SYNTHESIS OF COPOLYMERS FOR PROTECTIVE COATINGS

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ABSTRACT

Copolymers were obtained in this work and the methodology for their synthesis was worked out. Various fillers were selected for the polymer coating. Resulting copolymers have good adhesion required for composite protective coatings. An experiment was conducted to determine the corrosion resistance of metals coated with copolymers when exposed to aggressive environments, as well as to determine the hardness and thickness of the polymer coatings obtained. It was found that the polymer coating filled with bronze powder, despite the small thickness of 43.4 µm, has the best adhesion and corrosion properties, as well as having the highest hardness values of 80.5 HB. Such physical and mechanical properties of polymer coatings allow them to be used as protective coatings for metal products working under the influence of aggressive media.

Keywords: copolymer synthesis, copolymer, polymer, polymer coating, protective coating.

INTRODUCTION

Corrosion prevention is one of the most important scientific, economic, social, and environmental problems. This is due to the fact that technological progress in almost all industries is held back by a number of unresolved problems of corrosion prevention. This is especially true for countries with rich metal reserves, since industry increasingly uses not only high-strength materials, but also particularly aggressive environments, as well as high pressures and temperatures. This situation has led to a significant increase in loss rates due to dangerous forms of corrosion, such as pitting, intergranular corrosion, and corrosion cracking. In this regard, there is a need to find effective ways to develop new formulations of polymer protective coatings [1, 2].

Requirements for polymer coatings are quite strict. First, they shall not only cover the metal base, but also bond with it at the molecular level, which requires a very high adhesion to the surface. Secondly, there should be resistance to various deformations, which prevents the destruction of the coating from mechanical influences. Thirdly, the properties should not be lost even after prolonged exposure to solvents, alkalis, water, various chemicals, ultraviolet radiation, and high temperatures. If all these conditions are met, the area of application of coatings is greatly expanded [3 - 5].

Most often, polymer coatings with good adhesion to the protected surfaces are easily destroyed by chemical and mechanical effects of aggressive media. Only epoxy resins and some other polymers managed to combine high adhesion to metals with sufficient chemical resistance [6 - 8].

The main disadvantage is that polymer coatings have a high cost based on material quantities and special equipment application. However, along with this, the polymer layer does not require updating or touch-up and is applied for life, so the payback period of such coatings is very fast [5, 8].

Another disadvantage of such coatings is low strength characteristics and deterioration of performance properties at temperatures above 80°C. Therefore, protective coatings based on polyorganosiloxane binders or modified with polyorganosiloxanes are of great practical interest [9 - 13]. As a solution to this problem, the authors in present the results of created protective polymer coating, which has high strength properties, containing wastes of electroplating production [14]. The proposed coating was created to protect metal and concrete surfaces of various building structures from unwanted external and natural influences.

The authors of paper considered the problem of obtaining a new flame-retardant protective polyurethane coating filled with galvanic sludge and modified with polymethylphenylsiloxane [15]. Modification with polymethylphenylsiloxane improves thermal, waterproofing, physical and mechanical properties of polymer coatings. The obtained polymer protective coatings showed that the modification with polymethylphenylsiloxane and the use of galvanic sludge as a flame-retardant filler increases fire resistance, and also solves the problem of environmentally safe use of sludge waste.

In one of the papers, the author highlights the results of research on the selection of quantitative compositions for obtaining protective polymer coatings with improved properties of adhesion, water repellency and durability [16]. Conducted studies justify the fact that the use of the obtained polymeric compositions makes it possible to significantly improve the operational and water repellent properties of coatings, and this makes it possible to widely apply them to protect metal surfaces of different types and shapes.

Pavlov et al. first obtained zinc polymer coatings by joint electrodeposition of zinc cathodes and amine-containing epoxyamine adducts modified with isocyanates [17]. Metallopolymers are heterogeneous systems consisting of a polymer medium and a highly dispersed metallic phase, where chemisorption interaction is achieved at the interfaces. Therefore, metallic polymers have valuable properties such as elasticity, high adhesion, electrical and thermal conductivity inherent in both polymer and metal [18]. Especially promising is the use of metallic polymers as corrosion-resistant coatings, where the protective properties of the polymer coating are complemented by the protective or inhibitory effect of the metal [19 - 26].

