg109m@gmail.com

Abstract. The article discusses the problem of increasing the water resistance and strength of a composite gypsum binder for concrete. The effect of complex modification of a composite gypsum binder with a micro-reinforcing, polymineral micropuzzolanic additive filler and superplasticizer from Sika was investigated. The use of filler additives in optimal ratios made it possible to obtain a composite gypsum binder with a softening coefficient of 0.9.

1. Introduction

Energy efficiency, resource saving, lower operating costs and increased housing comfort is a priority in construction practice. Development trends of the modern construction industry are focused on increasing competitiveness, developing and introducing new technological solutions that provide resource and energy saving, high technical, economic and consumer indicators of product quality. The optimization of product quality indicators is ensured through the use of durable, sound, high-quality building materials. An important requirement is the environmental friendliness of building products, which can be achieved through the use of appropriate materials and compliance with the closed-cycle technological mode [1].

The materials of this type relates gypsum concrete - concrete based composite gypsum binder [2-4]. Gypsum materials and products are advanced building materials due to the simplicity and low energy consumption of production. For the production of 1 ton of gypsum binder, 4.5 and 4.9 times less fuel and electricity are used than for the production of 1 ton of Portland cement. The hardening time of a gypsum binder is 20–25 times less than that of Portland cement; a set of design strength for products based on gypsum binder is 30–40 times faster than that based on Portland cement. In addition, materials containing gypsum are high-tech, because already 15-20 minutes after the product is formed, it can be removal of formwork, because of this the mould reusability increases in 8-10 times, and manufacturing techniques are less energy-consuming in contrast to the use of other types binders, including Portland cement.

Ukraine has sufficient reserves of natural gypsum raw materials and a huge amount of gypsum-containing waste. One of the disadvantages of gypsum binders is the low water resistance. One of the
modern effective ways to increase water resistance can be a modification of the structure of the binder with polyminerul microdispersed filler additives [5-7].

The use of new types of volumetric hydrophobizers will provide a further improvement in quality. The compositions of hydrophobizers are available in the form of concentrates or ready-to-use solutions in aqueous or organic solvents. They create a water-repellent effect, while the vapor permeability of such composites is practically not reduced. The introduction of water repellents does not change the color of the product, hydrophobization significantly increases the frost resistance of any material. These properties allow the use of water repellents for composite gypsum binder.

**Objective:** to development of a complex of highly dispersed polyminerul additives, a microreinforcing filler additive and a superplasticizer for a composite gypsum binder with improved properties.

**Research Objectives:**
- selection and justification of the components of a complex polyminerul micropuzzolanic additive and micro-reinforcing filler additives for composite gypsum binder;
- selection of the ratio of the finely divided components of the complex polyminerul micropuzzolanic additive and the micro-reinforcing filler additive for the composite gypsum binder using experimental statistical modeling methods based on the mathematical theory of experimental design;
- evaluation of the interdependent on water resistance and compressive strength of the composite gypsum binder complex of a polyfunctional modifier, and a volume hydrophobization.

2. Materials and research methods

The following components were used as binder components: building gypsum G5 produced by PJSC Gypsum, cement pozzolanic non-additive produced by OJSC Euro Cement Ukraine PC-I-500D0 according to DSTU B B.2.7-46: 2010.

To obtain a composite gypsum binder, highly active metakaolin TU U 14.2-36363275-001: 2009 manufactured by LLC Meta-D, silica fume produced by Elkem AS EN 13263-1 as active pozzolanic additives were used.

Wollastonite TU 5777-006-40705684-2003 of different dispersion produced by Geocom CJSC was used as a micro-reinforcing additive.

Sika ViscoCrete 225 superplasticizer proshka VP SIA 162 (1989) and prEN 934-2, manufactured by Sika trademark, was used as an additive of retarder.

Composite gypsum binders' samples were tested for strength at the age of 28 days according to DSTU B B.2.7-187: 2009, followed by drying to constant weight. The determination of the softening coefficient was carried out according to TU 21-0284757-90.

3. Planning a Six-Factor Experiment

The choice of the experimental plan for solving specific materials science problems is dictated by the specific conditions of these problems and the requirements for the results of their solution. For the purpose of work, a 24-point plan in the form of "triangles on a cube" was applied. The use of MTQ plans is subject to the following provisions. Used to obtain modified composite gypsum binders, additives modifiers and fillers are mixtures with q various substances of different microdispersion.

