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Fillers for modification of polyester powder coating

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Abstract. In the article effectiveness of the use of fillers to improve the physical and mechanical properties of the coating varied depending on the average size of the filler particles and crystalline shape was studied. According to the indicators of impact strength to back impact and bending strength of the coating, the most effective is the use of microsilica with an average size of 2.9 μm . As the average filler size increases, the gloss and impact strength of the coating reduce. To increase the bending strength of the coating, it is advisable to use fillers in the form of wollastonite, which is characterized by a fibrous crystal shape, which improves the physical and mechanical properties of the coating and prevents cracking, acting as a reinforcing agent. In addition, the analysis of the obtained study results shows that the use of the studied Ukrainian-made fillers in the powder paint composition promotes obtaining a covering with adjustable physical and mechanical characteristics.

1. Introduction

With the strengthening of environmental requirements and the need to adapt Ukrainian legislation on quality and safety of construction materials to EU requirements, the priority direction of development is to minimize the negative impact of harmful substances in their composition on consumer health and the environment. This issue is especially acute for goods of the paint and varnish industry using volatile organic compounds [1-3]. In recent years, the world has paid considerable attention to the environmental aspects of production and use of paint and varnish materials, which has led to a steady increase in production and use of powder paints [4]. It should be noted that in Ukraine the powder paints are also becoming more common due to their environmental friendliness by virtue of the absence of harmful solvents, ease of use and storage, as well as obtaining a durable coating [5]. The factor of their efficiency is the durability of coatings. In most cases the coatings of powder paints show higher protective properties and have a longer service life than coatings of solutions and dispersions of polymers [6]. This is due to the fact that powder paints do not contain mediators of film formation (solvents, water) and surfactants, and are prepared mainly on thermosetting oligomers and polymers of crystalline structure with high chemical resistance. Therefore, further numerous experiments to improve the prescriptions will contribute to the rapid development of technology of manufacturing powder coatings and their application to construction products, which will reduce economic costs and increase the durability of the material.

2. Analysis of the latest research works and publications

The prescribed composition of the powder coating consists of five main components: polymer resin, hardener, pigments, functional additives and filler. In general, the polymer resin and hardener play a



major role in providing the required mechanical properties and durability of the powder coating. The introduction of pigments in paint and varnish materials is the main method of regulating the decorative properties of coatings - color and opacity (coverability). The additives are used to regulate the technological properties of powder paint and performance characteristics of the obtained coating. The fillers are used for two main purposes: the first is to reduce the cost of the material by replacing the most expensive component in the form of a polymer resin; the second is to modify the functional properties of the material, such as hardness, gloss, bending and impact strength, modulus of elasticity; permeability; corrosion, wear resistance; fire resistance.

The term “filler” is very broad and covers a wide range of materials. Most of fillers used in powder paints are inorganic and are usually extracted from rocks or ores, and with subsequent processing are converted into powders [7].

The most common fillers used in prescribed compositions of powder paints are barium sulfate (BaSO_4), calcium carbonate (CaCO_3) and talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$). As a rule, increasing their content in the powder paint helps to reduce the viscosity and maximum elongation of the system with increasing hardness of the coating [8]. In addition to their commonality, each filler also has its own unique effect. For example, the advantages of barium sulfate include its high corrosion and weather resistance [9]. Calcite contributes to the uniformity of the dispersion of the powder paint and the ability to obtain a matting effect of the coating. Talc has a high latent ability, matting effect and helps to obtain a satin type of coating.

However, the nomenclature of fillers by type and chemical structure is quite diverse, which is of great interest in the study of their effect on the properties of powder coatings [10]. This is especially true of Ukrainian-made fillers, as the main components for the production of powder paint are imported from abroad. Therefore, determining the effectiveness of the use of various Ukrainian-made fillers in systems of powder paints and coatings provides valuable information about their choice.

3. Definition of the goal and objective of the research

The objective of the work is to study the physical and mechanical properties of decorative and protective powder coatings using different types of fillers.

