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Adhesive Strength of Modified Cement–Ash Mortars

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Abstract: The main aim of this article, carried out in relation to ash–cement mortars, is to determine the effect of complex additives of polyfunctional modifiers, including, in addition to superplasticizers, air-entraining and water-retaining additives, at different values of water–cement ratios. With the use of experimental–statistical models, the complex effect on the adhesive strength of cement–ash mortars of water–cement and ash–cement ratios, as well as complex additives of polyfunctional modifiers, including air-entraining and water-retaining additives, is considered. The extreme nature of the water–cement and ash–cement ratios on the adhesive strength of ash–cement mortars are established. Their optimal values are in the ranges of 0.7–0.75 and 0.35–0.4, respectively. The addition of a naphthalene-formaldehyde superplasticizer makes it possible to increase the adhesive strength of mortars by up to 40%. A positive effect is achieved along with the addition of a superplasticizer by introducing optimal amounts of air-entraining and water-retaining additives into the mortar mixtures. Quantitative parameters of mortar compositions that positively affect adhesive strength are established. The influence on the adhesive strength of the fly ash was also investigated, as well as on the binder–sand ratio. In addition, a positive effect on the adhesive strength of modified cement–ash mortars was experimentally shown by increasing the specific surface area of fly ash by regrinding it and increasing the cement–sand ratio.

Keywords: adhesive strength; water–cement; ash–cement ratio; superplasticizer; air-entraining and water-retaining additives; fly ash



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1. Introduction

Adhesive mixtures are usually polymer–mineral systems containing mineral binders, fillers, and polymer additives that regulate the physico-mechanical and rheological properties of mortar mixtures and mortars [1,2].

The main requirement for hardened mortar mixtures is the strength of the adhesive bond [3]. It is crucial for many different building applications [4], including restoration work [5] and archeological applications [6]. Depending on the working conditions, the strength of the adhesive joints must comply with suitable regulations. One such applicable norm is Russian standard GOST R56387-2015. Their requirements are presented in Table 1.

The recipes for adhesive mixtures are quite diverse, and different additives are applied to the reinforced adhesive strength of concrete-based materials [7,8]. They are added when the content of Portland cement and quartz sand is between 25–40% and 25–75%, respectively. Reducing the consumption of cement and, in part, sand is achieved by introducing a dispersed mineral filler. Limestone flour is often used as a filler [9,10]. The

same effective filler is the active mineral additive—fly ash. The use of fly ash has a positive effect on the water-holding capacity of mortar mixtures, the strength of mortars, and their corrosion resistance. It also eliminates efflorescence formation and reduces shrinkage deformations [11,12]. At the same time, when fly ash as a component of dry mixtures, the stability of its chemical composition and the regulation of the content of unburned carbon particles become important [13]. The influence of the characteristics of the initial components of mortars, including ash-containing mortars, on their adhesive properties has not been sufficiently studied.

Table 1. Requirements for adhesive mixtures of various classes *.

Name of Indicator	Value for Class, MPa		
	C0	C1	C2
Strength of the adhesive bond after exposure to an air-dry environment for 28 days	≥0.5	≥0.5	≥1.0
Strength of the adhesive bond after exposure to the aquatic environment	-	≥0.5	≥1.0
Adhesive bond strength after exposure to high temperatures	-	≥0.5	≥1.0
Adhesive strength after cyclic freezing and thawing	-	≥0.5	≥1.0

Notes: * according to Russian standard GOST R56387-2015.

Due to the specificity and variety of phenomena that occur at different stages of the bonding process, the creation of a general theory of bonding (adhesion) becomes much more complicated [14]. One of the first theories proposed to explain the bonding process is McBain's hypothesis [15,16], which considers this process as a mechanical "wedging" of the adhesive into the pores (or depressions) of the bonded material. However, the provisions put forward by McBain have been refuted in subsequent works [3,16].

Currently, the adsorption, electrical, diffusion, and chemical theories of adhesion adsorption, electrical, diffusion, and chemical have gained the greatest importance [17,18]. None of these theories of adhesives are universal. Although none of these theories are currently preferred, it can be stated that each of them contributes to the general theory of the bonding mechanism [19,20].