The composition of such mixtures can be specified by the concentrations of the components in the form of mass, volume or molar fractions (percent) \( \nu_i \). Systems whose properties are determined by the group of dependent mixed factors \( \mathbf{v} = (\nu_1 \ ... \ \nu_q) \) and the group of independent technological factors \( \mathbf{x} = (x_1 \ ... \ x_k) \), are called systems "composition, mixture - properties" or "composition - technology - properties" and are designated by the abbreviation MTQ. To describe MTQ systems, an experimental statistical (ES) model is used, which has a general view:
The influence on the properties of the composite gypsum binder is described and analyzed. The content of additive - superplasticizer by Sika \((x_6)\) was fixed at 0.1%; 0.6%; 1.1% levels.

4. Analysis of experimental results on experimental statistical models

At this stage, the change in water resistance and compressive strength of the composite gypsum binder is described and analyzed.

ES models of the influence on the properties of composite gypsum binder additives of various types and purposes, taking into account the interactions between them, are calculated in the PC COMPEX program \([14, 15]\). The program implements a sequential regression analysis with an experimental error \(S\{Y\}\) of not more than 0.04 at risk degree \(\alpha = 0.02\).

**Compressive strength.** The full ES model (2), which describes the effect of the studied factors on compressive strength, has the following form:

\[
Y(f_{cm}) = A_1v_1 + A_2v_2 + A_3v_3 + D_1v_1x_2 + D_2v_1x_3 + D_3v_1x_6 + b_4x_1^2 + b_5x_1x_5 + b_6x_6^2 + b_7x_6x_5 + b_8x_3^2 + b_9x_3x_5 + b_{10}x_5x_6
\]

(a) \hspace{1cm} (b) \hspace{1cm} (c) \hspace{1cm} (1)

The graphical interpretation of the model (2) is presented in Figure 1 in the form of mixed triangles in coordinates \((v_1, v_2, v_3)\) on a quadratic diagram in coordinates \((x_4, x_5)\). The content of additive - superplasticizer by Sika \((x_6)\) was fixed at 0.1%; 0.6%; 1.1% levels.
Figure 1. The effect of polynomineral micropuzzolanic additives, micro-reinforcing filler additives (wollastonite) on the compressive strength of a composite gypsum binder.

The effect of the three fractions of wollastonite is illustrated on graphical images of ES models in the form of triangular diagrams. The needle-shaped form of wollastonite grain determines the main direction of its use as a micro-reinforcing filler. The physico-chemical affinity of wollastonite with composite building materials containing cement promotes active selective adsorption of binder hydration products, has a significant effect on rheological parameters, structure formation, strength and deformation properties of hardened composites.

Triangular diagrams show the effect on the properties of three dependent factors ($v_1$, $v_2$, $v_3$). These are dependent factors that are interconnected. The total content of all three components (Woll 1, Woll 2, Woll 3) remains constant, i.e. ($v_1 + v_2 + v_3 = 1$). Triangular diagrams located on the square of the analyzed property, in this case $f_{cm}$, allow you to choose the best fractional composition of wollastonite, the content, the ratio of two or three fractions to each other.

The effect on the properties of micropuzzolanic filler additives is graphically interpreted as isolines on the square of two factors $x_4 \rightarrow$ (highly active metakaolin), $x_5 \rightarrow$ (microsilica). The content of highly active metakaolin as well as microsilica varied experimentally in the range of (10 ± 5) %.

Micropuzzolanic filler additives are highly pozzolanic. Microsilica has pozzolanic activity of 350 – 450 mg of bound lime per 1 g of microsilica. Highly active metakaolin, due to the content of active alumina, is able to bind a significantly larger amount of lime than silica fume. Its activity reaches more than 1000 mg of bound lime per 1 g of highly active metakaolin. The introduction of these additives into the composition of the composite gypsum binder should provide early and prolonged strength.

The high-quality superplasticizer on polycarboxylate base by Sika Visco Creit, which consists of specially synthesized chemically pure substances, was introduced to improve workability, reduce water demand and slow down the setting time of the composite gypsum binder.

As follows from the diagram in Figure 1, the maximum value of compressive strength is equal to $f_{cm} = 14.5\pm15.0$ MPa. The obtained strength value is two to three times higher than the applied gypsum binder grade G-5. Portland cement PC-500-D0, non-additive in the amount of 21% from $\Sigma(G+PC)$, was used as the second component of the composite gypsum binder.

