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TECHNOLOGICAL AND OPERATIONAL PROPERTIES OF CATHODIC ELECTRODEPOSITION PRIMER

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Abstract. *The paper considers the impact of various dispersants on the dispersion time of pigment paste for cathodic electrodeposition primer and on some properties of the formed coating. The authors demonstrate that a dispersant based on nonionic surfactants (tetramethyldecynediol) in ethylene glycol enables dispersion of pigments and fillers in aqueous medium to the required particle fineness of ~15 μm (by grindometer) in minimal time. When using this dispersant, the best coating properties are observed.*

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Introduction

The market volume of electrodeposition materials is growing by an average of 5% annually. This is primarily due to tightening environmental regulations, growth in the automotive industry (particularly the global trend toward electric vehicles), and industrial/commercial construction and appliance manufacturing [1,2]. Cathodically electrodeposited materials provide excellent corrosion protection for metal products, uniform film thickness even on complex-shaped parts, and high painting efficiency [3]. Modern cathodic electrodeposition primers represent a two-component system consisting of a main binder dispersion and pigment paste. To maintain consistent bath parameters and coating properties in cathodic electrodeposition – especially in mass industrial production – the components must demonstrate long-term stability during operation [4-6]. Special requirements apply to the pigment paste, which is a suspension of pigments and fillers in synthetic resin with functional additives. In pigment paste production, the most critical, complex and energy-intensive stage is pigment dispersion, requiring proper selection of dispersing additives [7, 8]. The main challenges include [9]:

1. Preventing reflocculation of dispersed particles.
2. High water surface tension complicating wetting of low-surface-energy pigments.

3. High content of polar groups in water-dilutable resin.

Study [10] developed a production technology for cathodic electrodeposition primer pigment paste based on epoxy-amine oligomer.

The purpose of this work is to study the impact of various dispersants used in the production of pigment pastes for cathodic electrodeposition primer on the technological properties of the material and the operational properties of the coating.

The basis of the pigment paste for cathodic electrodeposition primer was a water-dilutable, acetic acid-neutralized epoxy-amine adduct (Fig. 1) with the following characteristics: apparent viscosity 6.24 Pa·s; mass fraction of non-volatile substances 57.2%; density 1.02 g/cm^3; flash point 58 °C [11-13]. This adduct was filled with titanium dioxide, kaolin, barium sulfate, and carbon black.

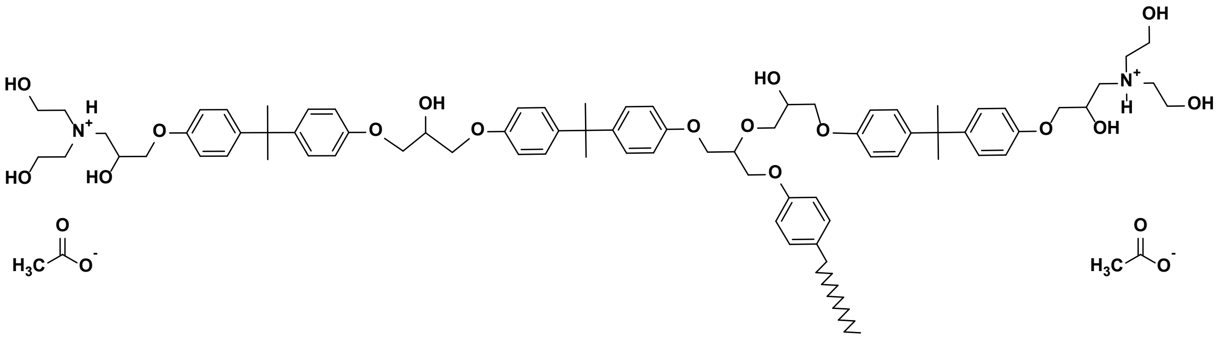

Fig. 1. General structural formula of the acetic acid-neutralized epoxy-amine adduct

The filling degree was 14.5 vol.%. We performed pre-dispersion of the pigment paste using a Dispermat LC-110 dissolver (manufactured by VMA-Getzmann) with a sawtooth disk at a peripheral speed of ~6 m/s for 15 minutes. We evaluated the dispersion fineness after this stage using a grindometer. Final dispersion was conducted in a Dispermat LC-110 immersion bead mill at a linear speed of 7.5-7.8 m/s until achieving a grinding fineness of 15 μm [14, 15].

To study the impact of dispersant type on the technological properties of the material and operational properties of the coating, we used the following substances:

1. Dispersant No. 1 is a transparent oily liquid, consisting of a 50% solution of a surfactant mixture (silicone-free) in ethylene glycol, recommended for aqueous systems. This dispersant provides pigment deflocculation through electrosteric stabilization.

2. Dispersant No. 2 is a transparent liquid with a slight yellow tint, consisting of a 50% solution of a nonionic surfactant (tetramethyldecynediol) in ethylene glycol, recommended for aqueous systems. It provides pigment deflocculation through electrosteric stabilization.

