

EPOXY-POLYESTER COATINGS WITH ADDITIVES OF PTFE PARTICLES AND NANOPARTICLES OF SiO₂

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Abstract. Hydrophobic and super-hydrophobic coatings based on epoxy-polyester powder compositions modified with polytetrafluoroethylene (PTFE) particles and nanoparticles silicon dioxide were obtained. The degree of hydrophobicity of the coatings depended on the concentration of PTFE particles. Coatings containing optimal amounts of PTFE exhibited superhydrophobic properties with a high contact angle of wetting with water up to 161° and a low roll angle <3° while maintaining a high level of physical and mechanical properties of the coatings. The synergistic effect of PTFE's low surface energy and the formation of multilevel surface roughness led to the formation of a superhydrophobic coating. The stability of the superhydrophobic coating was investigated by assessing the change in the contact angle and roll angle after holding the coatings in distilled water and 3% NaCl solution.

Keywords: powder polymer compositions, fluoroplastic additives, contact angle, surface energy, roughness, hydrophobicity, mechanical properties

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1. Introduction

The creation of coatings with hydrophobic properties is an important scientific and practical task [1,2]. Imparting hydrophobic properties to the surface of paint and varnish coatings allows, on the one hand, to significantly reduce the rate of penetration of moisture and water-soluble corrosive agents to the substrate, and, on the other hand, to reduce dust and dirt retention, facilitate surface cleaning and increase their service life. The sliding of the liquid flow is facilitated near the hydrophobic surface, which is an effective means for separating the aqueous and oily phases [3]. In addition, based on hydrophobic coatings, new technologies have been developed for non-biocidal protection of ships, hydraulic structures, and other objects from biofouling [4,5]. Due to the hydrophobization of the surface, it is also possible to create anti-corrosion [6] and anti-vandal coatings [7].

In practice, the creation of hydrophobic coatings can be solved by the following methods: modifying or processing the surface of the polymer coating; the use of non-polar film formers; physical and chemical modification of the polymer itself. In all cases, the most

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important and necessary condition is the low values of surface energy (<30 mJ/m²), which are possessed by organofluorine or organosilicon compounds [8]. In this case, polytetrafluoroethylene has the lowest surface energy [9]. However, many fluorine-containing materials are not directly used due to limitations associated with their solubility and low mechanical characteristics [10].

Superhydrophobic coatings are of particular interest. Superhydrophobic coatings are characterized by a high contact angle of water wetting larger than 150° and a low angle of inclination of the surface (within 5-10°) to the horizon, at which a drop of water rolls off the coating. In the case of obtaining superhydrophobic materials with a contact angle larger than 150°, it is necessary to use the combined effect of roughness and chemical structure of the surface. Superhydrophobic coatings are attracting a lot of attention due to their unique properties such as self-cleaning, protection against dirt adhesion, and corrosion resistance [11,12]. By imparting hydrophobic and superhydrophobic properties to the coating surface, it can solve the problem of not only corrosion of the inner surface of pipelines but also deposits on the surface of various paraffin compounds and an increase in the flow rate of transported energy carriers by changing the surface profile [13].

Special attention is paid to the use of superhydrophobic coatings to protect surfaces from icing. The effectiveness of using superhydrophobic surfaces in the creation of anti-icing coatings is determined by several factors: first, the water-repellent properties of such a surface, i.e. the minimum water content on the surface, which can crystallize; secondly, such surfaces are characterized by low adhesion of already formed ice. The latter leads to spontaneous removal of ice or frost under its own weight or wind load [14]. So, in our last publication, the effectiveness of using a superhydrophobic coating for protection against various types of icing has been shown [15].

There are a number of ways to achieve a superhydrophobic surface, such as template synthesis, surface etching followed by treatment with hydrophobic materials, chemical and electrochemical particle deposition, phase separation, controlled particle aggregation, etc. [1,8,16]. A widespread method for obtaining superhydrophobic coatings is based on the use of organic and inorganic fillers with a multimodal particle size distribution [5,17,18]. The particles used are nanoparticles of silicon oxide, zinc, carbon nanoparticles, etc.[18-22]. Such modified nanoparticles are introduced both into hydrophobic polymers [17,19] and into other polymer binders (epoxy, acrylate, polyurethane). Upon their application, textured highly hydrophobic coatings are formed with a contact angle of about 150° and larger [23-26]. An important aspect in the development of superhydrophobic coatings is the stability of the superhydrophobic layer. With prolonged contact with various polar and non-polar liquids, the superhydrophobic coating can be destroyed [20].

