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Study of dispersed additives for the formation of polymer composite materials to increase the performance characteristics of friction units of transport vehicles

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Abstract

The article studies powdered materials of various nature and dispersion, differing in antifriction properties: micamuscovite, hexagonal boron nitride, copper oxide, and tungsten disulfide. The specific surface area of fillers for mica, h-BN, CuO and WS₂ are investigated, its indicators are $S_{w1} = 0.89 \text{ m}^2/g$, $S_{w2} = 1.17 \text{ m}^2/g$, $S_{w3} = 0.19 \text{ m}^2/g$ and $S_{w4} = 0.12 \text{ m}^2/h$. The true density of dispersed for mica $\rho = 3000 \text{ kg/m}^3$, h-BN – $\rho = 2600 \text{ kg/m}^3$, CuO – $\rho = 6000 \text{ kg/m}^3$ and WS₂ – $\rho = 7500 \text{ kg/m}^3$. When using IR spectral analysis, the content of organic and inorganic compounds and chemical bonds on the surface of fillers has been identified. Based on a comparison of the spectral characteristics of the studied powder materials, it is also found that all spectra are characterized by a significant intensity of the absorption bands of surface-active –OH, =C=O, =C-H, -CH₂-, -NH₂ groups.

Keywords: dispersed additive, filler, IR spectral analysis, polymer composite, specific surface area.

Introduction

At present, polymer composite materials (CMs) based on epoxy binder are widely used in many industries. The use of epoxy composite materials is relevant in shipbuilding [1-3]. To improve the performance of parts and friction units of vehicles based on polymer composites, there is used physical and chemical modification of the binder [4–6]. At the same time, an effective way to modify polymers is a combination of two binders of different nature [7] to properties provide improved in combination. Additionally, a rational ratio of fillers of different dispersion provides an improvement in performance and, directly, in the tribological properties of the formed composites. It is known [8-10] that physical properties of a powdered material depend on the physicochemical nature and activity of its surface. When an active additive is introduced into the epoxy composite, there is ensured interfacial interaction of the binder filler system components, which leads to an increase in the performance characteristics of the developed materials. Given the above, filler surface studies are important for understanding and controlling interfacial interactions in polymer crosslinking. At the same time, the combination of IR spectral analysis methods and determination of the specific surface area of additives will provide extended information on the activity of the

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 © 2022, Ivano-Frankivsk National Technical University of Oil and Gas. All rights reserved. filler surface and the ability to interact physically with the epoxy-polyester binder.

The authors [11–12] investigated the physical properties and structure of hexagonal boron nitride powder. The specific surface area of the material particles was determined and the features of its structure were established. It has been proven that boron nitride has a high thermal conductivity, low dielectric constant, low electrical resistance and is characterized by a graphite crystal lattice structure.

In [13], the specific surface area of micamuscovite particles at different fineness after mechanical grinding was studied. It was shown that with a decrease in the dispersion of mica particles, the specific surface area increased. It is also noted that mica is a widespread mineral, which in turn indicates a different chemical composition of muscovite mined in different deposits.

Based on the analysis of works [14, 15], it was found that in copper oxide nanocrystals, with a decrease in the average particle diameter, the Cu–O– ionic bond increased. This was confirmed by X-ray diffraction and infrared spectroscopy. An analysis of the IR spectra of tungsten disulfide makes it possible to state the presence of active –O–H groups on its surface. However, most of the works do not provide data on the dispersity of particles, their specific surface area, and do not indicate the origin of the material of the powders.

The aim of the work is to study the activity of dispersed additives on the formation of polymer composite materials with improved performance characteristics for friction knots of transport means.