Along with the introduction of polymer additives, there are several ways to improve the adhesive capacity of cement stone while limiting its content in concrete. One of them is based on the concept that considers cement stone as micro concrete [21,22]. According to this concept, it is advisable to increase the dispersion of the cement adhesive, ensuring its complete hydration. For cement grains larger than 40 microns, practically non-hydrated, it is rational to replace them with dispersed fillers. This concept is based on the technology of dry and wet grinding of cement together with sand and other fillers, as well as the production of colloidal cement glue [23]. However, cement grinding has not gained popularity due to high energy consumption, imperfection of the design of grinding units, and rapid loss of activity due to finely dispersed cement. The use of colloidal cement adhesive obtained using vibratory mills and mixers is limited to a narrow range of adhesive mixtures.

Significant progress has been made in studies on the activation of cement binders, as well as mixtures with fillers [23,24]. To the greatest extent, activation methods have been developed in relation to cement. The main ones are regrinding, vibration activation, turbulent, acoustic, ultrasonic, thermal, aerothermal, and treatment [24,25].

Currently, there are several recommendations for the activation of binders and fillers by modifying the surface with various chemicals, including surfactants, halogen-containing, and organosilicon substances [23,26]. The essence of mechanical methods of activation is to increase the reactivity of powders by opening new active surfaces of grains, changing the crystal structure of minerals, the formation of a large number of unsaturated valence bonds, and, with deep grinding, their amorphization occurs. The expediency of activating

the filler by modifying its surface with surfactant additives follows from the Dupre–Young equation [27], which relates the work of adhesion W_{ad} to the surface energy of a solid:

$$W_{ad} = \sigma_s - \sigma_s^*(m + \cos \theta) \quad (1)$$

where σ_s is the surface energy of the solid body; σ_s^* is the free surface energy of a solid body in an atmosphere of vapors and gases; $m = \sigma_l^*/\sigma_l > 1$ (σ_l^* is the surface tension of the liquid oriented under the influence of the force field of the solid surface; σ_l is the surface tension of the wetting liquid); and θ —contact angle of wetting.

It follows from the equation that, to achieve high adhesive strength, it is important to ensure the necessary wettability of the filler with a binder and to reduce the interfacial surface energy, which is achieved by treating the filler with a surfactant. The decrease in interfacial surface energy during the creation of an adsorption-active medium is determined from the following equation:

$$\Delta U = K \cdot T \int_0^c n_a(c) dlnc, \quad (2)$$

where ΔU is the difference between the interfacial surface energy without surfactant and in the presence of surfactant with concentration c ; n_a is the adsorption value determined by the number of surfactant molecules adsorbed on 1 cm² of the interface; K is the Boltzmann constant; and T is the absolute temperature in K.

A necessary condition for the effectiveness of surfactants is the ability of the chemisorption effect on the surface of the filler particles. In general, cationic surfactants are recommended [23] for mineral fillers and active mineral additives of the acid type, and anionic surfactants for the basic type.

The adhesive characteristics of cement–ash mortars are considered in a number of works [28,29]. However, in these works, the possibility of increasing the adhesive strength with complex chemical admixtures and, in particular, those that have an air-entraining and water-retaining effect, has not been studied. Quantitative dependences are not given, allowing one to evaluate the overall positive effect of the introduction of these additives, depending on the composition of mortars and dispersion of fly ash. To obtain such dependences, it is necessary to use mathematical models that consider the complex influence of the technological factors. Obtaining such models is possible, only on the basis of experimental data. For this purpose, the use of the mathematical planning of experiments is the most rational way. This methodology makes it possible to obtain, as a result of experiments performed according to a statistically effective plan, adequate (with 95% confidence) experimental–statistical models that produce quantitative estimates of the influence on a studied parameter of individual factors and the effects of their interaction.

The purpose of this work, conducted in relation to ash–cement mortars, is to determine, based on experimental–statistical models, the effect of complex additives of polyfunctional modifiers, including, in addition to superplasticizers, air-entraining and water-retaining additives, at different values of water–cement ratios. The influence of the adhesive strength of the dispersion and the binder–sand ratio was also studied.