To date, most of the published work on the preparation of hydrophobic and superhydrophobic coatings dedicated liquid paint compositions. We have previously shown that the modification of epoxy-polyester powder composites with small additions of PTFE particles leads to the production of coatings with a wetting angle larger than 160° [27].

This work is devoted to the formation and study of stable epoxy-polyester coatings with low surface energy from powder compositions modified with PTFE and SiO₂ particles.

2. Materials and Methods

The object of the study was a commercial epoxy-polyester powder varnish composition manufactured by Ekolon (St. Petersburg, Russia), particles of polytetrafluoroethylene (PTFE) from OOO Fluralit synthesis (Russia) with a particle fraction up to 5 μm. The concentration of the PTFE particles in the powder composition varied from 0 to 2 wt%. To increase the stability of the superhydrophobic coating, 0.1 wt. % particles of amorphous hydrophobic silicon dioxide (SiO₂) with particles about 7 nm, from Evonic (Germany). The preparation of

the powder composition for obtaining hydrophobic and superhydrophobic coatings was carried out by dry mixing of an epoxy-polyester base with PTFE and SiO₂ particles in a planetary-type ball mill [28]. The resulting powder composition was applied to metal plates degreased with ethyl acetate by electrostatic spraying. The coating was obtained by heat treatment of the substrate with the applied layer of the powder at the temperature of 180°C for 20 minutes. The coating thickness was in the range of 70-90 microns. In order to assess the morphology of the coating surface, the coatings were obtained on an aluminum foil with a thickness of 50 μm.

The contact angle was determined on a DSA4 instrument from Kruss (Germany) by processing the video image of a drop on the coating surface using the Drop Shape Analysis System DSA30 software at the air temperature of +20°C. Distilled water and hexadecane were used as the working fluid. Determination of the static contact angle of wetting was carried out by the method of a lying drop. The analysis of the drop was carried out by the Young-Laplace method with a drop volume of 7 μl, dosed with a syringe with graduation of 0.3 μl. In this method, the complete contour of the droplet is estimated, and not only the interphase interactions determining the contour of the droplet, but the contribution of the weight of the droplet liquid is considered as well. The contact angle is defined as the slope of the tangent at the tangent point of the three phases. Five measurements were made at five locations on the surface of the coating and the average was reported. Along with the wetting angle, the roll-off angle was determined.

Scanning electron microscopy (SEM) was used to study the morphology of the coating surface using a Supra 55 VP microscope (Carl Zeiss, Germany). When examining the coating surface by the SEM method, in order to exclude the accumulation of surface charge during scanning and increase the contrast, a platinum layer was preliminarily deposited on the samples by cathodic sputtering. The secondary electron mode was used to study the coating surface. An ELCOMETER 224 digital surface profilometer (Elcometer Instruments Ltd., UK) was used to analyze the roughness of the coatings.

The impact strength of the coatings was determined in accordance with ISO 6272 using a U-2 instrument. Determination of adhesion was carried out by the cross-cut method according to ISO 2409. Tensile strength tests of the coatings were carried out according to ISO 1520 on an Eriksen press. Pencil hardness tests were carried out in accordance with ISO 15184-1998 [29].

The stability of the hydrophobic properties of the obtained coatings was carried out by fixing the change in the contact angle and rolling angle with liquids (water and hexadecane) after holding them in distilled water and 3% NaCl solution for 500 hours.

