

The influence of filler type on performance properties of thin-layer polymer composites

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Abstract: The study aims to establish the impact of environmental factors on the properties of operational stability of coatings based on aqueous dispersions, depending on the surface properties of the filler used. The dispersion of acrylic latex and epoxy resin preliminarily emulsified with sodium polyacrylate was taken as the basis. Dispersion was carried out at a temperature of 60°C. Crushed marble, sedimentary chalk, chemically precipitated chalk and grade kaolin were used as fillers. The hardener was polyethylenepolyamine. Compounding was carried out in a laboratory dissolver: a filler was introduced into the dispersion, and then it was dispersed. The hardener was introduced immediately before coating. As a result, the degree of influence on the coating properties was established. The process of hydrophilization of the coating surface due to the oxidation of the polymer phase was studied. The operational reliability of coatings under the action of aggressive factors depending on the filler was evaluated. It is shown that when calcium carbonate is replaced by kaolin, an increase in the resistance to defective coatings is observed, and adhesion remains at a high level. It has been established that the influence of environmental factors on the properties of the coating are arranged in the following order: thermal and moisture aging > action of electrolyte solutions > action of ultraviolet. Under ultraviolet light, hydrophilization of the coating surface occurs due to the oxidation of the polymer phase.

Keywords: water-dispersion coatings; kaolin; crushed marble; calcium carbonate; epoxy resin; thermal aging; electrolyte action; ultraviolet.

For citation: Domnichenko RG, Karmanova OV, Tikhomirov SG. The influence of filler type on performance properties of thin-layer polymer composites. *Journal of Advanced Materials and Technologies*. 2024;9(1):037-043. DOI: 10.17277/jamt.2024.01.pp.037-043

Влияние наполнителей на эксплуатационные свойства тонкослойных полимерных композитов

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Аннотация: Работа посвящена исследованию влияния факторов окружающей среды на эксплуатационные свойства покрытий на основе водных дисперсий, полученных в присутствии наполнителей разного типа. В качестве полимерной основы использовали дисперсию акрилового латекса и эпоксидной смолы, предварительно эмульгированную с полиакрилатом натрия. Диспергирование осуществляли при температуре 60 °С. В качестве наполнителей использовали дробленый мрамор, осадочный мел, химически осаждённый мел, каолин. Отвердителем служил полиэтиленполиамин. Компаундирование осуществляли в лабораторном

дисольвере: в дисперсию вводили наполнитель, после чего осуществляли его диспергирование. Отвердитель вводили непосредственно перед нанесением покрытий. Исследовано влияние выбранных наполнителей на свойства получаемых покрытий. Изучен процесс гидрофилизации поверхности покрытия, протекающий за счет окисления полимерной фазы. Оценена эксплуатационная надежность покрытий при действии агрессивных факторов в зависимости от типа наполнителя. Показано, что при замене карбоната кальция каолином наблюдается повышение устойчивости покрытий к образованию дефектов, при этом адгезионные свойства сохраняются на высоком уровне. Установлено, что по влиянию на свойства покрытия факторы окружающей среды располагаются в ряду: тепло-влажностное старение > действие растворов электролитов > действие ультрафиолета. Под действием ультрафиолета имеет место гидрофилизация поверхности покрытия за счет окисления полимерной фазы.

Ключевые слова: водно-дисперсионные покрытия; каолин; дробленый мрамор; карбонат кальция; эпоксидная смола; тепловое старение; действие электролитов; ультрафиолет.

Для цитирования: Domnichenko RG, Karmanova OV, Tikhomirov SG. The influence of filler type on performance properties of thin-layer polymer composites. *Journal of Advanced Materials and Technologies*. 2024;9(1):037-043. DOI: 10.17277/jamt.2024.01.pp.037-043

1. Introduction

Coatings based on film-forming polymers obtained by removing the solvent or dispersion medium after application to the substrate differ in their performance properties from coatings made from melts. First of all, this applies to thin-layer coatings based on aqueous dispersions, with a film formed due to sequential processes of dispersion dehydration, deformation of dispersed particles of the polymer phase and their fusion [1, 2]. Obviously, at the fusion site the material will be characterized by structural defects, as well as an increased content of stabilizers and other targeted additives [3, 4]. A similar situation is observed at the boundary of the interphase contact with the filler surface, which, obviously, will affect the performance properties of such coatings, especially their resistance to external aggressive factors – ultraviolet radiation, moisture, temperature, electrolytes, etc. [5, 6].