3. Dispersant No. 3 is a yellow-brown high-viscosity liquid, consisting of a 50% aqueous solution of a modified acrylic copolymer, recommended for aqueous systems, providing pigment deflocculation through steric stabilization.

We prepared four samples of cathodic electrodeposition primer pigment paste with the following functional additive contents:

1. Sample No. 1 – with 1 wt.% of dispersant No. 1.

2. Sample No. 2 – with 1 wt.% of dispersant No. 2.

3. Sample No. 3 – with 1 wt.% of dispersant No. 3.

4. Sample No. 4 – with 0,5 wt.% of dispersants No. 2 and No. 3.

The main binder in the two-component cathodic electrodeposition primer was an aqueous dispersion of an epoxy-amine adduct with blocked toluene diisocyanate as the crosslinking agent and functional additives. The aqueous dispersion of the epoxy-amine adducts had the following characteristics: apparent viscosity 0.25 Pa·s; mass fraction of non-volatile substances 37.1%; density 1.05 g/cm^3; flash point 97 °C; hydrogen index (pH) 5.51 units; acid milliequivalent 35.4 mmol/g.

The curing process catalyst was dioctyltin oxide in an amount of 3.3 wt.%.

The cathodic electrodeposition primer was prepared at a main binder to pigment paste ratio of 5:1 by weight. The calculated mass fraction of non-volatile substances was about 20%, with demineralized water used as the diluent [16].

To prepare the cathodic electrodeposition primer, we added the main binder to a container with demineralized water while stirring with a paddle mixer at a peripheral speed of 1.5 m/s. We mixed the resulting blend for 15 minutes, then gradually added the pigment paste in small portions while stirring. We stabilized the cathodic electrodeposition primer for 24 hours with constant stirring using a paddle mixer at a peripheral speed of 1 m/s.

Experimental part

The apparent viscosity of resins and pigment pastes with different dispersants was determined at (23.0±0.2) °C using a Brookfield viscometer (type A, spindle - disk No. 3 for dispersions and pigment pastes; disk No. 4 for dispersing resin) at 20 rpm [17]. The mass fraction of non-volatile substances in resins, obtained pigment paste samples and cathodic electrodeposition primers was determined according to [18], with a test sample weight of 1.0±0.1 g and drying time at 180 °C for 30 minutes. The density of resins was determined according to [19] at 20 °C, the flash point of resins in a closed cup was determined according to [20]. The acidity index of dispersions and obtained cathodic electrodeposition primers was determined at 25 °C according to [21], the acid milliequivalent of aqueous dispersion was determined according to [22], and specific electrical conductivity was determined according to [23].

Sedimentation of cathodic electrodeposition primers was evaluated as follows: a 500.0±1.0 g primer sample was poured into a 600 cm^3 glass beaker compliant with [24] using a class II precision balance [25]. The beaker was then covered and left undisturbed for 72 hours in a location without direct sunlight exposure at room temperature. After 72 hours without stirring, the test sample was stirred with a paddle mixer at a peripheral speed of 2.5 m/s, with the paddle mixer positioned 1 cm from the beaker bottom. The time required for complete redispersion of the settled sediment into the cathodic electrodeposition primer solution was evaluated according to [26].

Coating appearance was determined according to [27], coating thickness according to [28], coating tensile strength by Erichsen test [29], coating impact strength [30] using a U-1a tester (direct impact), adhesion by lattice cut method [31], gloss [32], resistance to 5% salt spray exposure (corrosion creep from scribe) (method B) [33].

To study the influence of functional additives on properties of coatings formed from cathodic electrodeposition primer, the following tests were conducted.

The cathodic electrodeposition primer was applied to 08ps steel plates with a pre-applied zinc phosphate layer [34-36]. A 08Kh18N10 stainless steel plate [37] served as the counter electrode, with a cathode:anode area ratio of 4:1. A P-TRP-15-400 converter serving as DC power source provided voltages up to 400 V. The working solution temperature of cathodic electrodeposition primer was maintained at 30.0-30.2 °C. Coating deposition time in bath was 150 s at potentiostatic mode of 290 V. After deposition, coatings were rinsed with distilled water, then placed in a drying chamber where thermal curing occurred at 165 °C for 20 minutes through reaction of unblocked isocyanate groups with hydroxyl groups [38, 39]. Coatings were conditioned at room temperature for 24 hours prior to testing.

Discussion of the results of the study

First, the study examined the influence of functional additives on the pigment paste dispersion process. Fig. 2 presents the results.