The authors of paper proposed a method for obtaining grafted polymer coating to impart water-repellent properties to metal surfaces. In this technology, the metal surface is pretreated to obtain hydroxyl groups on it and then treated with a solution of polymerization initiator with triethylamine in a solvent medium [27]. Then it is modified in a solution containing a monomer by its graft polymerization on the surface under a catalytic complex consisting of copper (I) bromide and organic ligand, for which bipyridine, dinonylbipyridine or pentamethyldiethylantriamine are used. In this method, metal surface modification is achieved by grafting hydrophobic polymer chains onto metal surfaces through surface-initiated polymerization by transferring atoms [28]. This process of protective coating leads to the formation of a polymer film on the metal surface with increased water repellency [29 - 44].

Thus, the authors of this article, having done a huge literature and patent analysis, propose a technology for creating a polymer coating with corrosion resistance. The scientific novelty of proposed research work consists in the development of technology for obtaining protective coatings of new formulation based on copolymers with corrosion properties, as well as the possibility of using them to protect against the effects of aggressive media of metals and nonmetals.

EXPERIMENTAL

In order to fulfill the problem, it is necessary to perform the following tasks:

1) development of technology for obtaining copolymers;

2) development of a comprehensive technology for obtaining protective polymer coating based on copolymers;

3) selection of a formula for a new polymer coating to protect metallic and non-metallic (concrete) materials against aggressive media.

During the studies, copolymers based on vinyl butyl ether (VBE), maleic anhydride (MA), and methyl methacrylate (MMA) were obtained (Fig. 1). Various protective compositions have been developed based on obtained copolymer.

A three-neck flask was filled with methyl methacrylate and vinyl butyl ether and loaded with maleic anhydride (30, 20, 20 g, respectively) (copolymer 1). After dissolution of maleic anhydride, 100 g of toluene was loaded. The heating temperature of monomer mixture is about 60°C. Then the polymerization initiator dinitrilazoisobutyric acid 0.2 g dissolved in 10 ml of solvent toluene in a beaker was added. Copolymerization occurs when heated slowly to a temperature of 70 - 80°C. The reaction mixture was held at 80°C for 1 - 2 hours. To mature the copolymer, the reaction mixture was held for 24 hours (1 day). After the copolymer had matured, the residual monomers were distilled. To find the dry residue, the mixture was weighed on analytical scales and the solvent was separated in a desiccator at 110°C (Fig. 2). Then the copolymer was re-weighed, and the product yield was calculated. The product yield was 57.7 %.

Similarly, we synthesized copolymer 2 and copolymer 3 with different ratios of initial components. The obtained copolymers were mixed with fillers to change their technological and operational properties in a targeted way. Thus, bronze powder (coating 1), titanium oxide (coating 2), and microsilica (coating 3) in the ratio 2:1 was selected as fillers. The obtained polymer coatings were applied to the metal plates. Table 1 shows the composition of developed protective polymer coatings and technological operations to obtain them.

To determine the behavior of obtained polymer coatings and calculate their corrosion resistance, the coated and uncoated plates were placed in different aggressive media. The composition of aggressive media is as follows: H_2SO_4 acid - 10 %; KOH alkali - 10 %; NaCl salt - 10 %. The plates were held in aggressive media for 24 hours (Figs. 2 - 4). After that, the corrosion resistance of polymer coatings obtained using the new formulation was determined.

Next, an analysis was performed on the adhesion of the coating to the metal base using the scratch grid method. The scratch grid method is an inspection method in which four to six parallel lines are drawn on the surface of the coating to be inspected with a steel point at 2 - 3 mm intervals from each other and four to six parallel lines at right angles to the first line. There shall be no flaking on the surface of the test coating.

Hardness was determined using a portable combined MET-UD hardness tester on the Brinell scale. Also the thickness of coatings according to GOST R52146-2003 “Polymer-coated rolled products” with the use of electronic micrometer was determined.

The characteristic of coating morphology was studied using a Zeiss EVO18 scanning electron microscope.