Research materials and methods

The following dispersed powder materials were investigated in the work:

material 1 – mica 20–40 μ m (fractionated micromica of the "Standard" series (TU 5725-005-40705684–2001) grade MC-20-80. Mineral composition: mica-muscovite KAl₂[(OH, F)₂AlSi₃O₁₀]. Chemical composition according to TU: silicon oxide (SiO₂) – 44–50 %, aluminum oxide (Al₂O₃) – 27–35 %, iron oxide (Fe₂O₃) – less than 5 %, magnesium oxide (MgO) – less than 2 %, potassium oxide (K₂O) – more than 8 %, water-soluble salts – less than 0.2 %);

material 2 – hexagonal boron nitride h-BN (TU 2-036-1045-88);

material 3 – copper oxide (Cu (II) oxide). It is a binary inorganic compound with the chemical formula CuO, amphoteric oxide of divalent Cu (GOST 16539–79);

material 4 – tungsten disulfide (WS₂). The purity of the initial product is 99.0-99.9 %.

In the work, the following properties of fillers were studied: specific surface area (S_w) and true density, and there was performed IR spectroscopy.

The specific surface area was calculated by measuring the air permeability of the powder layer at a pressure close to atmospheric using the T-3 device for determining the specific surface area of powdered materials (the Tovarov device). The constant of the device (for the upper extension) is K = 23.42. This device allows us to measure the specific surface area in the range of $S_w = 0.05-1.5 \text{ m}^2/\text{g}$. The error in determining the value of the specific surface area did not exceed $\Delta S_w = \pm 0.015 \text{ m}^2/\text{g}$.

The true density of dispersed fillers was determined by the pycnometric method (GOST 9758–2012). A pycnometer with a working volume of $5 \cdot 10^{-5}$ m³ (GOST 22524–77) was used for the study. Defatted kerosene was used as a pycnometric liquid. The fillers were previously subjected to heat treatment at a temperature of $T = 378 \pm 2$ K for a time of $\tau = 2.0 \pm 0.05$ h. The mass of filler samples was determined on analytical scales with an accuracy of ± 0.001 g. The deviation of true density values of fillers in parallel experiments was 4–6% from the nominal value.

IR spectral analysis was used to identify the content of organic and inorganic compounds and study of chemical bonds on the surface of fillers. IR spectra were recorded on an IRAffinity-1 spectrophotometer (Japan) in the wavenumber range $v = 400-4000 \text{ cm}^{-1}$ using a single-beam method in reflected light. The spectrum was scanned by wave numbers $\lambda^{-1} = v$ on the diagram within 225 mm in the range of selected frequencies. Wave numbers, transmission intensity, and absorption band area were determined using the IRsolution software. The error in determining the wave number $\Delta v = \pm 0.01 \text{ cm}^{-1}$, and in determining the accuracy of the peak location $\Delta v = \pm 0.125$ cm⁻¹. The photometric accuracy was 0.2% with software control of the slit and integration time t = 10 s. Integration step $\Delta \lambda = 4$ cm⁻¹. All samples of the studied fillers were pressed into a tablet with KBr.

Research results and discussion

Four powdered materials of different nature and dispersion were selected for the study: mica-muscovite $(KAl_2[(OH, F)_2AlSi_3O_{10}])$, hexagonal boron nitride (h-BN), copper oxide (CuO) and tungsten disulfide (WS₂). The selection criterion for powders is their antifriction properties, since in the future, it is planned to use them as fillers for the formation of epoxypolyester composites with improved tribological characteristics.

Muscovites are classified as minerals, the crystals of which have a lamellar structure. The chemical composition of mica minerals varies. On the other hand, morphological and physical properties are similar to different minerals. According to their chemical composition, these minerals mainly consist of aluminosilicates with the presence of alkalis and hydroxides. The main characteristic of mica minerals is the stratified and complex pseudo-hexagonal crystal structure. According to the above, mica minerals are characterized by specific physicochemical, mineralogical. and mechanical properties [13]. The hexagonal modification of boron nitride has a similar crystal structure to graphite (therefore it is known as white graphite). This crystal structure provides improved lubricity, high thermal conductivity, dielectric strength and low dielectric constant [12]. In each layer, the boron and nitrogen atoms are bound by strong covalent bonds, while the layers are held together by weak van der Waals forces [16]. Copper (II) oxide powder is widely used in various industries, in particular as an antifriction filler for polymeric materials. Due to its properties, it improves the resistance of the composite to corrosion and increases the physical, mechanical and thermal properties [10]. Tungsten disulfide, like hexagonal boron nitride, has a graphite-like hexagonal structure, and therefore affects the tribological properties of composite materials. Chemically, WS₂ is highly stable, insoluble in strong acids and alkalis, water, grease, acetone. WS₂ does not cause corrosion of metals [15].