3. Result and Discussion

The contact angle of the surface of the formed epoxy-polyester coating is found to be 73°. In this case, the surface is smooth. With the introduction of PTFE into the epoxy-polyester powder composition, an increase in the contact angle with water and hexadecane is observed. So, the introduction of already 0.5 wt% PTFE into the epoxy-polyester powder composition leads to an increase in the contact angle with water from 73° to 120°. With the further increase in the PTFE concentration up to 2 wt%, the contact angle with water reaches 161° (Fig. 1). However, the increase in the PTFE concentration up to 2.5 wt% does not lead to a noticeable change in the contact angle. The oleophobicity of the coating, characterized by the contact angle of wetting with hexadecane, changes in a similar way. When modifying 2 wt% PTFE epoxy-polyester powder composition, the contact angle with hexadecane exceeds 90°, that is an oleophobic coating is formed (Fig. 1). It should be noted that the contact wetting

angles of coating at the interface between the two coating/substrate phases do not change upon the introduction of PTFE and remain about 73°, indicating that the coating surface adjacent to the substrate is depleted in PTFE particles.

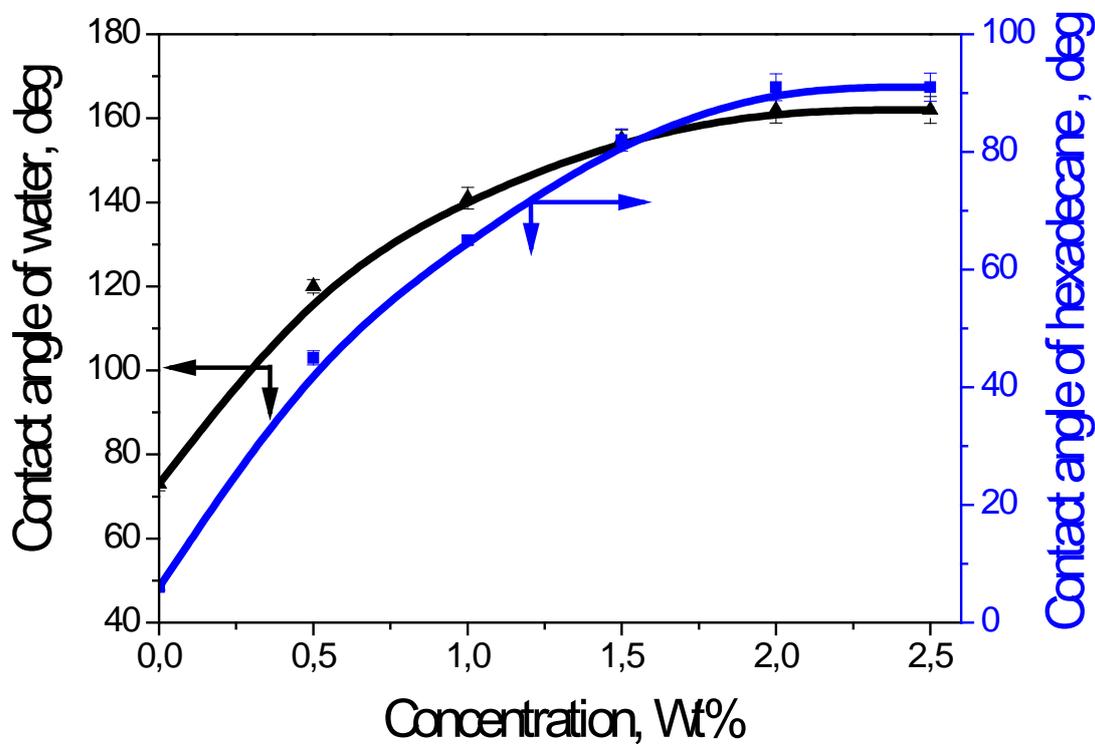


Fig. 1. The dependence of the contact angle with water and hexadecane on the PTFE content (wt.%)