Materials and methods of studies. Rationale for the choice of raw materials for powder coatings. Prescribed composition of powder paint (PP) consisted of the following components: film-forming component, filler, pigments and functional additives.

Crylcoat 2441-3T carboxyl-containing polyester resin produced by Alnex was used as a film-forming component. Its characteristics are given in the Table 1. For carboxyl-containing polyester resin, the use of a structure-forming hardener is also mandatory. This paper uses TGIC produced by Huangshan.

Table 1. Characteristics of the film-forming component.

Type of the resin	Appearance	Gloss 200/600, %	Viscosity (cP at 200 °C)	Indexes		
				Color Gardnes (10 % in DMF)	Acid Value (mg KoH/g)	Temperature cure and time
Allnex 2441-3T	Opaque granules	67	4000-5200	10	30-35	200°C 10 min

K-2190 titanium dioxide produced by Kronos was used as the white pigment. Resiflow PV88 agent produced by Estron chemical in the amount of 1 % by weight of PP was used as a rheological additive. Benzoin produced by Estron chemical in the amount of 0.6 % by weight of PP was used as a degasser.

Fillers of different chemical nature were used in the studies: 1 - salt in the form of *barium sulfate* produced by Changsha Lianda, *lime* produced by the Private Joint-Stock Company “Sumiagroprombud”; 2 - oxide in the form of *ZnO* produced by EverZinc; 3 - silicates in the form of *metakaolin* of Glukhovets deposit, *microsilica no. 1* produced by the Limited Liability Company

"Teravion", *microsilica no. 2* produced by the Limited Liability Company "Mining Engineering Company", *wollastonite* produced by Minco LTD, 4 - man-made product in the form of *fly-ash* produced by Ladyzhyn Thermal Power Station. Characteristics of fillers are given in the Table 2.

Table 2. Characteristics of the fillers.

Fillers	Appearance	pH value	Oil absorption, G/100g	Humidity, %	The average particle size, μm	Crystalline form of the filler
Barium sulfate	white powder	7,86	16,1	0,074	1,87	cubical
Fly ash	grey powder	6,7	19,4	0,042	16,43	spherical
Microsilica No 1	grey powder	6,9	27,3	0,15	15,2	spherical
Microsilica No 2	grey powder	6,8	25,1	0,12	2,9	spherical
Wollastonite	white powder	8,2	38,4	0,1	14,02	needlelike
Lime	white powder	8,6	17,2	0,21	5,47	cubical
Metakaolin	white powder	6,1	35,4	0,03	10,03	lamellar
ZnO	white powder	8,4	31,2	0,062	2,32	cubical

Study methods. The study of the properties of decorative and protective powder coatings using different types of fillers was carried out in the following sequence:

1. Using different types of fillers, the powder paint (color RAL 9016) was applied on plates (sized 150x60 mm) made of steel St3. The powder paint was applied by electrostatic method according to ISO 1514: 2016 using a spray gun Start 50.
2. Polymerization of the powder coating on the samples-plates was carried out in a polymerization furnace at a temperature of 200 °C within the period of 10 min.
3. Study of the coating gloss was performed according to ISO 2813: 2014.
4. Study of the coating adhesion was performed by the cross cut method according to ISO 2409-2013.
5. Determination of the impact strength of the coating to back impact was performed according to ISO 6272-2.
6. Determination of the bending strength of the coating was performed according to ISO 1519: 2011.

4. Basic part of the researches

Experimental studies of the effect of fillers of different chemical nature on gloss, adhesion, impact and bending strength of the coating were conducted in the laboratory of the Limited Liability Company "Lacover". The compositions of the studied paint and varnish powder coatings are given in the Table 3.

As a result of study, it is established that the change of chemical nature, average size of particles and crystalline form of filler in the composition of powder paint affects the physical and mechanical properties of the coating.

It is shown that the use of *barium sulfate* in the powder paint (control composition) contributes to a high gloss of the coating, which is 91 % (Fig. 1) while providing impact strength to back impact at the level of 30 cm / kg (Fig. 2), adhesion of the Gt0 class (Fig. 3) and bending strength, which corresponds to the diameter of the bending shaft of 10 mm (Fig. 4).