2. Materials and Methods

In the work, Portland cement CEM-1 with a strength class of 42.5 MPa and fly ash from the Burshtyn Thermal Power Plant (Burshtyn, Ukraine), were used as components of the binder. The chemical composition of Portland cement and fly ash, as well as their physical properties, are presented in Tables 2 and 3.

The mineralogical composition of the clinker was as follows: C₃S—57.10%; C₂S—21.27%; C₃A—6.87%; C₄AF—12.19%.

The chemical composition of the ash satisfies the standard requirements as additives for mortars. To obtain ash with an increased specific surface, we ground it in a laboratory ball mill. The specific surface of the ash was measured using the method of

air permeability (according to Blaine) and the grain composition was determined by sedimentation analysis.

Table 2. Chemical composition of raw materials.

Name Material	Oxide Content, %									
	L.O.I.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	CaO _{free}
Clinker	-	21.80	5.32	4.11	66.80	0.95	0.63	0.54	0.42	-
Fly ash	5.1		84.5		2.1	2.0	2.3		1.2	2.5

Table 3. Physical properties of fly ash and Portland cement.

Indicators	Portland Cement	Unground Ash	Ground Ash
Specific surface, m ² /kg	350	290	455
Fraction content, μm, %			
up to 20	-	17.8	29.7
20–40	-	35.9	40.25
40–80	-	32.6	27.3
>80	8	13.7	2.75
Normal consistence, %	25.2	26.0	28.5
Absorption activity CaO, mg/g	-	36.3	52.1
Compressive strength, MPa in 28 days	53.4	-	-

In the investigation, powdered naphthalene formaldehyde superplasticizer SP-1 was used. The SP-1 is a product of condensation of naphthalite sulfonic acids and formaldehyde. The content of the “active substance” in it was at least 69%, the ash content was not more than 38%, and the pH of a 2.5% aqueous solution was between 7 and 9%.

As air-entraining admixture (Air_{ad}), a dry powdery admixture “Mix-DH” was used—a mixture of synthetic air-entraining components and sodium salts of abietic acid.

As a water-retaining admixture, we used cellulose ethers (ECs)—methylhydroxyethyl-cellulose Tylose MH 15002P6—a product of the substitution of hydrogen atoms of cellulose hydroxyl groups for alcohol residues, obtained as a result of the process of activation of cellulose with sodium hydroxide and its subsequent esterification with methylene chloride and ethylene oxide. Silica sand with module fineness $M_f = 1.42$, content of dust and clay impurities 1.7% was used as mortar aggregates.

Mortar samples were made in a cement–ash binder ratio:sand = 1:3 (by weight). To determine the adhesive strength, the hardening of strength was conducted at a humidity of 90% and a temperature of 20 ± 2 °C.

The adhesive ability of the cement–ash mortars was determined as the peeling strength of the concrete base of a sample of 50×50 mm in size, cut from ceramic tiles. The influence of the water–cement (W/C) and ash–cement (A/C) ratios, as well as the type and content of the additives, was studied using experiments algorithmized according to plan B₄ [30].

3. Results and Discussion

The statistical processing of the results of the experiment was performed according to the three-level plan B₄ [30], including the necessity to take into account the testing of 24 series of samples, the compositions of which are determined by the matrix and are in the range specified in Table 4. The experimental data are shown in Table 5. As a result of the statistical processing of the data in Table 5, mathematical models were obtained, presenting the adhesive strength (Table 6).

Table 4. Conditions for planning an experiment on the study of adhesion properties of modified cement–ash mortars.

Technological Factors		Levels of Variation		
Natural view	coded view	−1	0	+1
Water–cement ratio (W/C)	X ₁	0.6	0.8	1.0
Ash–cement ratio (A/C)	X ₂	0	0.35	0.7
The content of the superplasticizer, % of the mass of cement	X ₃	0	0.35	0.7
The content of the air-entraining additive (Air _{ad}), % of the mass of cement	X _{4(I)}	0	0.025	0.05
The content of the water-retaining additive Tylose (EC), % of the mass of cement	X _{4(II)}	0	0.15	0.3

Table 5. Planning matrix and experimental adhesion-strength values.