The obtained maximum values of $f_{cm}^{\text{max}}$ are achieved when the content of Sika plasticizer is in the amount of 0.1 – 0.6% (from b.m.), the minimum content of highly active metakaolin = 5% and the content of microsilica in the amount of 5 – 10% (from b.m.). Woll 3 wollastonite has a positive effect on $f_{cm}$. The appending of wollastonite in the mixture increases $f_{cm}$ from 13 – 13.5 to 14.5 – 15.0 MPa, i.e. by 10 – 12.5%. Moreover, an increase in the content of Sika additive more than 1% reduces $f_{cm}^{\text{max}}$ by ~ 12% to 12 – 13 MPa.
**Water resistance.** At the next stage, an analysis of the change in water resistance by the value of the softening coefficient $K_p$ under the influence of micropuzzolanic additives, micro-reinforcing modifier additives and Sika superplasticizer was carried out. The range of variation of all additives is limited by the experimental plan.

The change in water resistance of composite gypsum binder under the influence of the above additives is described by the model (3):

$$Y(K_p) = +0.676v_1+0.209v_1v_2 + \pm0v_1x_4-0.033v_1x_5+0.069v_1x_6 + 0.711v_2+0.432v_1v_3 + +0.071v_2x_4-0.037v_2x_5\pm0v_2x_6 + +0.690v_3-0.437v_2v_3 + 0.027v_3x_4-0.071v_3x_5-0.071v_3x_6 + \pm0x_4^2-0.057x_4x_5 + \pm0v_1x_4^2-0.057x_4x_5 + \pm0v_2x_4^2-0.057x_4x_5 + \pm0v_3x_4^2-0.057x_4x_5 + +0.690x_4^2-0.057x_4x_5 + +0.027x_5^2+0.071x_5x_6 + 0.690x_5^2+0.071x_5x_6 + +0.690x_6^2+0.071x_6x_6 + +0.027x_6^2+0.071x_6x_6 + +0.056x_6^2+0.071x_6x_6$$

(a) (b) (c)

A graphical interpretation of the model in the form of two diagrams (triangles on a square) with a fixed content of additives Sika in the amount of 0.6 and 1.1% is presented in Figure 2.

Figure 2. The effect of polymineral micropuzzolanic additives, micro-reinforcing filler additives (wollastonite) on the water resistance of a composite gypsum binder

The softening coefficient varies in the range from 0.6 to 0.92. Most of the compositions of 24 series of samples are waterproof, because their softening coefficient is 0.8 – 0.92. The required conditions for research problems softening coefficient value obtained at the maximum $K_p \geq 0.85$ content metakaolin high activity in an amount from 10 – 15% m. c. microsilica content 5 – 10% of m.v. and subject to the content in the mixture of wollastonite a certain fraction. The fractional composition of wollastonite for different mixtures is different for different contents of Sika superplasticizer. The compositions, which in the framework of the experiment have the maximum values of water resistance $K_p = 0.9 – 0.92$, were obtained when 15% of highly active metakaolin and 5% of microsilica were added to the composition of composite gypsum binder. The content of Sika is 0.1 or 1.1% by weight. The choice of the fractional composition of wollastonite remains the task of the technologist and is assigned taking into account the levels of other properties and the conditions of the multicriteria optimization problem [16].

As follows from the analysis of the diagrams (Fig. 1-2), compositions that have a maximum compressive strength of 14.5 – 15.0 MPa are characterized by low water resistance $K_p = 0.7 – 0.75$. Compounds that have a maximum water resistance of $K_p = 0.85 – 0.92$ have $f_{cm} = 9.5 – 12.5$ MPa. In accordance with the purpose of the study, for further analysis of the properties, samples with $K_p \geq 0.85$ were selected. The strength of samples with such values of the softening coefficient is equal to $f_{cm} = 9.5; 10.5; 10 – 12.5$ MPa with the content of Sika superplasticizer in the amount of 0.1%, 0.6% and 1.1%, respectively.
The values of the softening coefficient \( K_p \geq 0.9 \) have compositions that contain superplasticizer Sika = 1.1%, highly active metakaolin = 15%, microsilica = 5%. That is, the specific surface area of the microreinforcing additive of wollastonite (Woll), acting as a structure modifier, is an effective factor that allows you to adjust the properties of composite mixtures in a wide range and should be assigned taking into account the content of microfillers.