The properties of cured coatings can be flexibly controlled through the use of mixed film formers. For example, there are known systems based on acrylic polymer – liquid glass, which exhibit a sufficient degree of vapor permeability and at the same time are impermeable to liquid [9]. Systems based on alkyd and polyurethane polymers demonstrate sufficient flexibility, while achieving a significant reduction in material costs compared to pure urethane ones. The properties of cured coatings can be flexibly controlled through the use of mixed film formers. For example, there are known systems based on acrylic polymer – liquid glass, which exhibit a sufficient degree of vapor permeability and at the same time are impermeable to liquid [9]. Systems based on alkyd and polyurethane polymers exhibit sufficient flexibility, while achieving a significant reduction in material costs compared to pure urethane ones.

The nature and condition of the film former determines such characteristics of the coating as wear resistance, mechanical strength, resistance to

aggressive environments, corrosion resistance, moisture resistance, elasticity, hardness, etc. [10].

These characteristics and the corresponding consumer properties of film formers can be significantly changed by adding functional additives to the coating, of which fillers occupy the most significant place [11].

The use of fillers not only improves the performance characteristics of the coating, but also increases the profitability of the paint and varnish material (paints and varnishes), since the price of fillers varies within 0.20–0.25 % of the price of the film former, especially when using local carbonate and silicate-type raw materials for their receiving [12, 13].

In modern paints and varnishes, the degree of filling can be increased to 50–60 % wt. and provide significant savings on expensive polymer [14–16].

It should be noted that the type of filler and its quantity determine the protective properties of the coating: liquid and vapor permeability, mechanical properties (strength, hardness, elasticity), adhesive strength between the coating and substrates, as well as the operational reliability of paintwork materials, assessed by such indicators as water resistance, corrosion stability, resistance to atmospheric factors [17, 18].

The influence of fillers on the structure of the coating and changes in its properties is associated primarily with the nature of their distribution in the polymer matrix, as well as with the interaction of dispersed fillers with the film-forming substance [19–21]. Consequently, increasing the stability of the interphase contact of the film former with the filler surface is a task, the solution of which will make it possible to control the operational reliability of such thin-layer systems. The purpose of this study was to establish the influence of environmental factors on the properties and operational stability of coatings based on aqueous dispersions, depending on the surface properties of the filler used.

2. Materials and Methods

2.1. Initial components

As a basis for obtaining the compositions, we used a hybrid dispersion based on acrylic latex UcarDL 450 (Dow Chemical, Germany, supplier LOTOS Trading LLC, Troitsk) and epoxy resin ED-20 (FKP Sverdlov Plant, Dzerzhinsk), pre-emulsified in the presence of 3 wt. % sodium polyacrylate (NPP Spetsavia LLC, Tver region). As a filler, components widely used in industry were chosen based on crushed marble (Normcal-20, produced by SomCalcite, Turkey), sedimentary chalk MMS-1 (Volcheyarovsk quarry, Lugansk region), chemically precipitated chalk (CPC) (Reaktiv LLC, Slavyansk) and kaolin grade KS-1 (JSC Microcalcite, Magnitogorsk).

2.2. Coating formation

Compounding was carried out in a laboratory dissolver: the filler was introduced into the dispersion at a speed of 300–400 rpm, after which it was dispersed at a speed of 1200 rpm for 15 minutes. Before applying the coatings, the hardener polyethylenepolyamine – PEPA (PJSC "Uralchimplast", Nizhny Tagil) was introduced into them in an equivalent amount, which was calculated according to the method [7].

To determine the basic properties of the coating, it was formed by spraying paintwork onto the prepared plates in one layer and dried in three stages:

I: at a temperature of $(20 \pm 2)^\circ\text{C}$ for 2 hours;

II: at a temperature of $(90 \pm 2)^\circ\text{C}$ in a drying cabinet for 1 hour;

III: at a temperature of $(20 \pm 2)^\circ\text{C}$ for 1 hour.

The thickness of the single-layer film after drying is 50–70 microns.