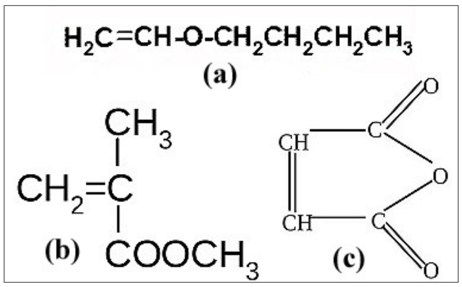
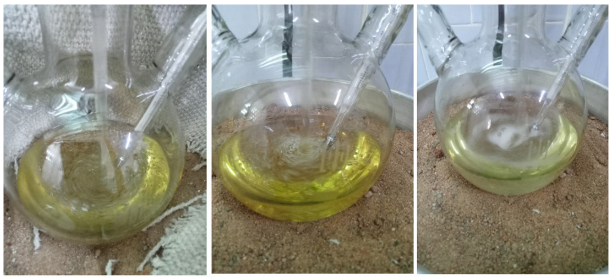


Fig. 1. Formulas of reagents used: (a) Vinyl butyl ether; (b) Methyl methacrylate; (c) Maleic anhydride.

Table 1. Composition of new protective polymer coatings and technological operations to obtain them.

Composition	Ratio/ amount, g	Initiator AIBN, g	Heating temperature, °C	Copolymerization temperature, °C	Holding time, h	Copolymer aging time, h	Drying temperature, °C	Product yield, %
Coating 1								
Copolymer 1, bronze powder	3:1	0.2	60	70 - 80	1 - 2	24	100	43.7
Coating 2								
Copolymer 2, titanium oxide	3:3:1	0.2	60	80 - 90	2 - 3	24	100	74.4
Coating 3								
Copolymer 3, myrosilica	3:3:1	0.2	60	60 - 70	2 - 3	24	100	36.7



(a) (b) (c)

Fig. 2. Copolymerization process: (a) Mixing of monomers; (b) Addition of the initiator; (c) Co-polymerization reaction.

Fig. 3. Samples with polymer coating No. 1 after exposure aggressive media: (a) Acid; (b) Alkali; (c) Salt.

Fig. 4. Samples with polymer coating No. 2 after exposure aggressive media.

RESULTS AND DISCUSSION

The corrosion resistance of the polymer coatings obtained with the new formulation is shown in Table 2.

Next, an analysis was performed on the adhesion of the coating to the metal base using the scratch grid method. The test results are shown in Table 3.

Fig. 3 shows the sample No. 1 (10 % NaCl): the coating is even, slightly rough, without bubbles, bloat, delamination, tears, cracks, no runs, and drips. It adheres tightly to the base. Sample No. 2 (10 % H_2SO_4): the coating is even, slightly rough. It adheres tightly to the base, without bulk and bloat, bubbles, cracks, but there is a small area with separations. Sample No. 3 (10 % CON): coating is even, slightly rough, without bubbles, bloat, peels, tears, cracks, no drips, or stains. It adheres tightly to the base.

Fig. 4 shows sample No. 1 (10 % NaCl): the coating is dense, but there are leaks, as well as areas of rust appear, i.e. the coating does not protect the metal base and corrosion processes occurred. There is a delamination of the coating. Sample No. 2 (10 % H_2SO_4): the coating is cracked after interaction with an aggressive environment, poorly adhered to the metal base. Sample No. 3 (10 % KOH): there are some runs, some roughness, but the coating is tightly adhered to the base.

Fig. 5 shows sample No. 1 (10 % NaCl: strong corrosion processes occurred while interacting with an aggressive medium. Almost the entire surface of the sample is corroded, and corrosion spots are visible, but the coating is not cracked. Sample No. 2 (10 % H_2SO_4): the coating is tight, but there is corrosion in some areas. The coating is not cracked, no blows, bubbles. Sample No. 3 (10 % KON): the coating is denser and more even, but there is corrosion in some areas. The coating is not cracked, no blows or bubbles.

The characteristics of the coating morphology were also determined, and the surface of the coatings is shown in Fig. 6. Surface studies of polymer coatings obtained were carried out with the scanning electron microscope (Fig. 6). Fig. 6(a) shows that the surface of polymer

Table 2. Corrosion resistance values of polymer coatings.