The choice of the wollastonite fraction remains the task of the technologist. It is possible to use monofraction wollastonite \((\text{Woll}2 = 1)\), bifraction wollastonite \((\text{Woll}1 + \text{Woll}2 = 1)\), or polyfraction wollastonite \((\text{Woll}1 + \text{Woll}2 + \text{Woll}3 = 1)\). The choice of the fraction of wollastonite may be due either to the technological capabilities of the preparation of mixtures, or to indicators of the quality of other properties.

5. Analysis of the effect of water repellents on the water resistance of a composite gypsum binder.

At the next stage of the study, the possibility of a further increase in water resistance due to volume and surface water repellents was analyzed.

![Figure 3](image_url)

**Figure 3.** The shape of the droplets lying on a flat hydrophobic surface: (a) – “heavy” drop; (b) – “small” drop; \( \theta \) – limiting contact angle, \( t \) – tangent to the meridian section of a drop at the point of separation of the three phases.

To evaluate the effect of water-repellent primers on the surface properties of samples from a gypsum binder, we measured the wetting angle by the method of a lying drop (sitting drop method). The shape of a drop resting on a horizontal flat surface in a gravitational field (Fig. 3(a)) is axisymmetric and is described by the Young – Laplace equation [17]:

\[
\rho g y + \sigma \left( K(y) - K(h) \right) = 0,
\]

where \( \rho \) is the density of the liquid, \( g \) is the acceleration of gravity, \( \sigma \) is the surface tension of the liquid (specific free energy of the liquid-gas interface), \( K(y) = \left( R_1(y) \right)^{-1} + \left( R_2(y) \right)^{-1} \), \( R_1(y) \) and \( R_2(y) \) – principal radii of curvature of the surface of the drop at the ordinate point \( y \), \( K(h) = K(y) \bigg|_{y=h} \), i.e. on top of a drop.

Equation (4) has no analytical solution; therefore, numerical methods are used to calculate the droplet shape with high accuracy and to find the contact angle.

Small droplets under the action of capillary pressure are able to maintain a spherical shape (see Figure 3b). In this case, the contact angle can be easily found from elementary geometry \( \theta \). A drop can be considered small if its weight is much less than capillary pressure; the radius of a small spherical
droplet $r$ must satisfy the dependence $r << \left( \frac{2\sigma}{g\rho} \right)^{\frac{3}{2}}$ (here the gas density is considered negligible compared to the density of the liquid). Estimates show that this approximation is applicable to water droplets ($\sigma \approx 0.072 \text{ N/m}$) with a radius of about 0.4 mm or less.

For larger droplets of radius $r \leq \left( \frac{2\sigma}{g\rho} \right)^{\frac{3}{2}}$ (up to 3.5 mm) their meridian section can be approximately considered as a segment of a conical section - an ellipse [18]. In this case, measuring the sizes of the semiaxes of the ellipse $a$ and $b$ (see Fig. 3(a)), it is easy to determine the positions of its characteristic point $F_1$ and $F_2$, then find the equations of the focal radii $p$ and $q$ connecting the foci of the ellipse with the point of separation of the three phases. The bisector of the external angle formed by the intersection of these focal radii will be the tangent $t$ to the meridian section of the drop at the interface between the three phases. The angle of its inclination, which is easy to calculate, knowing the equations for $p$ and $q$, is complementary to the desired angle $\theta$.

In the experiment, the size of the liquid droplets is determined by the inner diameter of the dropper channel. In our case, it was 0.53 mm (with an outer diameter of the needle 0.8 mm). The masses of droplets of distilled water produced by the dropper were measured on an analytical balance. The needle used formed droplets with an average weight of 15.6 mg. The radius of the corresponding spherical drop is approximately equal to 1.55 mm, which, obviously, exceeds the criterion of smallness of the drop. To analyze the shape of such a drop, we decided to use the conical section method.

For photoregistration of the droplet shape on the test surface the CANYON CNR WCAM820 web camera with a resolution of 1600 × 1200 pixels was used. The standard camera lens was replaced with an optical system composed on the principle of a microscope. The field of view of the frame was about 9 mm, the image scale was determined using an object micrometer and was 5.75 $\mu$m / pixel. Two LED illuminators with brightness controls made it possible to combine oblique reflected and transmitted lighting to establish the optimal image contrast at the phase boundaries. During working with water-absorbing samples, the camera switched to video mode with a resolution of 800 × 600 pixels and with a frame interval of 100 ms.