Test No.	Factors				Polyfunctional Modifier PFM ₁ (SP + Air _{ad})		Polyfunctional Modifier PFM ₂ (SP + EC)	
	X ₁	X ₂	X ₃	X ₄	R _{ad,7} , MPa	R _{ad,28} , MPa	R _{ad,7} , MPa	R _{ad,28} , MPa
1	+1	+1	+1	+1	0.22	0.41	0.28	0.54
2	+1	+1	+1	−1	0.18	0.35	0.21	0.42
3	+1	+1	−1	+1	0.15	0.35	0.21	0.38
4	+1	+1	−1	−1	0.11	0.29	0.14	0.27
5	+1	−1	+1	+1	0.23	0.38	0.27	0.46
6	+1	−1	+1	−1	0.19	0.32	0.19	0.33
7	+1	−1	−1	+1	0.16	0.37	0.19	0.36
8	+1	−1	−1	−1	0.12	0.31	0.12	0.25
9	−1	+1	+1	+1	0.28	0.45	0.33	0.58
10	−1	+1	+1	−1	0.25	0.41	0.26	0.48
11	−1	+1	−1	+1	0.21	0.39	0.26	0.42
12	−1	+1	−1	−1	0.18	0.35	0.20	0.33
13	−1	−1	+1	+1	0.27	0.44	0.32	0.52
14	−1	−1	+1	−1	0.24	0.39	0.25	0.42
15	−1	−1	−1	+1	0.20	0.42	0.24	0.42
16	−1	−1	−1	−1	0.17	0.38	0.18	0.34
17	+1	0	0	0	0.28	0.53	0.31	0.58
18	−1	0	0	0	0.34	0.59	0.36	0.64
19	0	+1	0	0	0.31	0.57	0.36	0.66
20	0	−1	0	0	0.31	0.57	0.34	0.62
21	0	0	+1	0	0.37	0.60	0.41	0.70
22	0	0	−1	0	0.30	0.57	0.33	0.58
23	0	0	0	+1	0.33	0.61	0.38	0.69
24	0	0	0	−1	0.29	0.56	0.32	0.59

Table 6. Mathematical models of the adhesive strength of cement–ash mortars.

Additive	Mathematical Models of Adhesive Strength	
Polyfunctional modifier PFM ₁ (SP + Air _{ad})	$R_{ad,7} = 0.356 - 0.028X_1 + 0.035X_3 + 0.017X_4 - 0.0459X_1^2 - 0.0459X_2^2 - 0.0209X_3^2 - 0.0459X_4^2 - 0.004X_1X_2$	(3)
	$R_{ad,28} = 0.639 - 0.028X_1 + 0.018X_2 + 0.057X_3 + 0.026X_4 - 0.0792X_1^2 - 0.074X_2^2 - 0.0542X_3^2 - 0.0542X_4^2 + 0.004X_1X_2 + 0.004X_1X_4 + 0.011X_2X_3$	(4)
Polyfunctional modifier PFM ₂ (SP + EC)	$R_{ad,7} = 0.392 - 0.027X_1 + 0.007X_2 + 0.036X_3 + 0.034X_4 - 0.0574X_1^2 - 0.0424X_2^2 - 0.0224X_3^2 - 0.0424X_4^2 + 0.002X_1X_4 + 0.002X_3X_4$	(5)
	$R_{ad,28} = 0.706 - 0.030X_1 + 0.021X_2 + 0.060X_3 + 0.052X_4 - 0.097X_1^2 - 0.067X_2^2 - 0.067X_3^2 - 0.067X_4^2 + 0.006X_1X_2 + 0.006X_1X_4 + 0.016X_2X_3 + 0.003X_3X_4$	(6)

The effect on the value of the adhesive strength was studied, as well as the ratio of cement–ash binder: aggregate. The results of the experiments are given in Table 7.

Table 7. Adhesion strength of modified cement–ash mortars using ash of various dispersions.