2.3. Instruments and research methods

The structure of fillers and polymer coatings was studied using a Specord IR 75 infrared spectrophotometer (Carl Zeiss, Germany). For dispersed fillers, tableted samples with KBr were used. For film formers and composite materials, spectra of free films with a thickness of 40–50 microns were obtained.

The specific surface area of dispersed materials was determined using a NOVA 2200 device (Quantachrome Corp. USA). Dispersity and granulometric composition were studied by optical microscopy using a JSM-5610 LV electron microscope with an EDX JED-2201 chemical

analysis system (JEOL, Japan). The contact angle of the surface with liquids was determined using a DIP-6 measuring microscope with a goniometric attachment (JSC LOMO, St. Petersburg).

The oil absorption capacity of the fillers was determined in accordance with Russian Standard 21119.8-75 (ISO 787-5-80). The method consists of gradually adding linseed oil to a sample of the test product, grinding them with a stick until a homogeneous mass is formed and determining the amount of oil consumed.

The moisture absorption of the free film was determined in accordance with Russian Standard 21513-76 on samples 3 cm wide, weighing 0.3–0.5 g. Samples in bottles were weighed on an analytical balance with an accuracy of 0.0001 g, after which they were placed in a desiccator with a relative humidity $(95 \pm 2)\%$ and after 24 hours weighed again to constant weight. Moisture absorption was calculated using the formula:

$$a = \frac{m_2 - m_1}{m_1 - m_0} \cdot 100,$$

where m_0 is weight of empty bottle, g; m_1 is weight of the bottle with film before testing, g; m_2 is weight of the bottle with film after keeping in a desiccator, g.

The adhesion of films to a metal substrate was determined by the method of grid cuts according to Russian Standard 15140-78 using a RMI-5 tensile testing machine (Polimermash Group LLC, St. Petersburg). The impact strength of coatings was determined using a U-1 device (Non-Destructive Control LLC, Yekaterinburg).

3. Results and Discussion

It has been found that carbonate fillers differ in the content of impurities. In particular, X-ray fluorescence analysis showed that the calcium content in the Normcal-20 sample was 99.1 wt. %, which was higher than that of the MMC-1 sample (98.0 wt. %).); the amount of silicon in Normcal-20 was up to 0.8 wt. % versus 1.2 and 0.9 wt. % for MMC-1 and CPC, respectively.

Based on the IR spectra, the corresponding differences in the parameters of the absorption bands responsible for the vibrations of the main structure-forming bonds of the carbonate anion were identified (Fig. 1): a noticeable shift in the maximum of the band for Normcal-20: a decrease in the wave number $1/\lambda = 1425\text{ cm}^{-1}$ to 1495 cm^{-1} for CPC. The ratio of their intensities (I_0/I) decreased accordingly from 45.33 to 35.25. The energy spectrum of vibrations of

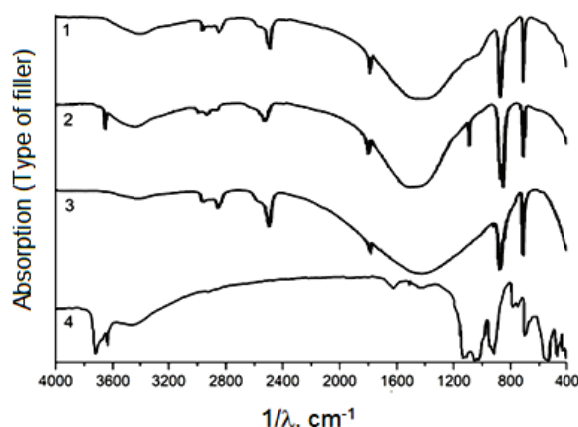


Fig. 1. IR-spectra of fillers:

1 – MMC-1; 2 – CPC; 3 – Normcal-20; 4 – kaolin KS-1

structural bonds also narrowed. The half-width of these same absorption bands decreased by 400 cm^{-1} for CPC and 235 cm^{-1} for MMC-1 chalk. The change in the parameters of the remaining bands was less pronounced.

The presence of hydroxyl groups on the surface was detected only in the case of chemically deposited chalk (absorption band at 3643 cm^{-1} with intensity ratio $I_0/I = 0.23$) (Fig. 1).

