CORROSION AND ELECTROMAGNETIC SHIELDING PROPERTIES OF POWDER PAINT COATINGS PREPARED FROM GALVANIC WASTE

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Abstract. This work is focused on the study of the properties of powder paint coatings modified with fillers, which are obtained from ferritization waste processing of spent technological solutions of galvanic industries. The physical, mechanical and shielding properties of powder systems with iron-containing products introduced into their composition were investigated in the work. It is shown that the use of iron-containing waste in general contributes to the increase of both mechanical characteristics and corrosion resistance of coatings. It was determined that the introduction of $Ni_{0.5}Cu_{0.5}Fe_2O_4$ and $Zn_{0.5}Cu_{0.5}Fe_2O_4$ into the composition allows to significantly increase their mechanical and shielding properties.

Keywords: powder paint, coating, corrosion, galvanic waste, ferritization, electromagnetic shielding, ferrites


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INTRODUCTION

The development of the construction sector leads to increased requirements for the quality and reliability of protection of construction products and structures, including metal, in conditions of complex action of destructive effects, namely: temperature, corrosive environment, erosion, mechanical stresses, etc. The most common way to protect metal products and structures from destructive effects is the surface application of liquid paint and varnish materials [1]. The main disadvantage of using liquid paint and varnish materials is the solvents content in their composition up to 40%, which leads to their emissions during production and application, as well as environmental pollution.

Therefore, the development of

environmentally safe and high-quality paint and varnish compositions that don't contain organic solvents and other harmful substances is relevant. The most promising way for the metal products and structures protection is the use of powder paints [2]. The high efficiency of these materials is associated with the possibility of obtaining high-quality corrosion and chemically resistant protective coatings with a thickness of 30... 80 microns during single-layer application. The world production of powder coatings is about 80% of the total amount of powder paint materials, that confirms their effectiveness [3]. The formulation of the powder coating consists of the following components: polymer resin, hardener, pigments, functional additives and filler [4]. The polymer resin and hardener play a major role in providing the desired mechanical characteristics and durability of the powder coating. The introduction of fillers into paint materials is used for two main purposes: the first is to reduce the coating material cost; the second - increasing the functional properties of the material, such as hardness, bending strength and impact, modulus of elasticity; permeability; corrosion, wear resistance; fire resistance [5]. Most fillers used in powder paints are inorganic and are usually extracted from rocks or ores, and then processed into powders [6]. Therefore, in modern conditions, research is relevant, which is aimed at expanding the use of various types of fillers in powder paints, in particular industrial waste, taking into account technical, economic and operational indicators.

One of the types of fillers for powder paints can be used the liquid galvanic waste processing products. Flushing wastewater and spent highly concentrated solutions are sources of environmental pollution in the galvanic production [7]. This liquid galvanic waste can't be discharged into the central sewage systems due to its danger to the environment, given the presence of various heavy metal compounds. Storages of wastewater treatment products in production also carries increased environmental risks, worsens the economy of production and requires constant supervision of this waste [8]. For a now, there is an urgent need to use effective technologies for the processing of spent technological solutions and the disposal of wastewater treatment waste accumulated directly at industrial enterprises or at specially designated places for their storage.

The ferritization technology of heavy metals is one of the most effective ways of galvanic waste processing in order to obtain safe waste [9]. Ferritization treatment of liquid galvanic wastes results in ferrite sediments of hazard class IV [10]. These deposits(sediments) have a crystalline structure and are quickly sedimented during processing, and their volume and humidity are much lower than in sediments obtained from traditional reagent treated wastewater [11]. In addition, the resulting ferrite deposits are ferrimagnetic, and therefore they can be used to shield electromagnetic radiation.

Thus, the aim of this study is to obtain and determine the properties of powder paint coatings that are modified with fillers obtained from the waste of ferritization processing of spent technological solutions of galvanic production. The creation of such technology will facilitate the introduction of closed-loop processes in the conditions of galvanic production and the production of high-quality and environmentally friendly coatings.