Along with the contact angle, the other characteristic of the surface is the value of its surface energy, which is a consequence of the uncompensated cohesion energy at the solid/air interface. The surface energy of solids cannot be directly measured, but it can be calculated indirectly (by determining the contact angles). In this work, based on the values of the wetting contact angle of the coatings with water and hexadecane, we calculated the free surface energy by the extended Fowkes method [30]. The calculation was done according to the following equations:

$$\gamma_L \cdot (1 + \cos \theta) = 2 \cdot \sqrt{\gamma_S^d \cdot \gamma_L^d} + 2 \cdot \sqrt{\gamma_S^p \cdot \gamma_L^p}, \quad (1)$$

$$\gamma_S = \gamma_S^d + \gamma_S^p, \quad (2)$$

where $\gamma_L^d, \gamma_L^p, \gamma_L$, – dispersion, polar and total surface energies of the wetting liquid, respectively, mJ/m^2 ; $\gamma_S^d, \gamma_S^p, \gamma_S$ – dispersion, polar and total surface energies of the polymer surface, respectively, mJ/m^2 . The results of calculating the surface energy are shown in Table 1.

When PTFE is introduced into the epoxy-polyester powder composition, a significant decrease in the surface energy is observed. So, the surface energy decreases from 37.2 mJ/m^2 and reaches a value of 8.7 mJ/m^2 .

Table 1. Surface free energy (SE) parameters of epoxy-polyester coatings modified with PTFE particles

PTFE content, wt%	Contact angle, grad		Polar component of SE, mJ/m ²	Dispersion component of SE, mJ/m ²	Surface free energy, mJ/m ²
	water θ_w	hexadecane θ_h			
0	73	6	9.7	27.5	37.2
0.5	120	45	0.2	20.1	20.3
1.0	141	65	1.8	13.9	15.7
1.5	155	82	2.3	8.9	11.2
2.0	162	91	2.1	6.7	8.7
2.5	161	91	2.1	6.7	8.7

A sharp increase in the contact angle and a decrease in the surface energy upon the introduction of the PTFE particles into the epoxy-polyester powder composition is associated with the formation of a structured rough hydrophobic layer on the coating surface (see Fig. 2b). Initially, the epoxy-polyester coating is smooth without significant roughness (Fig. 2a). Apparently, upon mixing, the PTFE particles are adsorbed on the surface of the epoxy-polyester powder composition particles. In this case, the forces due to which the PTFE particles are retained on the surface of the epoxy-polyester composition, are of an electrostatic nature. When the particles of the powder composition coalesce in the process of film formation, the fluoroplastic particles will migrate onto the surface of the interface of the two coating/air phases due to the action of capillary pressure and the difference in the surface energies of the PTFE particles and the epoxy-polyester powder composition [31]. Further, the particles of the powder composition, due to the adsorbed PTFE layer, move in the process of film formation to the near-surface layers, forming a hydrophobic multilevel surface (Fig. 2 a). In this case, migration will be carried out faster than those particles of the powder composition, the surface of which is to a greater extent covered with PTFE particles. Apparently, at a concentration of PTFE particles of 2.0 wt%, the limiting degree of coverage of the particles surface of the powder composition is achieved, and a further increase in the PTFE content up to 2.5% does not lead to a change in the contact wetting angles with water and hexadecane. In the process of film formation, a multi-level surface of the coating is formed, consisting of a macro-level – incompletely coalescing particles of the powder composition, a micro-level – PTFE particles, and a nano-level formed by SiO₂ nanoparticles (Fig. 2). The reason for such high contact angles upon the introduction of PTFE is not only the surface chemistry but also the formation of a certain multilevel structure. It can be assumed from all the above that the surface of the epoxy-polyester coating is enriched in fluoroplastic since there is a sharp decrease in the surface energy and an increase in the contact wetting angle of the coatings with water and hexadecane when small additions of PTFE are introduced into the powder composition.

