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THE EFFECT OF MONTMORILLONITE ON THE RHEOLOGICAL AND PHYSICO-MECHANICAL PROPERTIES OF A THERMOSETTING POLYMER COMPOSITE

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Abstract: The study investigates a thermosetting polymer filled with montmorillonite. Montmorillonite is a naturally occurring layered mineral with particle sizes ranging from 8 to 12 μm. The thermosetting polymer was DER-330 epoxy resin and FCH-S hardener. Rheological and physical-mechanical studies of the properties of epoxy resin modified with montmorillonite were carried out. Viscosity versus shear rate is shown for uncured pure epoxy resin and resin modified with montmorillonite particles at room temperature. At low shear rates, a significant effect of montmorillonite on the properties of the composite is observed. The low shear rate further allowed epoxy molecules to leak between the layers of montmorillonite platelets, causing swelling and increasing the distance between them. As the shear rate increased from 8 to 20 s-1, the percolation time of montmorillonite decreased, which affected the decrease in viscosity. When the shear rate increases above 20 s-1, no significant changes in viscosity occur; for both samples a monotonic line is observed on the graph. The addition of montmorillonite to epoxy resin changes the chemical composition of the composite, which leads to changes in rheological and physical-mechanical properties. The addition of montmorillonite retards gelation, which can affect processability, cure time, and final properties of the composite. Morphological studies were carried out to determine the degree of dispersion of montmorillonite particles in the polymer composite using a transmission electron microscope. The uneven distribution of montmorillonite particles predominated in all samples. Apparently this is due to the curing conditions of the polymer composite and insufficient dispersion time. Physical and mechanical studies were carried out on samples made of a composite with montmorillonite in comparison with samples from pure epoxy resin. The flexural strength of samples made from the resulting composite is comparable to the strength of samples made from pure epoxy resin. In turn, the viscosity increased by 170%.

Keywords: polymer materials, epoxy composite, montmorillonite, polymer fillers, thermosetting resins

ВЛИЯНИЕ МОНТМОРИЛЛОНИТА НА РЕОЛОГИЧЕСКИЕ И ФИЗИКО-МЕХАНИЧЕСКИЕ СВОЙСТВА ТЕРМОРЕАКТИВНОГО ПОЛИМЕРНОГО КОМПОЗИТА

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Аннотация: В данной работе были проведены исследования терморактивного полимера, наполненного монтмориллонитом. Монтмориллонит представляет собой встречающийся в природе слоистый минерал, размер частиц которого составляет от 8 до 12 мкм. В качестве термореактивного полимера использовалась эпоксидная смола DER-330 и отвердитель FCH-S. Проведены реологические и физико-механические исследования свойств эпоксидной смолы, модифицированной монтмориллонитом. Показана зависимость вязкости от скорости сдвига для неотвержденной чистой эпоксидной смолы и смолы, модифицированной частицами монтмориллонита при комнатной температуре. При низкой скорости сдвига наблюдается значительное влияние монтмориллонита на свойства композита. Низкая скорость сдвига дополнительно позволила молекулам эпоксидной смолы просачиваться между слоями пластинок монтмориллонита, вызывая набухание и увеличение расстояния между ними. По мере увеличения скорости сдвига с 8 до 20 $с^{-1}$ время перколяции монтмориллонита уменьшалось, что повлияло на уменьшение вязкости. При увеличение скорости сдвига выше значения 20 $с^{-1}$ каких-либо значимых изменений вязкости не происходит, для обоих образцах наблюдается на графике монотонная линия. Добавление монтмориллонита в эпоксидную смолу изменяет химический состав композита, что приводит к изменениям реологических и физико-механических свойств. Добавление монтмориллонита замедляет гелеобразование, что может влиять на технологичность, время отверждения и конечные свойства композита. Были проведены морфологические исследования для определения степени дисперсности частиц монтмориллонита в полимерном композите с использованием просвечивающего электронного микроскопа. Неравномерное распределение частиц монтмориллонита преобладало во всех образцах. По-видимому это связано с условиями отверждения полимерного композита и недостаточности времени диспергирования. Проводились физико-механические исследования образцов из композита с монтмориллонитом в сравнении с образцами из чистой эпоксидной смолы. Прочность на изгиб образцов из полученного композита сопоставима с прочностью образцов из чистой эпоксидной смолы. В свою очередь вязкость увеличилась на 170%.

Ключевые слова: полимерные материалы, эпоксидный композит, монтмориллонит, наполнители для полимеров, термореактивные смолы

1. INTRODUCTION

Thermosetting polymers are widely used in the construction industry as repair compounds for cracks (defects), anti-corrosion protection of building structures, and in decorative and finishing works. Depending on the conditions of use of thermosetting polymers, it is necessary to use various structural and functional fillers to obtain the necessary physical, mechanical and rheological properties. To improve the mechanical and thermal properties of the composite, small amounts of montmorillonite are introduced into its composition [1-3]. Montmorillonite has a unique morphology in the form of flakes with a thickness of about 1 nm with linear surface dimensions of 50–150 nm, which provides good barrier properties to moisture and volatile substances [4]. Studies [5] have shown that montmorillonite accelerates the rate of chemical reaction of the polymer composite. The interaction of montmorillonite particles with epoxy resin molecules affects the viscosity and mobility of the reacting particles during the curing process, which changes the curing rate, reaction time, enthalpy, glass transition, activation energy, viscoelastic properties and cross-linking density of the final composite [5, 6]. In addition, it was shown in [7, 8] that the type and amount of fillers and organic modifiers, as well as their interaction with the polymer base and their hardeners, affect the properties of the resulting composites.