In connection with it the objective of this work is to study the utilization of iron-containing products of galvanic wastewater treatment into powder coatings in purpose to determine their mechanical, corrosion and electromagnetic shielding properties.

In order to the goal, the following tasks were set:

– investigate the physical and service properties of powder systems with ferrites in their composition;

- determine the corrosion resistance of the obtained materials;

- investigate the shielding properties of powder coatings modified with various ferrites from electroplating wastewater treatment.

MATERIALS AND EXPERIMENTAL TECHNIQUES

Composition of powder coatings

The carboxyl-containing polyester resin “Crylcoat 2618-3” produced by "Allnex" was used as a film-forming substance. For carboxyl-containing polyester resin a structure-forming hardener it is also required. There is the hydroxoalkylamide (HAA) Primid XL-552 produced by the company "EMS-Griltech" was used. To ensure the metal surface coverage, the white pigment in the form of titanium dioxide (TiO2) was chosen. A powder coating system that contains the barium sulfate, which is a traditional filler for powder coating materials, was used as a test composition (Table 1).

Table 1. Compositions of paint systems

Sample number	Composition (%)				
	Crylcoat 2618-3	HAA	TiO_2	$BaSO_4$	Ferrite powder
1	57	3	10	40	–
2 ÷ 10				25	15

There are nine samples of powder paint coatings modified with ferrite waste from water treatment were studied. According to the of X-ray diffraction results these wastes contain following phases see Table2.

Table 2. Phase composition of samples

Sample No	Phase
2	$NiFe_2O_4$
3	$ZnFe_2O_4$
4	$CuFe_2O_4$
5	$Ni_{0.5}Zn_{0.5}Fe_2O_4$
6	$Ni_{0.5}Cu_{0.5}Fe_2O_4$
7	$Zn_{0.5}Cu_{0.5}Fe_2O_4$
8	$Zn_{0.5}Mn_{0.5}Fe_2O_4$
9	$Ni_{0.5}Zn_{0.5}Al_{0.15}Fe_{1.85}O_4$
10	$CrFe_2O_4$

Phase analysis of powders of dried water treatment sediments was carried out by X-ray diffractometry in a step-by-step mode with Cu-Kα radiation on an Ultima IV setup (Rigaku, Japan). Shooting was carried out in the 2θ range 6 ÷ 65° with the step of 0.05° and exposure time at a point of 2 s. In the coating samples of powder systems No. 2 ÷ 10, iron-containing waste was used as a partial replacement of the barium sulphate filler. The properties of the obtained coatings were compared with the of standard coating No. 1.

Experimental techniques

The study of the effect of iron-containing waste on the corrosion resistance of the coatings based on powder paints was carried out in the following sequence. Powder paints of various compositions was applied to the plates (size 30x15 mm) prepared from 1.0038 / S235JR steel. The powder coating was applied using an electrostatic method according to ISO 1514:2016 [13] and hardened in a polymerization oven at a temperature of 180 ºC for 10 minutes. The reverse impact resistance was determined according to ISO 6272-2:2011 [14]. The bending strength testing was performed according to ISO 1519:2011 [15]. The corrosion resistance of decorative and protective powder coating systems was performed in a salt fog chamber with condensation of a 5% aqueous solution of sodium chloride (NaCl) on the surface of the samples for 480 hours at a temperature of 35 ºC in accordance with ISO 9227:2017 [16] "Corrosion tests in artificial atmospheres. Salt fog test”. The average delamination of the coating and the corrosion propagation was determined according to the technique of ISO 12944-6:2018 [17]. The classification of the studied coating compositions into the categories of atmospheric corrosion aggressiveness was carried out in accordance with ISO 12944-2:2017 [18], as well as their durability in accordance with ISO 12944-1:2017 [19].