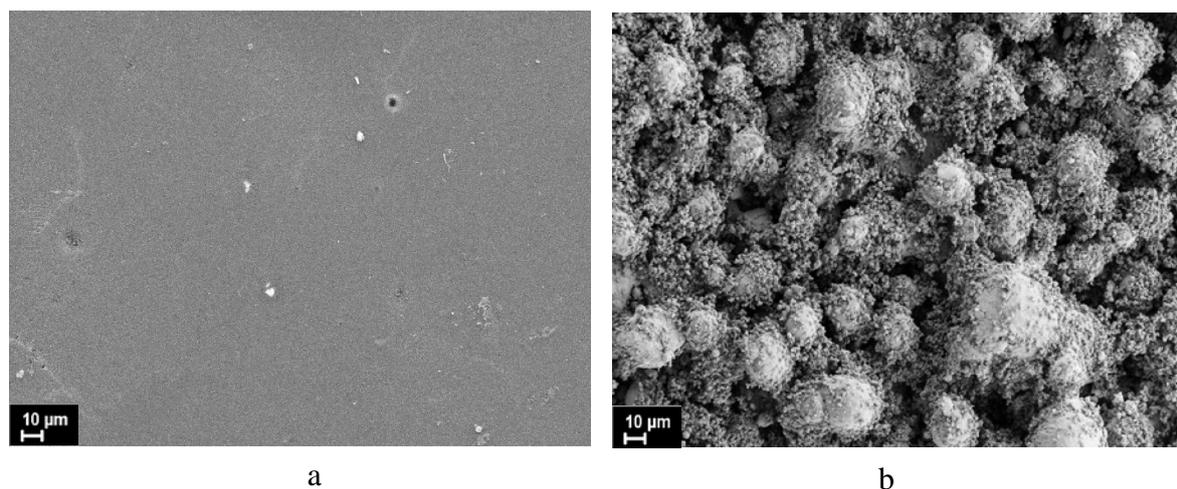


Fig. 2. SEM image of the coating surface without PTFE (a) and with 2 wt% PTFE (b)

Table 2. The effect of concentration PTFE on the physico-mechanical properties of epoxy-polyester coatings

PTFE content, wt%	Strength on impact, forward / reverse, cm	Tensile strength, mm	Adhesion point	Hardness	Roughness Ra, μm
0	100/100	8	1	2H	-
0.5	100/100	8	1	3H	1.7
1	100/100	8	1	2H	2.3
1.5	100/100	8	1	2H	4.3
2.0	100/100	7	1	2H	9.8
2.5	100/20	5	2	H	10.2

Data on the study of the physical and mechanical properties of coatings are given in Table 2. The introduction of fluoroplastic up to 2 wt% into the epoxy-polyester powder composition practically does not affect the change in the mechanical properties of the coatings, and they remain at a high level. However, at a PTFE concentration of 2.5 wt%, there is a decrease in mechanical characteristics. Apparently, with such a PTFE content, there is an excess of the particles adsorbed on the surface of the powder composition, which is no longer able to form a continuous coating, and the effect of chalking of the paintwork arises.

Since the PTFE introduction into the epoxy-polyester powder composition leads to the formation of certain multi-level surface topography, the average surface roughness of the coatings was investigated with a special digital profilometer. With an increase in the PTFE concentration in the epoxy-polyester powder composition, the surface roughness (Table 2) of the resulting coatings increases. The data obtained confirm the presence of a microstructure on the coating surface.

The effectiveness of the superhydrophobic coating is determined by its stability. The investigated coatings modified with 2 wt% PTFE and 0.1% SiO₂ had good storage stability under ambient conditions (the contact angle did not change even after a year). The stability of the superhydrophobic layer is determined by the preservation of its water-repellent properties upon contact with the medium. Distilled water and 3% NaCl solution were used as a medium in our work. The results are shown in Fig. 3 and Fig. 4. It was found that the coatings modified with PTFE and SiO₂ particles retained superhydrophobicity and oleophobicity even after holding for about 400 hours. However, after 400 hours of immersion of the coating, an

increase in the roll-off angle from 2 to 15° was observed. Moreover, for coatings kept in a saline solution, a decrease in the contact angle and an increase in the roll-off angle occurred more intensively and earlier. This is probably because salt crystals are deposited on the coatings and somewhat destroy the superhydrophobic surface. An increase in the roll angle can be associated with surface defects and irregularities. It is assumed that immersion forms hydrophilic defects on the surface, which leads to an increase in the roll angle without affecting the statistical wetting angle. It should be noted that after heating the coatings at the temperature of 120°C for 15 minutes, the roll-off angle and contact angles return to their initial values before immersion in the environment.