However, the use of filler in the form of *lime* in the powder paint composition reduces the gloss and impact strength to back impact of the coating (Fig. 1, Fig. 2) to 82 % and 20 cm/kg, respectively, compared with the control composition (filler in the form of barium sulfate) - gloss 91%, impact strength to back impact 30 cm/kg. This can be explained by the change in diffusion reflection of light by increasing the average size of the filler particles in the form of chalk compared to barium sulfate (Table 2) and the deterioration of the powder paint dispersion uniformity, which determines the reduction of gloss and impact strength. It should be noted that the adhesion and flexural strength of the coating does not change compared to the control composition, which is Gt0 and 10 mm, respectively.

Table 3. Compositions of the powder coating.

No	Resin	Fillers	Compositions of the powder coating, %				
			Resin	Hardening agent	Filler	TiO ₂	Additive
1	Crylcoat 2441-3T	Barium sulfate	55,8	4,2	18,4	20,0	1,6
2		Fly ash	55,8	4,2	18,4	20,0	1,6
3		Microsilica No 1	55,8	4,2	18,4	20,0	1,6
4		Microsilica No 2	55,8	4,2	18,4	20,0	1,6
5		Wollastonite	55,8	4,2	18,4	20,0	1,6
6		Lime	55,8	4,2	18,4	20,0	1,6
7		Metakaolin	55,8	4,2	18,4	20,0	1,6
8		ZnO	55,8	4,2	18,4	20,0	1,6

The use of oxide in the form of **ZnO** to improve the mechanical properties of the powder coating is ineffective. Thus, the introduction of ZnO into the powder paint composition leads to a decrease in impact strength to 10 cm/kg (Fig. 2) and flexural strength, which corresponds to a diameter of the bending shaft of 12 mm (Fig. 4). The adhesion of the coating using ZnO corresponds to the Gt0 class (Fig. 3). It should be noted that the use of ZnO in the powder paint also reduces the gloss to 77 %.

In contrast to ZnO, the use of silicates in the form of **microsilica no. 1** to improve the mechanical properties of the powder coating is effective. Thus, with the introduction of microsilica no. 1 there is an increase in impact strength to 50 cm/kg (Fig. 2) and bending strength (8 mm) of the coating compared to the control composition. This is due to the increase in the compaction coefficient of the system by changing the crystalline form of the filler from cubic (ZnO) to spherical (microsilica), which determines the increase in the mechanical properties of the coating. The adhesion of the coating using microsilica corresponds to the Gt0 class (Fig. 3). The gloss of the coating is 84 %.

However, increasing the average size of the filler particles (Table 2) from 2.9 µm (microsilica no. 1) to 15.2 µm (**microsilica no. 2**) leads to a decrease in the mechanical properties of the coating. Thus, the use of microsilica no. 2 with an average particles size of 17.2 µm helps to reduce impact strength to back impact (10 kg / cm) and bending strength (14 mm) compared to the control composition. It should be noted that increasing the size of the filler particles leads to a decrease in the gloss of the coating to 64%.

The use of filler in the form of **fly ash** reduces the gloss to 51 % compared to the control composition - gloss 92 % (Fig. 1). This is explained by the change in the diffusion reflection of light by increasing the average size of the filler particles in the form of fly ash compared to barium sulfate (Table 2), which determines the decrease in the gloss of the coating. The impact strength to back impact and the bending strength of the coating using fly ash is similar to the strength of the control composition, which is 30 kg/cm and 10 mm, respectively (Fig. 2, Fig. 4). The adhesion of the coating using fly ash corresponds to the Gt0 class (Fig. 3).