The shielding properties of powder coatings was carried out on the plates with dimensions 30x15 mm and an average coating thickness of

180 microns. The effectiveness of the powder coatings shielding against electromagnetic emission was investigated by the level of attenuation of high-frequency radiation at a frequency of 3.3 GHz in comparison with the power level of the reflected electromagnetic wave in front of the control sample. Studies of high-frequency losses of the samples were carried out using two hollow copper cylindrical resonators, on the resonance mode H_{011}, with the replacement of one of the end caps with a sample (Fig. 1).

Fig. 1. An empty copper cylindrical resonator with a resonant frequency of 3.3 GH with cover, coaxial input and output of high-frequency radiation.

Measurements were made using a Keysight P9375A [13] microwave circuit analyzer using the two-port method, where the microwave power from port #1 of the analyzer passing through the resonator into port #2. Thus, the amplitude-frequency characteristics (frequency response) of the insertion losses IL(F) of the were obtained. At the same time, two parameters were determined, specifically the maximum amplitude of the resonant peak IL(F0) which corresponds to the resonant frequency F0 and the loaded Q factor of the resonator Ql, which was calculated from the frequency response. In different places of the plate, 5 measurements were made with subsequent averaging of the test results. The heterogeneity of plate parameters was 2 ÷ 3%.

Unloaded q-factor of resonators with copper covers $Q_{0=}$ 29806 for the resonant frequency of 3.3 GHz. The Q-factor of the sample was calculated as inversely proportional to the microwave losses in it. Losses in the control sample with paint without ferrite additives were taken as $P_0 = 1$. Losses in other samples of coatings with the addition of ferritic waste were calculated relative to this reference sample P_n/P_0.

RESULTS AND DISCUSSION

Corrosion and operational properties of coatings

The results of studies of the powder materials modified with iron-containing waste operational characteristics are shown in Table 3 and Figures 2-4. According to experimental data, the introduction of all types of ferrites obtained from water treatment waste into the composition of powder paints has a different effect on the properties of powder coatings. Thus, the control composition of the powder coating with a filler in the form of barium sulphate ensures the strength reverse at the level of 30 sm/kg (Table 3), and the bending strength which corresponds to the diameter of the bending shaft of 10 mm (Fig. 2). The coating is characterized by peeling at the level of 7.5 mm after 480h soaking in the salt fog. The average width of metal corrosion is 5.5 mm. The category of corrosion resistance of the coating corresponds to class C3 with an average durability class (M) from 7 to 15 years.

Table 3. Strength to reverse impact results of powder paints modified with iron-containing waste

Sample No	Filler	Strength to reverse impact, kg/sm
1	$BaSO_4$	30
2	$BaSO_4$; $NiFe_2O_4$	40
3	$BaSO_4$; $ZnFe_2O_4$	20
4	$BaSO_4$; $CuFe_2O_4$	25
5	$BaSO_4$; $Ni_{0.5}Zn_{0.5}Fe_2O_4$	45
6	$BaSO_4$; $Ni_{0.5}Cu_{0.5}Fe_2O_4$	30
7	$BaSO_4$; $Zn_{0.5}Cu_{0.5}Fe_2O_4$	27,5
8	$BaSO_4$; $Zn_{0.5}Mn_{0.5}Fe_2O_4$	35
9	$BaSO_4$; $Ni_{0.5}Zn_{0.5}Al_{0.15}Fe_{1.85}O_4$	5
10	$BaSO_4$; $CrFe_2O_4$	5