(Cement + Ash)/Sand (C + A)/S	Specific Surface $S_s, m^2/kg$	Adhesion Strength, MPa, Aged	
		7 Days	28 Days
Modifier PFM ₁ (W/C = 0.8; A/C = 0.6; SP = 0.5%; Air _{ad} = 0.03%)			
1:3	290	0.34	0.60
	340	0.38	0.66
	390	0.43	0.71
1:4.5	290	0.28	0.53
	340	0.32	0.57
	390	0.35	0.62
Modifier PFM ₂ (W/C = 0.8; A/C = 0.6; SP = 0.5%; EC = 0.3%)			
1:3	290	0.38	0.64
	340	0.42	0.69
	390	0.45	0.73
1:4.5	290	0.29	0.55
	340	0.31	0.59
	390	0.33	0.62

An analysis of the adhesive strength shows that the influence of both W/C and A/C on it is significant (Figures 1 and 2). Therefore, with an increase in the W/C mortar from 0.6 to 0.7–0.75, the adhesive strength increases by 8–10%; with a further increase in W/C, it decreases by 20–25% of the maximum values. In this case, the maximum R_{ad} is observed at A/C = 0.35–0.4. This effect of the mentioned technological factors on adhesion can be explained by the influence of not only the porosity of the contact layer, but also the degree of wetting with the base mortar [31,32].

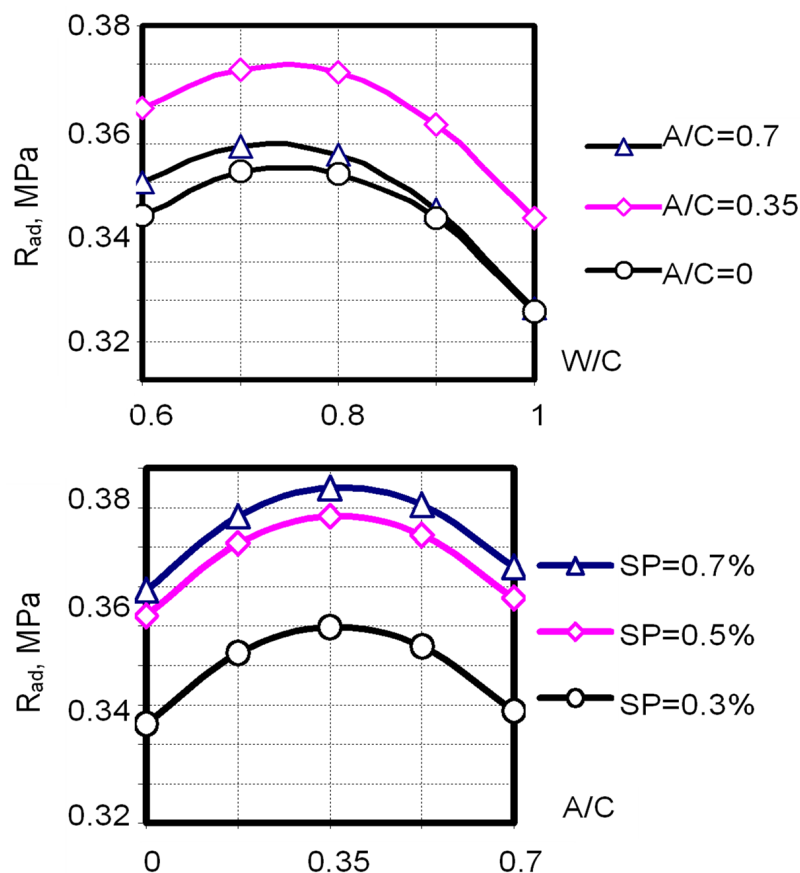


Figure 1. Dependence of the adhesive strength (MPa) of PFM₁-modified cement–ash mortars on water–cement and ash–cement ratios at 7 days.