The image obtained by the camera was saved on a computer and analyzed using a graphical editor. In the graphical editor an ellipse that satisfactorily coincided with the contour of the drop was selected. After that, the sizes of its semiaxes, the coordinates of the center and the coordinates of the points of separation of the three phases was read, then the contact angle was calculated by the method described above.

Figure 4 shows a drop of distilled water on the surface of a sample that has not been primed. The surface of the sample is hydrophilic, as evidenced by a contact angle of less than 90°. Water spreads and is absorbed by a gypsum binder: the drop on the figure shows at various time intervals that have passed since the moment of contact.

Water-absorbing surfaces can be characterized, in addition to the contact angle, by the rate of absorption through a single contact surface. The absorption rate can be estimated by the decrease in droplet volume over time, if the evaporation rate can be neglected, which is acceptable at a high absorption rate.

The calculation of the droplet volume was carried out under the assumption that the droplet is a truncated ellipsoid of rotation. It is easy to show that in this case the volume $V$ is determined by the height of the droplet $h$ and the dimensions of the semiaxes of its meridian section:

$$ V = \frac{\pi b^2}{3} \left( h - \frac{b}{3} + \frac{(b - h)^3}{3b^2} \right) $$

(5)
The experiments showed that the droplet absorption rate into the samples can be considered high, because during the time of complete absorption, the volume of the control drop on the metal surface remained unchanged within the measurement error.

On untreated samples, the contact angle decreases during the first two seconds of contact from 86° to 72°, but the drop volume remains unchanged within the measurement error, therefore, only the drop spreads. The start of absorption is recorded by us from the 3rd second. The maximum absorption rate is 5–6th second and amounts to 0.017 g·cm⁻²·s⁻¹. The total absorption time of the droplet varied in the range of 12-30 s with an average contact area of 0.15 cm².

Figure 5 are shown drop of distilled water on the sample surface treated with a hydrophobic primer. The surface of the sample acquired hydrophobic properties: the contact angle for water exceeds 90°. On these surfaces was not observed the spreading and absorption of a drop.

The average contact angle for gypsum surfaces treated with PGK-12S and PGK-24 primers is, respectively 129 ± 7° and 130 ± 5°. Taking into account the specified error, these contact angles of wetting can be considered the same, thus, primers PGK-12S and PGK-24 demonstrate the same efficiency in relation to hydrophobization of the gypsum binder surface. A slightly worse result is given by the WHITE primer, for which the average contact angle was 121 ± 4°.

The hydrophobizing primer introduction into the volume of a gypsum binder at the mixing stage without subsequent sample surface treatment leads to a different result. Fig. 6 shows the evolution of a distilled water drop on the surface of such a sample.

Immediately after the drop hits the surface and stabilizes its shape, the contact angles of wetting take on values exceeding 90°, which is typical for hydrophobic surfaces. The drop does not spread on the surface, and the area of the contact spot does not increase. However, the drop is absorbed into the sample volume. As the droplet volume decreases while maintaining the area of the contact spot, the contact angle decreases, and after a short time takes on the values characteristic of hydrophilic surfaces.
Figure 6. A drop of water on the surface of the sample with WHITE primer added to the volume of the binder after the onset of contact: (a) – 1.0 s (θ = 122°); (b) – 3.9 s (θ = 90°); (c) – 7.0 s (θ = 81°)

6. Conclusions
An analysis of the results of the study allows us to conclude that the proposed composition of the multifunctional modifier allows to obtain a waterproof modified composite gypsum binder with a softening coefficient $K_p \geq 0.9$ on gypsum grade G-5, rather than gypsum grade G-10 used for concrete.

It was shown that the complex modification of a composite gypsum binder on gypsum of the G-5 grade and Portland cement PC500-D0 with micro-micropozzolanic and micro-reinforcing filler additives and Sika superplasticizer in optimal proportions allow to obtain a composite gypsum binder with a softening coefficient of 0.9. That is, the specific surface area of the micro-reinforcing wollastonite additive, acting as a structure modifier, is an effective factor that allows you to adjust the properties of composite mixtures over a wide range and should be assigned taking into account the content of micro-fillers.

Volumetric hydrophobization provides an additional increase in compressive strength and water resistance. Further studies are aimed at obtaining and optimizing multifunctional modifiers with a wide spectrum of action.

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