At the first stage, the specific surface area and the true density of the aforementioned particulate fillers were calculated. When analyzing the technological parameters of powdered materials, it was found that the value of the specific surface area of particles was of decisive importance [17]. The results of the study of the specific surface area and the true density of the studied samples of fillers are given in Table 1. It is known that the specific surface area depends on the fineness of the filler [13]. Therefore, fillers with different particle sizes were used. The particle sizes of the powders and their molecular weights are taken according to the technical information.

It has been established that the obtained indicators of the true density of these powdered materials correlate with similar studies indicated in the works [13, 18–20]. The accuracy of determining this characteristic of the filler has a direct impact on the reliability of the research results [21].

The specific surface area S_w of the studied powders was determined by the layer air permeability method. Permeability methods are based on the study of

Characteristics	KAl ₂ [(OH,F) ₂ AlSi ₃ O ₁₀]	h-BN	CuO	WS ₂
True density, kg/m ³	3000	2600	6000	7500
Molecular mass, g/mol	—	24.82	79.55	247.97
Particle size, µm	20–40	8-10	8-10	8-10
Specific surface area, m ² /g	0.89	1.17	0.19	0.12





filtration processes in a cellular material. The filter medium is a layer of powder with a known degree of compaction. For mica-muscovite with a particle size of $d = 20-40 \ \mu m$, the value of the specific surface area $S_{w1} = 0.89 \text{ m}^2/\text{g}$ was obtained. It should be noted that the Tovarov device allows us to determine only the external specific surface area of powders. In contrast to our research results, in [13], the specific surface area was calculated by the BET method. With an average diameter of mica particles $d = 32.59 \mu m$, the value $S_w = 0.47 \text{ m}^2/\text{g}$ was obtained. That is, different methods for determining the specific surface area were used. This difference is important because the value of S_w depends on the method and conditions of the experiment. Therefore, it is inappropriate to compare the results of testing samples of the same material, but obtained by different methods or with different particle sizes

Accordingly, for h-BN, CuO and WS₂, the following specific surface area values were obtained: $S_{w2} = 1.17 \text{ m}^2/\text{g}$, $S_{w3} = 0.19 \text{ m}^2/\text{g}$, $S_{w4} = 0.12 \text{ m}^2/\text{g}$. As a result of experimental studies, it was found that with the same dispersion of h-BN, CuO and WS₂ powders, the copper (II) oxide material had a low specific surface area ($S_{w3} = 0.19 \text{ m}^2/\text{g}$). Accordingly, the value of the specific surface area of the hexagonal boron nitride powder increased significantly (up to $S_{w2} = 1.17 \text{ m}^2/\text{g}$), compared to the CuO material. These results show that the h-BN material is characterized by a large number of surface layer molecules. This indicates that there are more active groups on the surface of the material particles that can interact with the chemical bonds of the polymer binder.

IR spectral analysis was used to study chemical bonds on the surface of powdered materials particles. The group frequency approach is effective for structural analysis [16]. From the frequencies and intensities of the spectral bands, we can determine which chemical groups are present in the molecule, how they are related to other groups, and it is possible to establish the structure of the molecule under study.