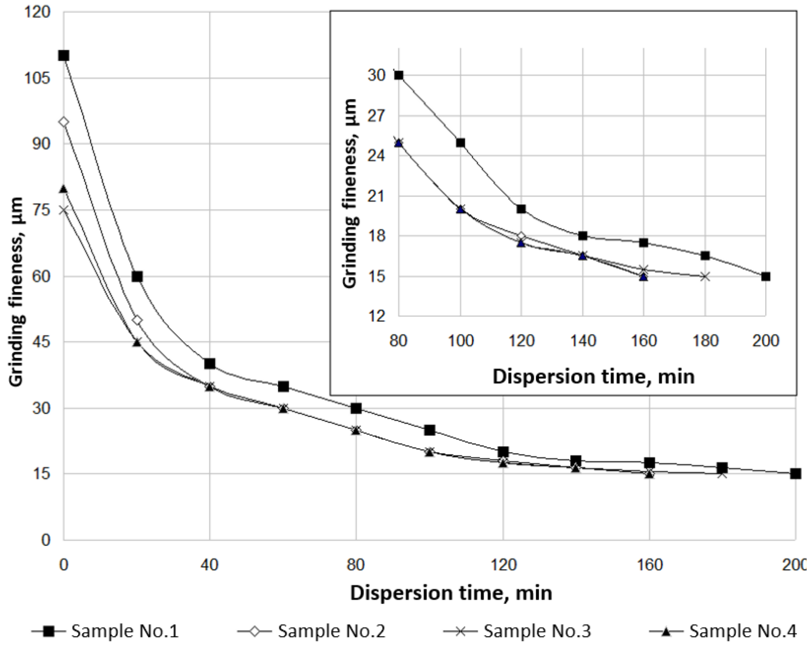

Fig. 2. Effect of dispersion time on grinding fineness of pigment pastes

The required grinding fineness of the pigment paste (15 µm) is achieved most quickly when using Dispersant No. 2 and a mixture of Dispersants No. 2 and No. 3 (Sample No. 4).

Table 1 shows some parameters of the obtained pastes.

Table 1. Parameters of cathodic electrodeposition pigment pastes

Indicator	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
Initial indicators				
Apparent viscosity, Pa·s	2.78	2.05	1.82	1.92
Mass fraction of non-volatile substances, %	55.6	55.1	55.4	55.4
Grinding level, µm	15	15	15	15
Parameters after 3-month aging				
Apparent viscosity, Pa·s	5.90	2.89	2.33	2.55
Viscosity change, %	112	41	28	33
Mass fraction of volatile substances, %	55.7	55.2	55.7	55.5
Grinding level, µm	15	15	15	15

It should be noted that all pigment paste samples have approximately the same mass fraction of non-volatile substances and grinding fineness. Moreover, storing the pigment pastes in closed containers for 3 months without exposure to direct sunlight at room temperature has practically no effect on these parameters. Only the impact of the functional additive type and storage duration on the apparent viscosity of the pastes is observed. Sample No. 1 has the highest initial apparent viscosity, which increases most rapidly during storage. This likely indicates that the dispersants used (especially Dispersant No. 1) significantly affect the structuring of the film-forming system. From a technological standpoint, it is more practical to use Dispersant No. 3 in the pigment paste, as pastes containing it show the lowest initial apparent viscosity and the slowest rate of viscosity increase over time.

Using samples of pigment pastes with different dispersants, cathodic electrodeposition primers were prepared according to the scheme described above. Table 2 presents the research results for the obtained primers.

Table 2. Characteristics of cataphoretic primers

Name of parameter	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
pH	5.35	5.40	5.32	5.32
Specific electrical conductivity, μS/cm	1538	1560	1547	1563
Mass fraction of non-volatile substances, %	19.95	20.01	20.09	19.99
Sedimentation, s	205	172	211	187

It has been established that the nature of the dispersants used affects only the sedimentation stability of the primers. Primer sample No. 2 demonstrates sufficiently high sedimentation stability; when the primer is aged for 72 hours, a sediment forms which completely redisperses within 172 s.

It has been determined that the nature of the dispersant does not affect the operational properties of the formed coatings. All parameters presented in Table 3 have similar numerical values, except for the coating resistance to 5% salt spray exposure. The corrosion creep from the scribe for primer sample No. 2 after 1500 hours is the smallest.

Table 3. Properties of the obtained coatings

Name of parameter	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
Appearance	Uniform, smooth, free from craters, pores and wrinkles	Uniform, smooth, free from craters, pores and wrinkles	Uniform, smooth, free from craters, pores and wrinkles	Uniform, smooth, free from craters, pores and wrinkles
Thickness, μm	18-20	18-20	18-20	18-20
Coating tensile strength (Erichsen test), mm	8.1	8.3	8.3	8.2
Impact resistance of coating, cm	50	50	50	50
Adhesion grade	0	0	0	0
Gloss, units at 20° angle at 60° angle at 85° angle	19-22 66-69 92-94	18-21 64-67 91-93	19-22 63-66 91-93	19-22 65-68 92-94
Resistance to 5% salt spray exposure, h with corrosion creep from scribe, mm	1500 1.9	1500 1.5	1500 1.8	1500 1.7

Conclusions

Hence, sample No. 2 of the cathodic electrodeposition primer, whose pigment paste was obtained using a solution of nonionic surfactant - tetramethyldecynediol (providing pigment deflocculation through electrosteric stabilization), demonstrates optimal technological and operational properties.

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