Coating composition	Acid	Alkali	Salt
Copolymer 1 + bronze powder	99.68	99.93	100.97
Copolymer 2 + titanium oxide	99.97	99.48	100
Copolymer 3 + myrosilica	99.42	99.56	100

Table 3. Adhesion test results.

Coating No. 1	Only the bronze coating remained intact during the test on all samples. The coating adheres tightly to the metal base, i.e. it has good adhesion
Coating No. 2	The coating on the samples is very fragile, but the coating on the samples that have been in an alkaline environment, does not crack or peel, tightly adhere to the metal base, unlike the coating that has been in acidic and neutral environments; this coating is strongly peeling and crumbling
Coating No. 3	Coatings that have been in alkaline and acidic environments peel and crumble during the test, especially at the corrosion points

Table 4. Coating hardness values.

Name	Sample		
Coating types	polymer coating No. 1	polymer coating No. 2	polymer coating No. 3
Hardness HB	80.5	49.7	44.3
Coating thickness*, μm	43.4	251.0	144.1

**Average coating thickness (after exposure to various aggressive media)*

Fig. 5. Samples with polymer coating No. 3 after exposure aggressive media.

coating No. 1 (filler - bronze powder) after exposure to aggressive media has not undergone critical changes. Thus, there are minor microcracks on the coating surface, as well as local porosity, while only the coating is damaged, but its integrity is preserved. Corrosion processes have not affected the metal base.

Polymer coating No. 2 (filler - titanium oxide) has cracks and porosity on the surface after exposure to aggressive media. Likewise, as on the surface of coating No. 1, the pores damaged only the coating and the corrosive effect did not affect the metal base (Fig. 6(b)).

That cannot be said about polymer coating No. 3 (filler - microsilica). Thus, Fig. 6c-d shows that after the corrosive action in some places the integrity of polymer coating is broken, up to the metal.

The results of hardness and thickness measurements of polymer coatings are given in Table 4.

Table 4 shows that the bronze coating has the highest hardness, followed by coating No. 2. And the polymer coating with microsilica 2 and 3 has the lowest hardness. By the way, the hardness was measured in coated samples after exposure to an aggressive environment. Apparently, the low value of hardness is influenced by the composition of the coating, as well as the aggressive environment. As for the influence of aggressive media, it corroded not only the coating, but also the top layer of base metal, while lowering the hardness of coated samples and the metal itself. This fact affects the cost of base metal, reduces its service life, as well as the replacement frequency of the product, and therefore increases energy and resource costs.

As for the thickness of coatings, coating No. 2 has the highest value, but this does not affect the increase in hardness value, since this coating after exposure to aggressive environments has reduced the hardness of the base material. Polymer coating No. 3 after the corrosive media series also has a high thickness value due to the initial decrease in the hardness of base material. And coating No. 1 with bronze powder has a small thickness, indicating good adhesion to the base metal, thereby not reducing the hardness of the metal base.

Thus, polymer coating No. 1 with bronze powder, which has a high value of hardness and small thickness of coating in spite of aggressive environment effects, is appropriate for use in production. This leads to a