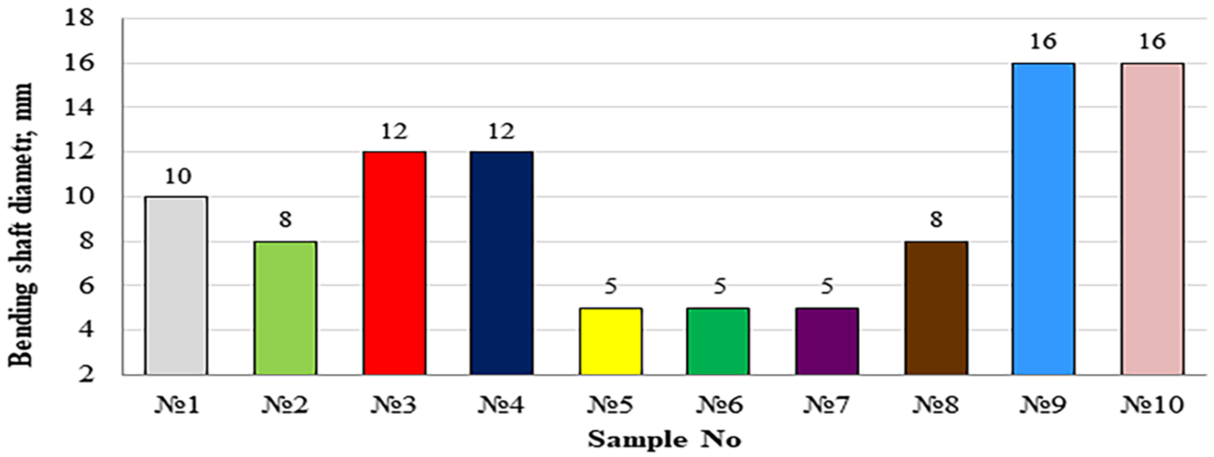

Fig. 2. Bending strength of the coatings.

The partial replacement of barium sulfate with iron-containing waste of phase composition $Zn_{0.5}Mn_{0.5}Fe_2O_4$ there both strength and corrosion resistance of the investigated coating is increasing. The $Zn_{0.5}Mn_{0.5}Fe_2O_4$ as filler contributes to the increase of strength to a reverse impact up to 35 cm/kg and bending strength to 8 mm in comparison with the control composition (Table 3, Fig. 2). At the same time, the average width of metal corrosion decreases by 29% from 5.5 to 3.9 mm. (Fig. 4) This may be due to the creation of a passive film on the surface of the steel, due to the introduction of the specified waste that is slowing down the anodic process of electrochemical corrosion. Corrosion category class corresponds to C4 (high) with the provision of an average durability class (M) from 7 to 15 years.

Filler in the form of $NiFe_2O_4$ powder also contributes to the improvement of coating properties. The $NiFe_2O_4$ contributes to the increase of strength to a reverse impact up to 40 kg/cm and bending strength to 8 mm compared to the control composition. It should be noted that the average coating delamination width is also reduced by 12% and is 6.6 mm. The average metal corrosion width is 3.7 mm, which is in 33% less compared to the control composition. The corrosion category corresponds to class C4 (high) with the provision of an average durability class (M) from 7 to 15 years.

A similar result is also demonstrated $Ni_{0.5}Zn_{0.5}Fe_2O_4$, in the composition of the powder pain which also contributes to increase of strength to reverse impact up to 45 kg/cm and the bending strength to 5 mm. At the same time, there is a decrease in the width of the coating delamination to 6.5 mm, and the width of the expansion of metal corrosion to 3.25 mm compared to the control composition was observed. The category of corrosion resistance corresponds to the class of sample No2.

Introduction of $CuFe_2O_4$ powder to the paint is effective only to increase corrosion resistance. Also, it leads to a 15% reduction in strength to reverse impact (25 cm/kg Table 2), and bending strength corresponding to a bending shaft diameter of 12 mm (Fig. 2). However, the use of $CuFe_2O_4$ in the composition contributes to the reduction of the average width of peeling of the coating from 7.5 to 3.75 mm, as well as the average width of the expansion of metal corrosion from 5.5 to 2 mm compared to the control composition. The category of corrosion resistance corresponds to class C4 (high) provided a high class of durability (H) from 15 to 25 years.

A similar result is observed when $ZnFe_2O_4$ powder is introduced into the paint – the strength of the coating decrease (Table 3, Fig. 2) and the average delamination of the coating increases compared to the control composition from 7.5 to 9.8 mm (Fig. 3), but at the same time the average corrosion width also decreases by 37% up to 3.5 mm (Fig. 4). Corrosion resistance corresponds to the C4 class (high) with an average durability class (M) from 7 to 15 years.