The maximum amount of adsorbed water ($I_0/I = 0.34$) was found in the CPC composition, and the minimum was found in Normcal-20 ($I_0/I = 0.09$). Judging by the position of the absorption bands responsible for the stretching vibrations of adsorbed water, it is noted that it is most firmly bound by the surfaces of CPC and MMC-1 (band at 3423 cm^{-1}). For ground marble, the absorption maximum is shifted by 18 cm^{-1} to the region of higher frequencies.

The IR spectrum of kaolin (Fig. 1) of the KS-1 grade contains absorption bands responsible for vibrations of surface and structural hydroxyl groups bound by silicon and aluminum atoms, as well as water [8]. It is these functional groups and compounds, as is known [9], that, together with the chemical composition and structural-chemical structure, determine the basic physicochemical properties of the surface of kaolins, namely: bands at 3680 and 3640 cm^{-1} are stretching vibrations of internal surface structural hydroxyl groups; maximum at 3613 cm^{-1} is vibrations of internal OH groups of the kaolin set; the maxima at 926 and 900 cm^{-1} are due to deformation vibrations of the structural hydroxyl groups of kaolinite associated with octahedral Al^{3+} cations; the bands at 3440 and 1605 cm^{-1} belong to stretching and bending vibrations of adsorbed water, respectively.

The analysis of the granulometric composition of natural mineral fillers showed that the content of the fraction from 0 to 5 microns in the composition of MMS-1 was 61.5 wt. %, and for kaolin KS-1 it was 28.7 wt. %.

It was found that the minimum specific surface area (Table 1) was observed in Normcal-20, which is 3 times less than that of MMC-1, 5 times less than that of CPC and 8.5 times less than that of kaolin KS-1.

The noted features of the composition and structure of the studied fillers influence their physicochemical properties in the dispersed state (Table 1).

It was found that kaolin had the lowest water contact angle with respect to water ($\theta = 43^\circ$). Calcium carbonates are more hydrophilic and are characterized by close values of contact angles, taking into account the error in measuring this indicator by the Washburn method, which does not exceed 3 degrees. The development of the surface and its polarity determine the physicochemical properties of the filler. The values of the dielectric loss tangent confirm the higher surface energy of kaolin compared to carbonates, of which sedimentary chalk and crushed marble turned out to be the least active. In particular, the moisture absorption of kaolin was greater compared to calcium carbonates: with Normcal-20 – 4.7 times; with CPC – 2.5 times.

In terms of oil absorption capacity, kaolin was somewhat inferior to chemically precipitated chalk and was almost twice as high as Normcal-20 and MMC-1.

The ultraviolet radiation factor is one of the main factors determining weather resistance. Under test conditions (intensity of total ultraviolet radiation $54\text{ W}\cdot\text{m}^{-2}$, exposure 20 hours at 60°C and relative humidity 35 %) the performance properties of epoxy-acrylic coatings changed significantly (Table 2).

The effect of UV radiation on coatings was photo-oxidation of the polymer part, which was accompanied by an increase in its polarity, and, consequently, in contact angles. This effect was more pronounced for composites with carbonate filler, which can be explained by the greater activity of the kaolin surface in terms of neutralizing peroxide radicals [8, 9]. An increase in the defectiveness of coatings also led to an increase in water absorption by 30–35 % for carbonates and by 16 % for kaolin. Accordingly, the impact strength indicator also decreased, but the most vulnerable under test conditions was adhesion, which for all coatings decreased by 21–23 %. Noteworthy is the fact that the adhesion index of coatings filled with kaolin decreased to a greater extent compared to the index of coatings obtained in the presence of other fillers.

Table 1. Physical and chemical properties of the surface of dispersed fillers

Type of filler	Properties*					
	θ , degree	S_{ads} , $\text{m}^2 \cdot \text{g}^{-1}$	$\text{tg}\delta$	W , wt. %	A , $\text{g} \cdot \text{g}^{-1}$	X , $\text{g} \cdot (100 \text{ g})^{-1}$
Sedimentary chalk MMS-1	26	2.15	0.018	1.56	0.70	23
Chemically precipitated chalk	31	3.18	0.035	1.70	1.65	57
Crushed marble Normcal-20	30	1.40	0.019	0.90	0.41	19
Kaolin KS-1	43	15.08	0.193	4.20	–	44

* θ is water contact angle; S_{ads} is BET specific surface area; $\text{tg}\delta$ is dielectric loss tangent; W is moisture absorption; A is water adsorption; X is oil absorption capacity.