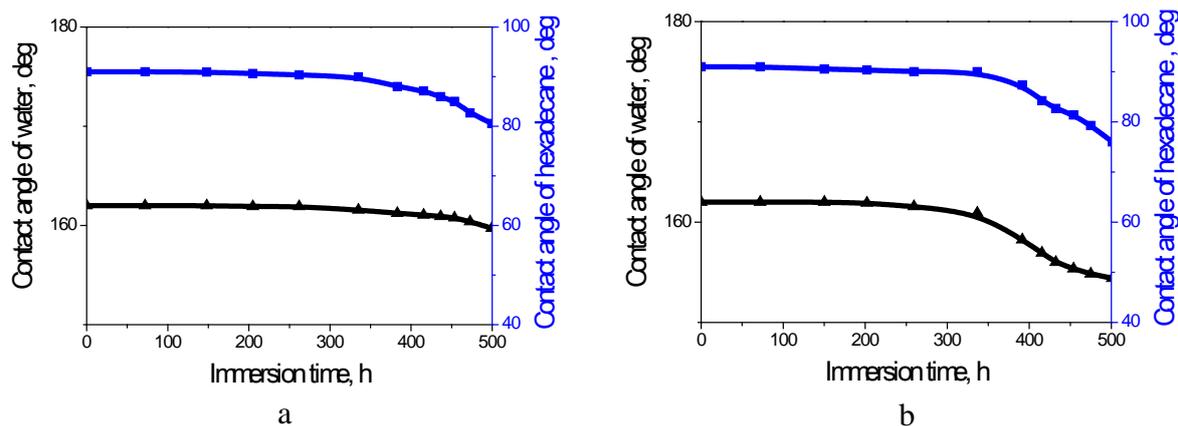


Fig. 3. Effect of immersion time in water (a) and solution 3% NaCl (b) on the contact angle of water and hexadecane of epoxy-polyester coatings modified by 2% wt PTFE and 0,1% SiO₂

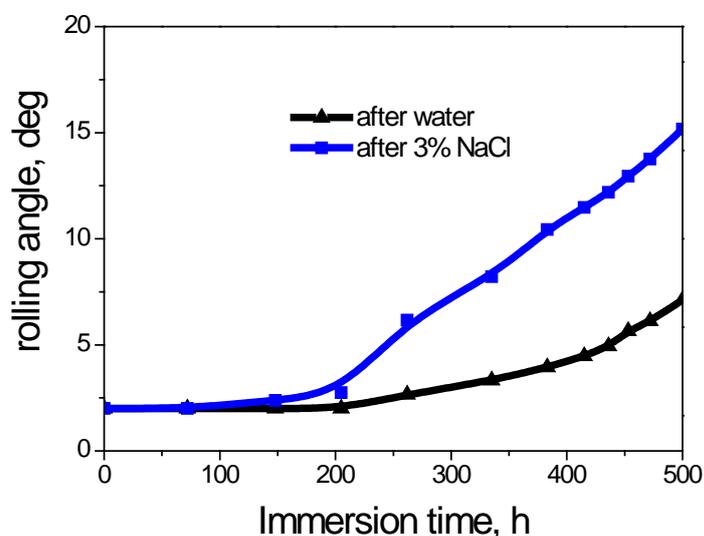


Fig. 4. Effect of immersion time in water and solution 3% NaCl on the rolling angle of water of epoxy-polyester coatings modified by 2% wt PTFE and 0,1% SiO₂

4. Conclusions

Modification of the epoxy-polyester powder compositions with the PTFE particles (up to 2 wt%) in the presence of trace amounts of hydrophobic SiO₂ nanoparticles makes it possible to obtain rough superhydrophobic polymer coatings with low surface energy and good physical and mechanical characteristics. By varying the PTFE content, it is possible to obtain hydrophobic and superhydrophobic coatings.

Investigation of the stability of the obtained superhydrophobic coating showed that they had good stability when exposed for 400 hours in water and saline solution (3% NaCl).

The proposed method is simple and economical. It can be used for the manufacture of stable coatings with varying degrees of roughness and with the values of contact wetting angles with water, characteristic of a hydrophobic, highly hydrophobic, and superhydrophobic surface.

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