The introduction of the filler in the form of **wollastonite** also reduces the gloss from 94 % (control composition) to 62 % by increasing the average size of the filler particles from 1.87 (barium sulfate) to 14.0 (wollastonite). There is an increase in the bending strength of the coating, which corresponds to the diameter of the bending shaft 8 mm (Fig. 4). This is due to the change in the crystalline form of the filler from cubic (barium sulfate) to fibrous, which improves the physical and mechanical properties of the coating and prevents the formation of cracks, acting as a reinforcing agent. The adhesion of the coating using wollastonite corresponds to the Gt0 class (Fig. 3).

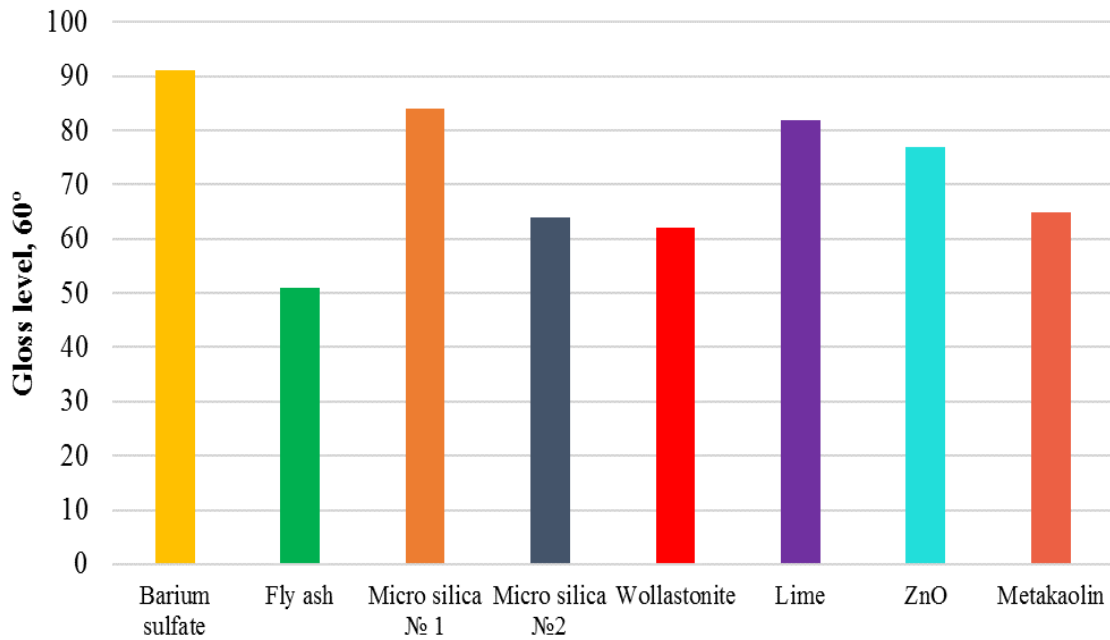


Figure 1. Gloss level of the powder coating depending of the type of filler (Table 2).