Table 2. Changes in the physical and technical properties of filled epoxy-acrylic coatings under the influence of UV radiation

Type of filler	Properties*			
	θ , degree	Δ , wt. %	P , N·m	σ_A , MPa
Sedimentary chalk MMS-1	60 / 50	1.4 / 2.1	19.5 / 18.6	7.26 / 5.77
Crushed marble Normcal-20	65 / 59	1.3 / 2.0	20.6 / 18.6	7.32 / 5.85
Kaolin KS-1	83 / 76	2.1 / 2.5	23.8 / 22.0	7.64 / 5.51

* θ is water contact angle; W is moisture absorption in 24 hours; P is impact strength; σ_A is adhesion; in the denominator – data after testing, in the numerator – initial data.

Table 3. Corrosion resistance of filled epoxy-acrylic coatings

Type of filler	Properties			
	θ , degree	W , wt. %	P , N·m	σ_A , MPa
Sedimentary chalk MMS-1	60 / 58	1.4 / 1.5	19.5 / 19.3	7.26 / 6.84
Crushed marble Normcal-20	65 / 63	1.3 / 1.4	20.6 / 19.1	7.32 / 6.45
Kaolin KS-1	83 / 77	2.1 / 2.2	23.8 / 21.9	7.64 / 6.92

Table 4. Changes in the properties of filled epoxy-acrylic coatings in the process of thermal and moisture aging

Type of filler	Properties			
	θ , degree	W , wt. %	P , N·m	σ_A , MPa
Sedimentary chalk MMS-1	60 / 57	1.4 / 1.5	19.5 / 18.9	7.26 / 7.20
Crushed marble Normcal-20	65 / 61	1.3 / 1.6	20.6 / 19.0	7.32 / 7.23
Kaolin KS-1	83 / 80	2.1 / 2.4	23.8 / 22.4	7.64 / 7.61

When exposed to sodium chloride coatings of 3 wt. % for 120 hours, there was a weaker hydrophilization of their surface than in the case of UV radiation (Table 3).

At the same time, there was no significant change in the defectiveness of the structure, which is confirmed by a slight change in the water absorption

index (within the error limits of the method). The same patterns of changes in properties were observed for indicators of impact strength and adhesion. For kaolin compositions, adhesion remained at a fairly high level, decreasing by only 9 %.

It has been established that the aging of the coating under the influence of the heat and humidity

factor (24 hours at a temperature of 100 °C and relative humidity 100 %, Russian Standard 9.401) was least pronounced compared to the action of UV radiation and immersion in an electrolyte solution (Table 4).

The change in the values of water contact angles was on the limit of the measurement error, that is, under the influence of heat and humidity aging, significant destruction of the polymer did not occur. Water absorption of coatings increased, probably due to the accumulation of diffusing water at the interfaces. This effect was most pronounced (19 %) when using filler with a minimum surface activity – crushed marble. The adhesion index of coatings to the substrate remained at a high level with virtually no changes.

4. Conclusion

Thus, it has been found that, according to the degree of influence on the properties of coatings, the effects of external factors are arranged in increasing order: heat and humidity aging > the effect of electrolyte solutions (3 wt. % NaCl) > the effect of ultraviolet radiation. Moreover, only in the latter case significant hydrophilization of the coating surface due to oxidation of the polymer phase was observed.

The operational reliability of coatings under the influence of the listed aggressive factors depends, among other things, on the type of filler used. It has been shown that when replacing calcium carbonate with a relatively inactive surface with kaolin, an increase in resistance to increasing defectiveness of coatings under the influence of all three considered factors is observed, and adhesion remains at a high level. In general, we can conclude that increasing the orienting ability of the filler surface is one of the ways to increase the operational reliability of polymer compositions.

5. Funding

This study received no external funding.

6. Conflict of interests

The authors declare no conflict of interest.

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Received 30 January 2024; Accepted 29 February 2024; Published 26 April 2024



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