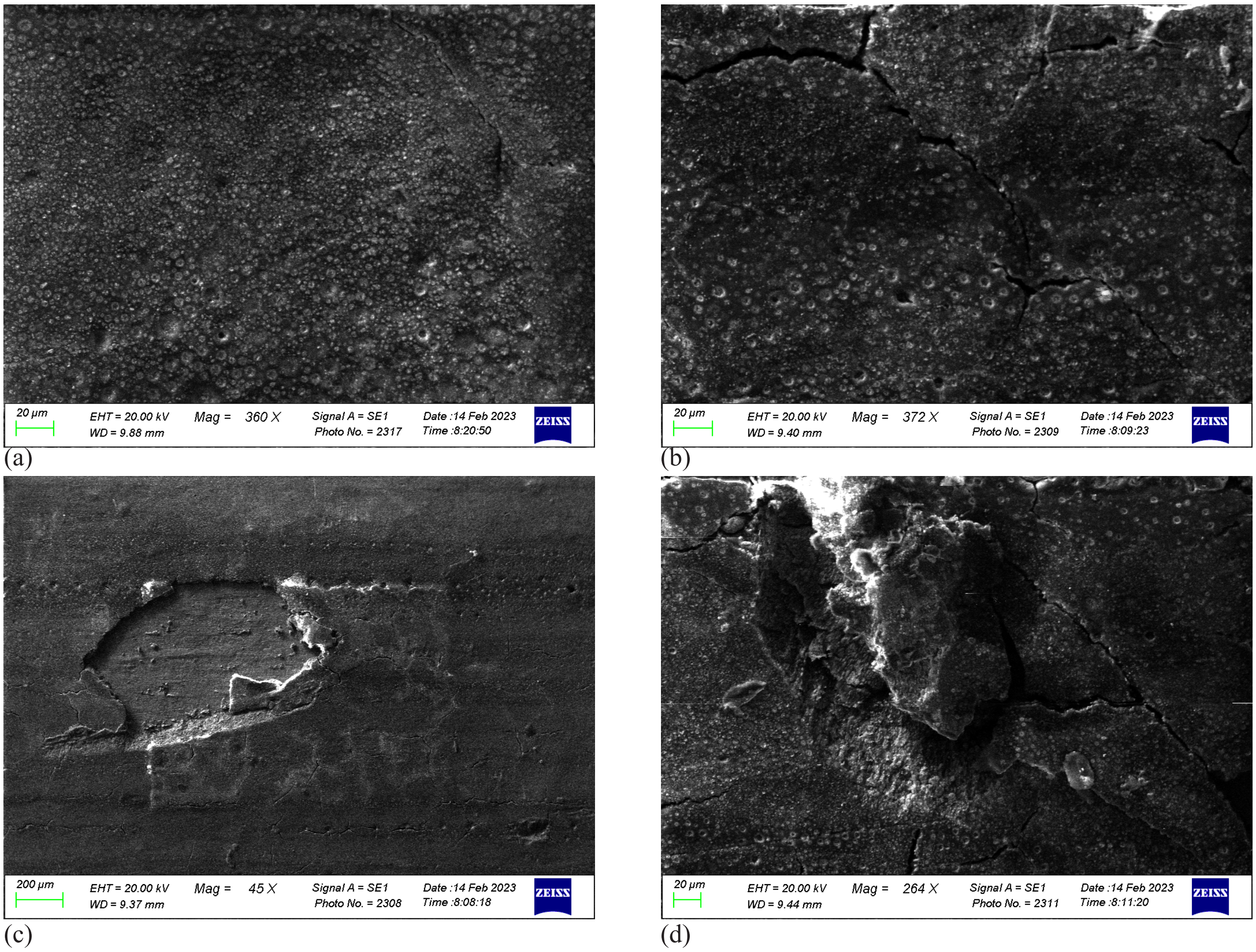


Fig. 6. Coating surface: (a) No1, (b) No2, (c, d) No3.

reduction in specific quantity of metal per structure and the cost of additional expenses (paints, polymers, fillers, repair of structures, etc.).

The new formula of developed polymer protective coatings favorably distinguishes the proposed composition from the existing ones. Thus, the technology was developed for protective coatings of new composition used for metallic and nonmetallic products operating in aggressive environments.

CONCLUSIONS

Thus, the ternary copolymer has the ability of complex substrate formation on metallic and nonmetallic bases due to the anhydride grouping. Obtained polymer coatings have improved adhesion resistance, i.e. they have similarity to different base materials, which allows the coatings to work under aggressive conditions. The use of such polymer coatings is advisable in mechanical engineering, construction, metallurgical production, as it leads to a reduction in specific quantity of metal per structure and the cost of additional expenses (paints, polymers, fillers, repair of structures, etc.).

The described new protective polymer coatings guarantee reliable, long-term protection and high decorative properties throughout the life of equipment, metal structures and non-metallic building products, which contributes to solving the problem of mechanical engineering, metallurgical industry to increase the operating life of products. Therefore, there is a need to find out effective measures, since currently in Kazakhstan there are no production facilities for the creation of polymer corrosion protection systems. Setting up such production will make it possible to obtain our own protective polymer coatings, save money on the purchase of polymer coatings in foreign countries, guarantees the expansion of the country's raw material base, preservation of metal stock, increased import substitution and thereby improve the competitiveness of Kazakhstan.

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