The fillers introduced into its composition have a significant impact on the curing parameters of a polymer composite. Therefore, the manufacturer's recommended instructions for the use of thermosetting polymers and hardeners are not suitable for correct use. It is necessary to take into account the influence of fillers on the kinetic parameters of curing (activation energy) of polymer composites.

Gelation is of decisive importance in a thermosetting reaction. The use of a liquid polymer composite beyond the gelation point becomes virtually impossible due to the onset of crosslinking coupled with the rapid increase in viscosity. Therefore, understanding the influence of montmorillonite morphology on the rheological properties of thermoset polymer composites is critical, since it is directly related to the consistency of fluidity during use and the effect on the physical and mechanical properties of the thermoset polymer composite product.

2. MATERIALS AND METHODS

The following materials were used for the study: epoxy resin DER-330 (diglycidyl ether of bisphenol A, DGEBA); slow hardener FCH-S, manufactured by The Dow Chemical Company (Germany); montmorillonite Garamite 7305 (BYK, Germany). The montmorillonite used is a naturally occurring layered mineral with a chemical composition of $A_{0.3}(Al_{1.3}Mg_{0.7})[Si_4]O_{10}(OH)_2H_2O$, where "A" is the exchange cation, K^+, Na^+ or $0.5Ca^{2+}$. The chemical structure is shown in Figure 1. The particle size distribution of montmorillonite clusters ranged from 8 to 12 microns. This section specifies and describes in detail the chosen research method. The description should be in such a way that a qualified reader can reproduce it as a whole.

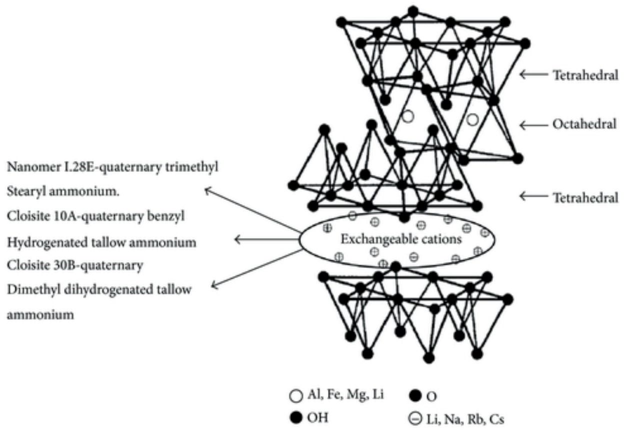

Figure 1. Chemical structure of montmorillonite [9]

The components were mixed using a magnetic stirrer MSH-300 (Biosan, Latvia). All formulations contained 10.0 g of resin and 3.3 g of hardener. A hardener and montmorillonite were alternately added to the working epoxy mixer. The mixing time of each component was 2 minutes, which made it possible to obtain a homogeneous mixture.

Morphological studies were carried out using an EMV-100A electron microscope (JSC SELMI, Ukraine).

The rheological properties of the formulations were measured under normal environmental conditions using a REOTEST RV2.1 rotary viscometer (VEB MLW, Germany) with a cylinder–cylinder working unit in the concentration range of 0.5 – 20.0%. The shear rate range was 0.3–1312 s^{-1}.

Samples for physico-mechanical studies were carried out with dimensions of 61×12×3.2 mm. The obtained samples were hardened in two stages at two temperature conditions – first at 100 °C for 24 hours, and then at 220 °C for 2 hours in a drying cabinet SHSU-M1 (manufactured by NPO Laborkomplekt, Russia).

Before taking measurements, the samples were purified with acetone. Three samples were tested for each composition. Bending tests were performed at room temperature on a universal testing machine TC 101-5-1-U (TEST SYSTEM production, Russia). In accordance with GOST R 56810-2015 "Polymer composites. Bending test method for flat samples" the dimensions of the sample span are regulated depending on its thickness. With a thickness greater than 1,6 mm, the span between the supports should be 16 times the thickness of the sample (tolerance ± 1 time). Therefore, during the bending test, the span between the supports was 51 mm, and the slider speed was 1.3 mm/min.

3. RESULTS AND DISCUSSION

The change in the rheological and physicomechanical properties of a thermosetting polymer composite using montmorillonite directly depends on the degree of dispersion, the uniform distribution of montmorillonite in the volume of the composite. Improving the properties of the composite also depends on the chemical reaction of the epoxy resin molecules occurring on the large surface area of the montmorillonite particles.