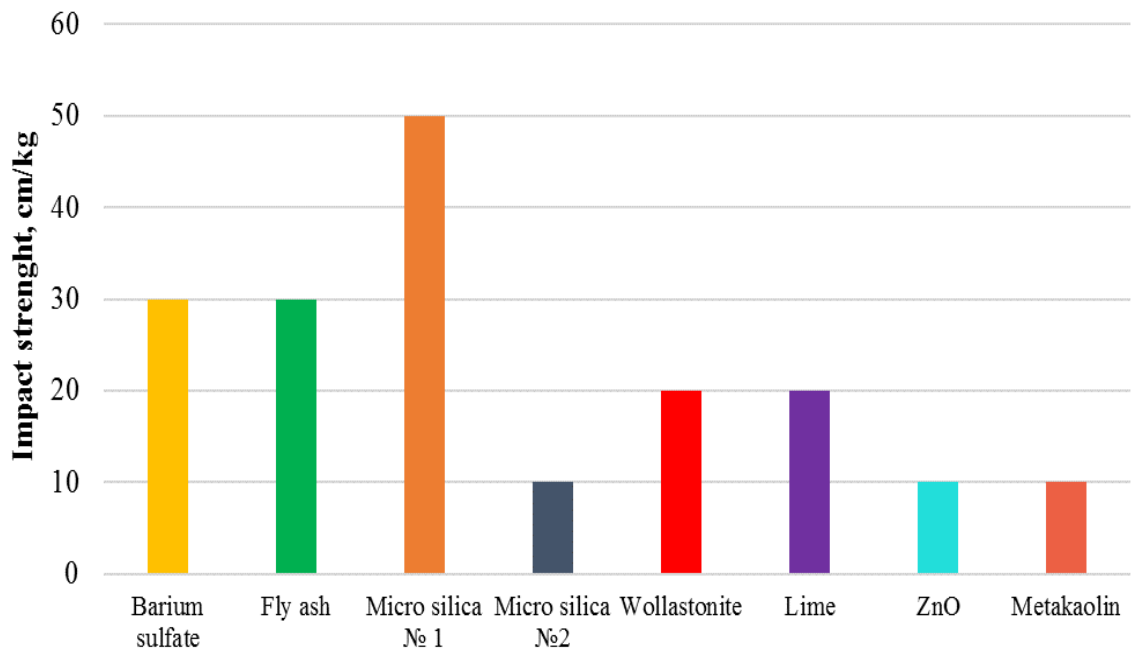


Figure 2. Impact strength of the powder coating depending of the type of filler (Table 2).

The use of *metakaolin* to improve the mechanical properties of the powder coating is ineffective. Thus, the introduction of metakaolin into the powder paint composition reduces the impact strength to back impact (10 cm/kg) and bending strength, which corresponds to the diameter of the bending shaft of 12 mm (Fig. 2, Fig. 4). The adhesion of the coating using metakaolin corresponds to the Gt0 class (Fig. 3). However, its introduction helps to reduce the gloss of the coating to 65 % (Fig. 1) by increasing the average size of the filler particles (Table 2).

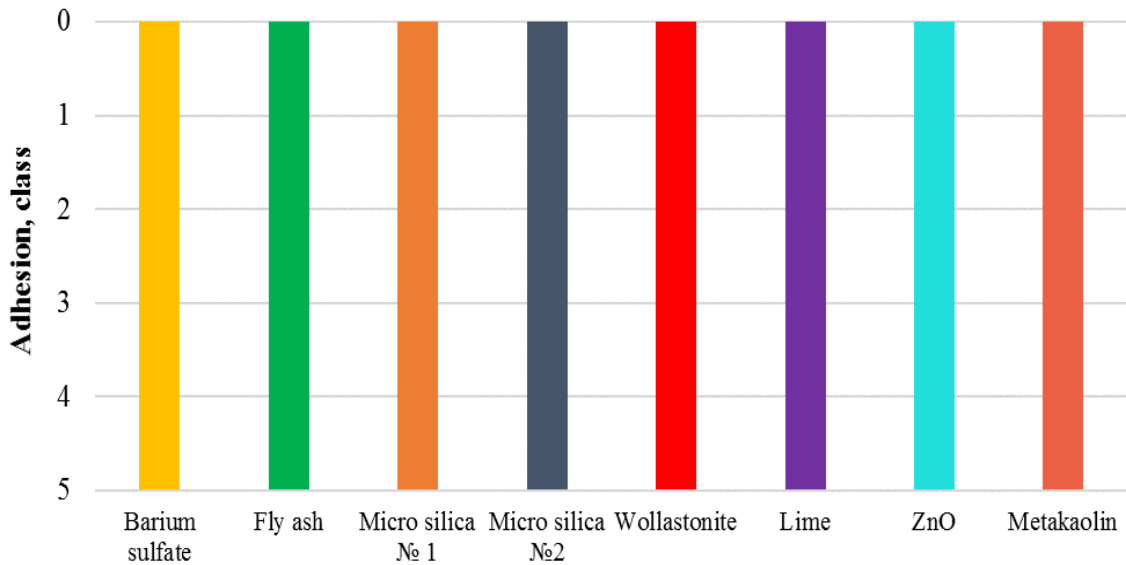


Figure 3. Adhesion of the powder coating depending of the type of filler (Table 2).