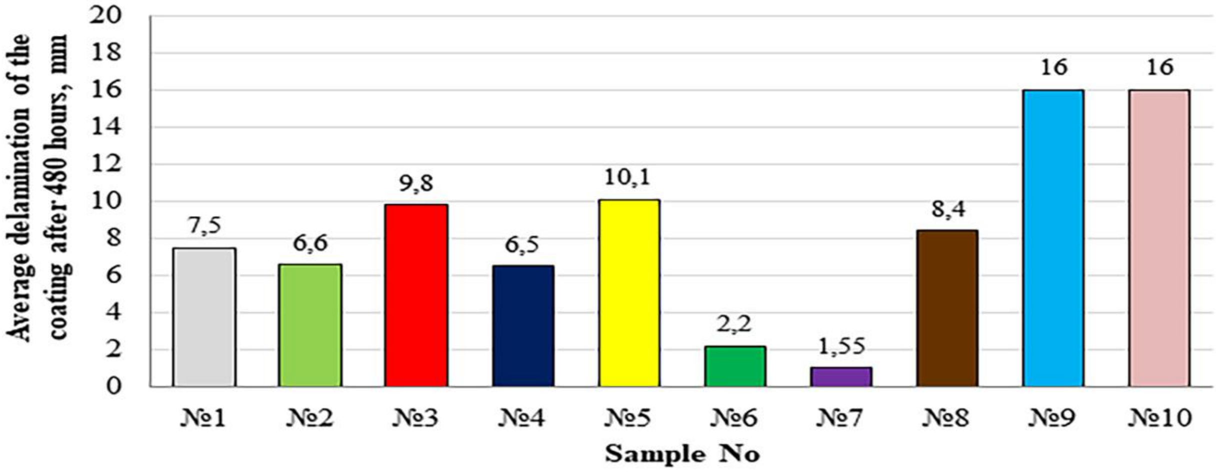

Fig. 3. Average coatings delamination.

It should be noted that the fillers in the form of $Ni_{0.5}Zn_{0.5}Al_{0.15}Fe_{1.85}O_4$ and $CrFe_2O_4$ is not effective, in connection a significant deterioration of the coating mechanical characteristics (Table 3, Fig. 2), a decrease in the corrosion resistance of the coating due to 100% peeling of the from the surface of the substrate (Fig. 3), as well as an increase in metal corrosion up to 12 and 14.5 mm (Fig. 4), respectively, compared to the control composition. The category of corrosion resistance of the coating corresponds to class C1 (very low).