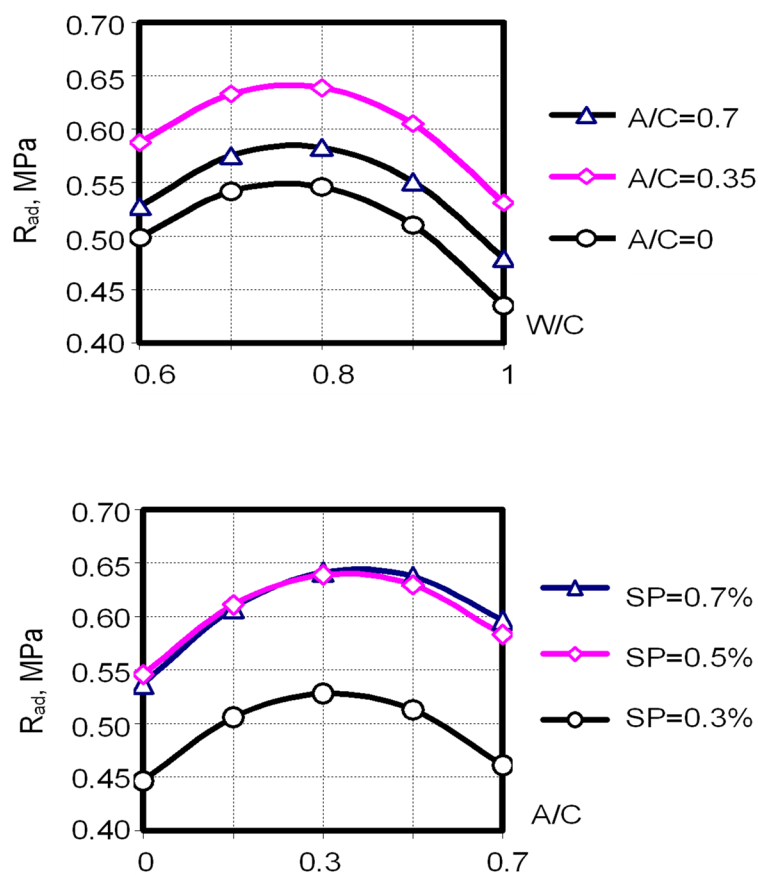


Figure 2. Dependence of adhesive strength (MPa) of cement ash mortars modified with PFM₁ from the water–cement and ash–cement ratios at 28 days.

The superplasticizer (SP–1) and the air-entraining admixture MixDH have a positive effect on the adhesion of mortars (Figure 3), as a result of a change in their surface energy and a change in the qualitative characteristics of the contact layer. The introduction of the hydrophilic additive SP improves the mortar characteristics of the contact layer, obviously, primarily as a result of improving its wettability and reducing the content of excess moisture. With an increase in the superplasticizer content from 0 to 0.35% by weight of cement, the adhesive strength increases by 16–35% at $W/C = 0.6$ and by 25–40% at $W/C = 1.0$. A further increase in the content of SP to 0.7% leads to another increase in R_{ad} by another 13–20%.

The introduction of the air-entrainment and, especially, polymeric additive Tylose into the mortars provides the necessary water-retention capacity of the mortar mixture and reduces the thickness of the adhesive layer, which also has a positive effect on the adhesion value [33]. *Ceteris paribus*, an increase in the content of air-entraining additive (Air_{ad}) from 0 to 0.025% by weight of cement increases the adhesive strength by 30–45%; with a further increase in the content of Air_{ad} , the adhesive strength decreases slightly. An increase in the content of the Tylose water retention additive from 0 to 0.15% leads to an increase in adhesive strength by 25–55%; a further increase in the content of the additive has little effect on the adhesion of mortars containing ash.

An additional factor that contributes to an increase in the adhesive strength of mortars is an increase in the dispersion of fly ash (Table 7). Therefore, the regrinding of ash to a specific surface area of 390 m²/kg makes it possible to increase the adhesion strength of the mortar with the 7-day-old base by 14–26%, at 28 days old—by 13–18%.