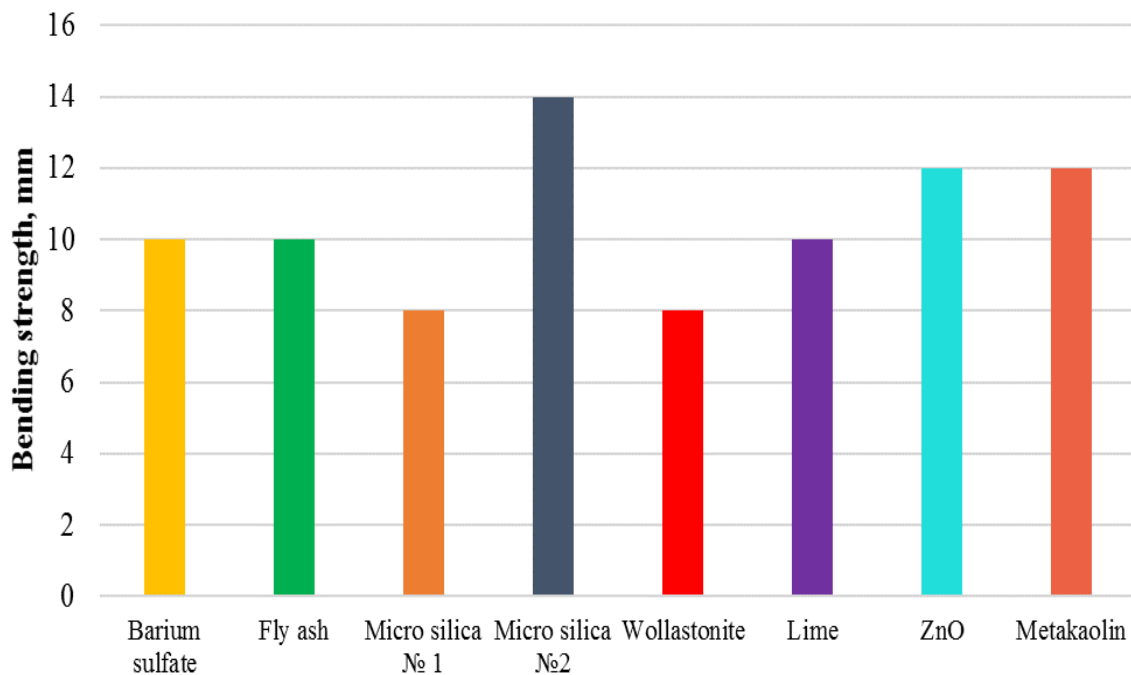


Figure 4. Bending strength of the powder coating depending of the type of filler (Table 2).

Thus, the replacement of fillers in the powder paint composition affects the change in gloss, impact strength to back impact and bending strength of the coating depending on the average size and crystalline shape of the filler. Increasing the average size of the filler particles reduces the gloss of the coating by changing the diffuse reflection of light on the surface of the coating. Changing the crystalline shape of the filler from cubic to spherical helps to obtain a denser structure of the system with increasing mechanical properties of the coating. When using a filler of fibrous structure, there is an increase in the bending strength of the coating.

5. Conclusions

The study has found that the effectiveness of the use of fillers to improve the physical and mechanical properties of the coating varied depending on the average size of the filler particles and crystalline shape. According to the indicators of impact strength to back impact and bending strength of the coating, the most effective is the use of microsilica with an average size of 2.9 μm . As the average filler size increases, the gloss and impact strength of the coating reduce. To increase the bending strength of the coating, it is advisable to use fillers in the form of wollastonite, which is characterized by a fibrous crystal shape, which improves the physical and mechanical properties of the coating and prevents cracking, acting as a reinforcing agent. In addition, the analysis of the obtained study results shows that the use of the studied Ukrainian-made fillers in the powder paint composition promotes obtaining a covering with adjustable physical and mechanical characteristics.

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