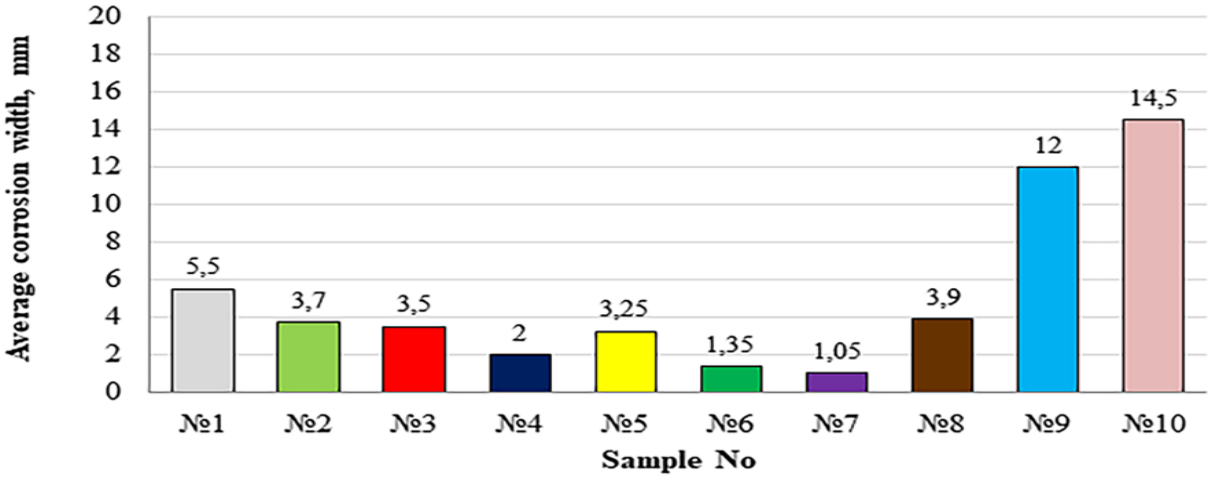

Fig. 4. Average corrosion width

Thus, one of the most effective ways to increase the corrosion resistance of the powder coatings is the use of fillers in the form of $Ni_{0.5}Cu_{0.5}Fe_2O_4$ and $Zn_{0.5}Cu_{0.5}Fe_2O_4$. When $Ni_{0.5}Cu_{0.5}Fe_2O_4$ is introduced the average width of coating delamination decreased by 71% from 7.5 to 2.2 mm compared to the control composition, and the average width of expansion of metal corrosion by 75% - from 5.5 to 1.35 mm. When $Zn_{0.5}Cu_{0.5}Fe_2O_4$ was used as a filler the average coating delamination width decreased by 79% up to 1.55 mm, and the average metal corrosion expansion width decreased by 80% up to 1.05 mm. The category of corrosion resistance of coatings corresponds to class C4 (high) with a high durability class (H) from 15 to 25 years. At the same time, the introduction of $Ni_{0.5}Cu_{0.5}Fe_2O_4$ and $Zn_{0.5}Cu_{0.5}Fe_2O_4$ the mechanical characteristics of the coatings do not deteriorate at the level of the control composition.

Shielding to electromagnetic emission properties of coatings

Table 4 shows the results of the obtained materials electromagnetic emission shielding capabilities for an external field with a frequency of 3.3 GHz This frequency is intermediate between the wireless frequencies of the 4G and 5G standards and allows to evaluate the shielding effectiveness of both of them.

The results of the experiments (Table 4) show that the samples containing $CuFe_2O_4$, $Ni_{0.5}Cu_{0.5}Fe_2O_4$ and $Zn_{0.5}Cu_{0.5}Fe_2O_4$ compounds demonstrates the largest shielding coefficients. Their common feature is the presence of copper in the filler of matrix. It increases the specific conductivity of the material which is dominant for shielding electromagnetic emission of ultra-high and higher frequencies.

The shielding coefficients of the coatings are relatively small, from 2.4 to 3.2. But taking into account the small thickness of the layer, such a result can be considered satisfactory and promising. Regarding the contribution of reflection and absorption of electromagnetic waves into the overall shielding coefficient, the obtained result indicates the predominant absorption of electromagnetic energy.

Table 4. The efficiency of electromagnetic emission shielding

Sample No	Losses at the top of the resonance curve, IL (dB)	Q-factor under loading, Q_l	The quality of the resonator, Q_O	Quality of the sample, Q_{sample}	Normalized losses in samples, P_n/P_0
1	-26,433	7591,7	7971,8	9202,4	1
2	-28,666	5859,1	6083,4	6774,7	1,35
3	-29,184	5591,1	5792,3	6415,6	1,43
4	-33,226	3499,3	3577,3	3805,7	2,41
5	-32,145	3924,5	4023,9	4315,1	2,13
6	-34,281	3074,7	3135,2	3309,3	2,78
7	-35,634	2700,3	2745,6	2878,2	3,19
8	-32,999	3573,6	3655,4	3894,2	2,36
9	-30,468	4725,4	4871,3	5304,8	1,73
10	-31,732	4154,6	4265,0	4593,7	2,00
Cu	-15,981	25072	29806,3	59612,6	0,15