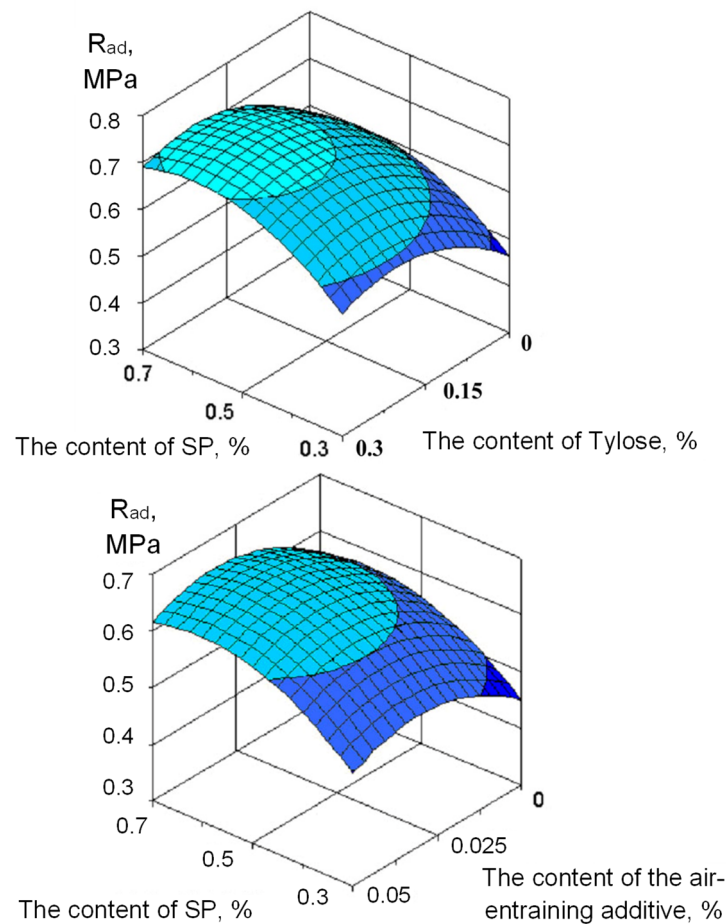


Figure 3. Dependences of adhesive strength (MPa) of cement–ash mortars at 28 days on the content of modifier additives.

Freshly formed surfaces of mineral materials are known to have significantly higher surface energy, which determines their high adhesive activity [34,35]. Mechanical processes during grinding of mineral and organic materials cause, along with an increase in their surface energy, an increase in the isobaric potential of powders and, accordingly, their chemical activity, which also contributes to high adhesive strength when they come into contact with a binder. However, the tendency of ground powders to rapidly deactivate should be considered. The duration of the existence of radicals in the air environment, which arise during mechanochemical treatment, is only 10^{-3} – 10^{-6} s [27]. The adsorption of water vapor and carbon dioxide from the air by freshly milled powders and the saturation of uncompensated molecular forces lead not only to the “aging” of the filler surface, but also serve as an additional barrier to the formation of reliable adhesive contacts. Therefore, the mechanochemical activation of fillers is effective when a primary contact layer of a structured binder is created on the grains directly in the grinding process.

Changes in the ratios of the cement–ash binder and the sand also affect the adhesive strength.

4. Conclusions

The results obtained show the effect of complex additives of polyfunctional modifiers, including, in addition to superplasticizers, air-entraining and water-retaining additives, at different values of water–cement ratios. Moreover, the influence of the adhesive strength of the fly ash dispersion, as well as the binder–sand ratio, was presented. The provided investigations allow us to formulate the following conclusions:

- The effect of the water and ash cement ratios on the adhesive strength of the ash–cement mixtures was studied. A significant decrease in adhesive strength occurred when the W/C ratio exceeded 0.75 and the ash–cement ratio was 0.4.
- The introduction of a superplasticizer additive of the naphthalene-formaldehyde type increased the adhesive strength of the mortar in the studied dose range. An increase in adhesive strength was also observed with the additional introduction of an air-entraining additive up to 0.025 by weight of cement.
- A significant increase in adhesive strength was also observed with the introduction of a complex modifier additive, including a superplasticizer and a water-retention additive Tylose in an amount of up to 0.15% of the mass of cement.
- For cement–ash mortar mixtures, an increase in adhesive strength was characteristic with an increase in the dispersion of ash and in the ratio of cement–ash binder to sand aggregate.

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