Fig. 1 shows a typical IR spectrum for a micamuscovite sample. Each absorption band, calculated in the work, corresponds to specific vibrations of molecular units in a muscovite crystal [22-23]. The absorption band at $v = 500 \text{ cm}^{-1}$ is characteristic of ≡Si–O– bending vibrations. It was also found that there is an absorption band at v = 646 cm⁻¹, which corresponds to bending vibrations of the =Si-O-Si= group. Intense absorption bands at v = 1065 cm⁻¹ correspond to the stretching and bending vibrations of SiO_4 , which affect the substitution of Al^{3+} for Si^{4+} cations in the [AlSi₃O₁₀] tetrahedral sites. The observed absorption bands at v = 2353 cm⁻¹ are characteristic of =C=O stretching vibrations. Weakly pronounced absorption bands at v = 2853 cm⁻¹ and v = 2922 cm⁻¹ frequencies are characteristic of structural -CH2asymmetric and symmetric stretching vibrations, respectively. The absorption band v = 3645 cm⁻¹ corresponds to the stretching vibrations of -O-H groups. It is known that the -O-H vibration frequency depends not only on the strength of the -O-H bond and the reduced mass of the vibrational system, but also on the mass and valence of cations associated with -O-H groups in dioctahedral micas [24]. Therefore, this band $(v = 3645 \text{ cm}^{-1})$ is assigned to -O-H groups associated

Characteristics of the band	IR spectrum		
Group	$v, \text{ cm}^{-1}$	<i>T</i> , %	<i>S</i> , %
≡Si–O– deformation oscillations	500	28.8	12.8
=Si–O–Si≡ deformation oscillations	646	12.9	66.7
SiO ₄ valence and deformation vibrations	1065	30.5	22.3
=C=O valence vibrations of the CO_2 molecule	2353	21.9	7.5
-CH ₂ - asymmetric valence oscillations	2853	20.0	41.5
-CH ₂ - symmetric valence vibrations	2922	18.6	34.5
-O-H valence vibrations	3645	8.3	11.4

Table 2 – Spectral characteristics of mica-muscovite

Characteristics of the band	IR spectrum		
Group	v, cm^{-1}	<i>T</i> , %	<i>S</i> , %
B-N-B deformation oscillations	814	15.8	22.5
e-BN fluctuations	918	22.8	37.5
w-BN fluctuations	1123	21.2	5.1
w-BN fluctuations	1269	20.2	5.3
=C=O valence vibrations of the CO ₂ molecule	2332	24.0	30.6
-NH ₂ symmetric valence vibrations	2521	23.2	28.6
=C-H valence vibrations	2795	24.8	50.2
-CH ₂ - symmetric valence vibrations	2922	26.4	22.0
=C-H valence vibrations	3024	25.0	32.9
–O–H valence vibrations	3522	15.1	7.8

Table 3 – Spectral characteristics of hexagonal boron nitride



with Al^{3+} cations in octahedral layers. Table 2 shows the characteristic absorption bands of $KAl_2[(OH,F)_2AlSi_3O_{10}]$ and the intensity parameters of the absorption bands (*T*, %) and the relative peak area (S, %).

At the next stage, the surface activity of the boron nitride powder was studied by IR spectral analysis. Spectral characteristics of h-BN are given in Table 3. These parameters of the spectrum correlate with the studies of the authors [11–12]. There is revealed an absorption band v = 814 cm⁻¹, characteristic of bending vibrations B-N-B (Fig. 2). The absorption bands at v = 918 cm⁻¹, which is associated with the presence of

the e-BN phase together with h-BN, are calculated. Insignificant absorption bands, which are observed in the studied range at v = 1123 cm⁻¹ and v = 1269 cm⁻¹, are of interest. These peaks characterize the content of the phase of hexagonal wurtzite-type boron nitride (w-BN). An absorption band = 2332 cm⁻¹, which is characteristic of stretching vibrations of the =C=O group, is also revealed. The absorption band v = 2521 cm⁻¹ characterizes NH₂ symmetric stretching vibrations related to the surface B-NH₂ amino groups during the formation of amide bonds.