The substrate of the samples consists of iron, which has high reflection coefficients inherent in all metals and alloys (up to 0.7 ÷ 0.9, depending on the composition and texture of the surface). Fillers of the ferrite type provide in advance a significantly lower specific conductivity of the surface. The impedance of the surface layer is much higher than that of metals and is close to the indicator of the medium of propagation of electromagnetic waves - air (377 Ohms). Based on the fundamental relations of electrodynamics of continuous media, this reduces the reflection coefficients of electromagnetic waves. In this case, two mechanisms of electromagnetic energy losses are the most important - conduction losses and losses at the transition of the "composite - metal" boundary.

The obtained result shows the possibility of increasing the efficiency of composites by adjusting the content of the protective filler in the matrix. At certain concentrations of the filler, the threshold of the percolation effect is reached - a sharp increase in the conductivity of the material due to the formation of elongated conductive structures. This automatically leads to a significant increase in shielding coefficients. But at the same time, the reflection coefficient can increase.

The determination of the desired

concentration of the filler is based on the prediction of the acceptable ratio of the reflection and absorption coefficients of electromagnetic waves.

It is known that the protective properties of composite materials depend on the size of the filler particles, as well as on their shape.

Therefore, it is advisable to investigate the effect on the protective properties of the obtained compositions of average sizes and morphology of filler particles from different components. Another possibility is to create a number of compositions with different electrical and magnetic properties. Increasing the electrical and magnetic conductivity of the multilayer structure from the surface layer to the surface of the coating will increase the overall shielding factor with minimal surface layer reflectance.

CONCLUSIONS

As a result of the research, it was found that the use of iron-containing waste in general contributes to the increase of both mechanical characteristics and corrosion resistance of the coatings. The effectiveness of their use also depends on the structural composition.

In order to increase the strength indicators of polymer systems, the most effective among the studied samples is the introduction of fillers in the form of $Ni_{0.5}$ $Zn_{0.5}Fe_2O_4$, $NiFe_2O_4$, which contribute to an increase in reverse impact strength by 20 and 25% and bending strength up to 50%.

In terms of corrosion resistance, it is the most effective introduction of iron-containing $Ni_{0.5}Cu_{0.5}Fe_2O_4$, and $Zn_{0.5}Cu_{0.}$ fillers into the powder systems composition, which contribute to the reduction of the peeling width of the coating by 65 and 79%, as well as the width of metal corrosion by 75 and 80%, respectively, compared to the control composition. Corrosion resistance class of the specified paint systems responds C4 (high) when providing the appropriate durability class (H) from 15 to 25 years.

It has been established that paint coatings modified with iron-containing fillers have high shielding properties for electromagnetic emission at a resonant frequency of 3.3 GHz. The most effective is the introduction of $Zn_{0.5}Cu_{0.5}Fe_2O_4$ (15% by mass). in the coating composition; this coating improves the shielding properties by more than 3 times compared to the control sample.

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Корозійні та електромагнітні захисні властивості порошкових лакофарбових покриттів отриманих з гальванічних відходів

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Анотація Робота присвячена вивченню властивостей порошкових лакофарбових покриттів модифікованих наповнювачами, які отримують із переробки відпрацьованих технологічних розчинів гальванічних виробництв методом феритизації. У роботі досліджено фізико-механічні та захисні властивості порошкових систем із введенням до їх складу залізовмісних продуктів. Показано, що використання залізовмісних відходів в цілому сприяє підвищенню як механічних характеристик, так і корозійної стійкості покриттів. Визначено, що введення до складу $Ni_{0,5}Cu_{0,5}Fe_2O_4$ та $Zn_{0,5}Cu_{0,5}Fe_2O_4$ дозволяє значно підвищити їх механічні та екрануючі властивості.

Ключові слова: порошкова фарба, покриття, корозія, гальванічні відходи, феритизація, електромагнітне екранування, ферити.