Vibration bands at $v = 2795 \text{ cm}^{-1}$ and $v = 3024 \text{ cm}^{-1}$ (=C-H stretching vibrations) provide information about



Figure 3 – IR spectrum of oxide (II) copper

Table 4 – Spectral characteristics of CuO

Band characteristics	IR spectra		
Group	<i>v</i> , cm ⁻¹	<i>T</i> , %	<i>S</i> , %
Cu(II)–O stretching vibrations	457	26.0	7.5
Cu(I)–O stretching vibrations	631	11.0	70.1
=C=O stretching vibrations of the CO ₂ molecule	2324	16.1	9.1
=C=O stretching vibrations	2355	15.4	3.1

hydrogen bonded to carbon atoms and impurities. As indicated above (in the spectrum of muscovite), the absorption bands are weakly pronounced at v = 2922 cm⁻¹, which is typical for structural -CH₂-symmetric stretching vibrations (Table 3). The absorption band v = 3522 cm⁻¹ refers to free -OH stretching vibrations in the surface B-OH groups.

Figure 3 shows the IR spectrum of copper (II) oxide powder in the infrared region v = 400-4000 cm⁻¹. Four absorption bands v = 457 cm⁻¹, v = 631 cm⁻¹, v = 2324 cm⁻¹ and v = 2355 cm⁻¹ were determined in the studied range (Table 4). The absorption band v = 457 cm⁻¹ characterizes the Cu(II)–O stretching vibrations. The strongly pronounced absorption band v = 631 cm⁻¹ corresponds to Cu(I)–O stretching vibrations. There are figured absorption bands v = 2324 cm⁻¹ and v = 2355 cm⁻¹, which are characteristic of stretching vibrations of the =C=O group in the CO₂ molecule. The resulting absorption bands are in good agreement with the spectra of CuO in previous works [14, 25].

At the last stage, the surface activity of the tungsten disulfide powder was studied in the infrared site of 400–4000 cm⁻¹. Spectrum analysis made it possible to reveal three absorption bands in the studied range (Fig. 4). There was determined a weak absorption band v = 820 cm⁻¹ attributed to W–S vibrations. Absorption bands v = 2340 cm⁻¹ and v = 2355 cm⁻¹ are characteristic of stretching vibrations of the C=O group in the CO₂ molecule. The absorption bands presented in the study of WS₂ powder (Table 5) agree with similar

spectra obtained in previous studies by the authors of [15].

IR spectral analysis made it possible to reveal the dominant groups on the surface of the powders under study. When comparing the spectral characteristics of the studied powder materials (Tables 2-5), it was found that all spectra were characterized by a significant intensity of the absorption bands of surface-active -OH, =C=O, -C-H, -CH₂-, -NH₂ groups. On the IR spectrum of mica, a group of intense bands can be distinguished in the range of wave numbers 450–1200 cm⁻¹. These absorption bands cause vibrations of the Si and O atoms along the connecting lines, i.e. vibrations of the =Si-Obond in the SiO₄ tetrahedron. All absorption bands corresponding to silicates are located in a wide spectral range. This is explained by the complexity of the structure and the presence of a large number of atomic groups, including not only Si and O atoms, but also others. The vibration frequency of the bond of the =Si-O- group is significantly affected by the nature of the atoms surrounding it, especially the atoms bonded to oxygen. When analyzing the hexagonal spectrum of boron nitride, there was established the presence of three phases of the crystal structure: h-BN, e-BN and w-BN. Obviously, during production, due to high temperatures, some h-BN can be converted to the w-BN modification.

The simultaneous combination of methods for determining the specific surface area and IR spectral analysis in the identification of substances and materials provides a synergistic effect in obtaining data on the composition and structure of the studied materials.



Table 5 – Spectral characteristics of tungsten disulfide

Band characteristics	IR spectra		
Group	$v, \text{ cm}^{-1}$	<i>T</i> , %	<i>S</i> , %
W–S fluctuations	820	30.3	11.9
=C=O stretching vibrations of the CO ₂ molecule	2340	20.6	7.9
=C=O stretching vibrations	2355	19.5	31.0

Conclusions

The article studies the specific area and surface activity of the following powders and powder materials: mica-muscovite $KAl_2[(OH,F)_2AlSi_3O_{10}]$, hexagonal boron nitride (h-BN), copper (II) oxide (CuO), tungsten disulfide (WS₂). It can be noted from the results of the experiments the following.

It has been established that the true density and specific surface area for mica are $\rho=3000~kg/m^3$ and $S_{w1}=0.89~m^2/g,$ respectively. For materials with a dispersion of $8...10~\mu m$, respectively, we obtained: h-BN $-\rho=2600~kg/m^3$ and $S_{w1}=1.17~m^2/g,$ CuO $-\rho=6000~kg/m^3$ and $S_{w1}=0.19~m^2/g$ and $WS_2-\rho=7500~kg/m^3$ and $S_{w1}=0.12~m^2/h.$

An IR spectral analysis of mica, h-BN, CuO, WS₂ was carried out, which made it possible to estimate the activity of the surface of the materials under study. In the range v = 400-4000 cm⁻¹, there are calculated the spectral characteristics for each of the powders. IR spectral analysis of muscovite particles enabled us to reveal absorption bands at $v = 500 \text{ cm}^{-1}$, $v = 646 \text{ cm}^{-1}$ and v = 1065 cm⁻¹, characterizing the nature of atoms and the structure of the mica material. The spectrum of hexagonal boron nitride has been analyzed and the type of absorption corresponding to B-N-B deformation vibrations has been found. Three phases of the crystal structure on the spectrum of boron nitride are stated: h-BN, e-BN and w-BN ($v = 814 \text{ cm}^{-1}$, $v = 918 \text{ cm}^{-1}$, v = 1123 cm⁻¹, and v = 1269 cm⁻¹, respectively. In the studied region of the spectrum of copper oxide, there were computed four absorption bands $v = 457 \text{ cm}^{-1}$ $v = 631 \text{ cm}^{-1}$, $v = 2324 \text{ cm}^{-1}$ and $v = 2355 \text{ cm}^{-1}$, corresponding to stretching, deformation and symmetric vibrations of groups, characteristic of the CuO material.

Three characteristic absorption bands were established for the spectrum of tungsten disulfide: $v = 820 \text{ cm}^{-1}$, $v = 2340 \text{ cm}^{-1}$, and $v = 2355 \text{ cm}^{-1}$. All investigated powder spectra are characterized by a significant intensity of the absorption bands of surface-active -O-H, =C=O, -C-H, $-CH_2-$, $-NH_2$ groups.

The results of the study of the specific surface area and IR spectral analysis of powdered materials allow us to assert the effectiveness of using these powders in the form of antifriction fillers for epoxy and epoxypolyester composites in order to improve the operational characteristics of friction units of vehicles.

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Дослідження дисперсних добавок для формування полімеркомпозитних матеріалів з метою підвищення експлуатаційних характеристик вузлів тертя засобів транспорту

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У роботі досліджено порошкоподібні матеріали різної природи та дисперсності, які відрізняються антифрикційними властивостями: слюда-мусковіт, гексагональний нітрид бору, оксид міді та дисульфід вольфраму. Вивчено питому площу поверхонь наповнювачів для слюди, h-BN, CuO та WS₂, її показники становлять $S_{w1} = 0.89 \text{ m}^2/\text{r}$, $S_{w2} = 1.17 \text{ m}^2/\text{r}$, $S_{w3} = 0.19 \text{ m}^2/\text{r}$ та $S_{w4} = 0.12 \text{ m}^2/\text{r}$. Визначено густину дисперсних добавок слюди $\rho = 3000 \text{ кг/m}^3$, h-BN – $\rho = 2600 \text{ кг/m}^3$, CuO – $\rho = 6000 \text{ кг/m}^3$ та WS₂ – $\rho = 7500 \text{ кг/m}^3$. Із використанням ІЧ-спектрального аналізу ідентифіковано вміст органічних і неорганічних сполук та хімічні зв'язки на поверхні наповнювачів. На основі порівняння спектральних характеристик досліджуваних порошкоподібних матеріалів встановлено, що всі спектри характеризуються значною інтенсивністю смуг поглинання поверхнево-активних –OH, =C=O, =C-H, -CH₂-, -NH₂ груп.

Ключові слова: дисперсна добавка, ІЧ-спектральний аналіз, наповнювач, питома площа поверхні, полімерний композит.