The greatest change in the rheological and physicomechanical properties of a thermosetting polymer composite and the effectiveness of using montmorillonite particles will be achieved with a uniform distribution of the latter in the volume of the polymer composite with the largest surface area of montmorillonite particles participating in a chemical reaction with epoxy resin molecules.

Morphological studies were carried out to determine the degree of dispersion of montmorillonite particles in the polymer composite using a transmission electron microscope. The distribution of montmorillonite particles in the polymer composite is shown in Figure 2.

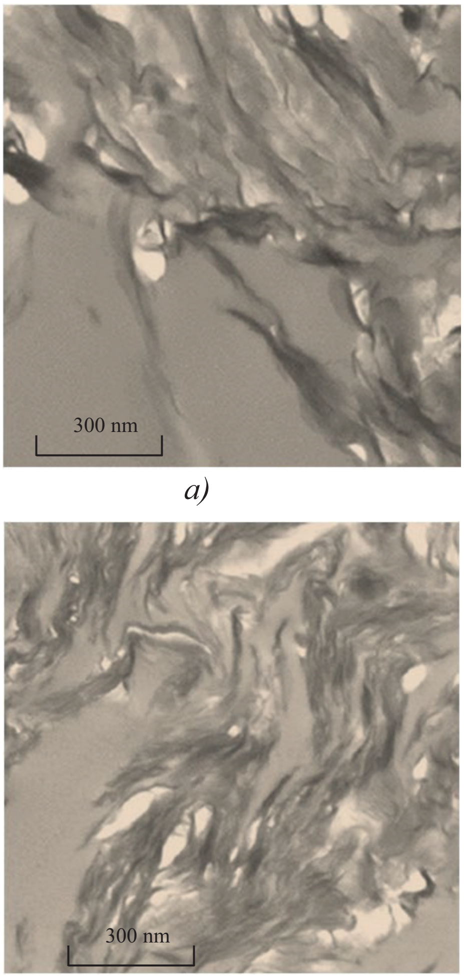

Figure 2. Polymer composite with montmorillonite particles

The uneven distribution of montmorillonite particles predominated in all samples. Apparently, this is due to the curing conditions of the polymer composite and insufficient dispersion time.

Experimental viscosity versus shear rate curves for uncured pure epoxy resin and resin modified with montmorillonite particles at room temperature are shown in Figure 3.

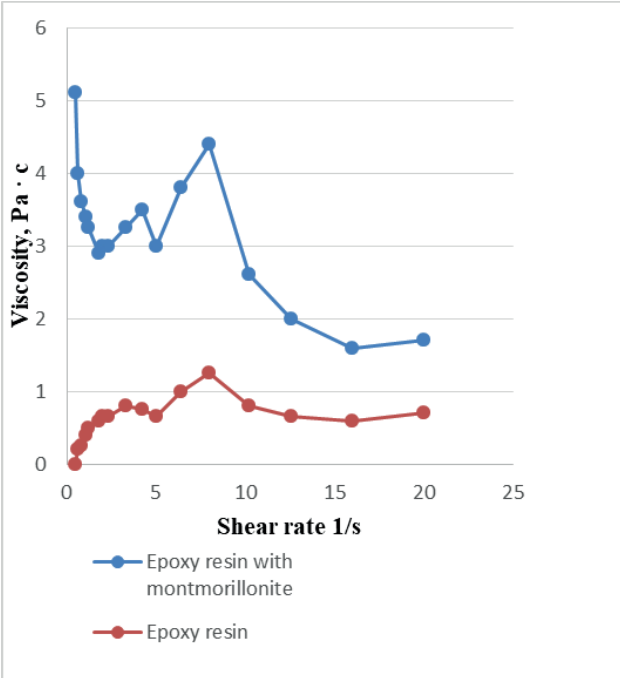

Figure 3. Dependence of viscosity on shear rate

At low shear rates, the effect of montmorillonite on the properties of the composite is very noticeable. The interaction of the montmorillonite particles with the epoxy resin significantly influenced the initial viscosity response to the applied shear stress. These interactions, combined with interactions between montmorillonite particles within the epoxy resin in response to applied stress, resulted in higher viscosity values at low shear rates (0–5 s^{-1}). The low shear rate further allowed epoxy molecules to leak between the layers of montmorillonite platelets, causing swelling and increasing the distance between them. As the shear rate increased from 8 to 20 s^{-1}, the percolation time of montmorillonite decreased, which affected the decrease in viscosity. When the shear rate increases above 20 s^{-1}, no significant changes in viscosity occur;

a monotonic line is observed in both samples. This behavior may have been due to insufficient time for the epoxy molecules to move further into the montmorillonite layers to form a mesoscopic mixture in response to increasing shear rates. Identical rheological properties at different viscosities demonstrate the dependence of viscosity on “particle–particle” and “particle–host interactions”, as studies have shown [5, 10–14]. In general, the viscosity of the montmorillonite sample showed higher values throughout the study compared to pure epoxy resin. This indicates an anisotropic microstructural behavior in which epoxy molecules move into the interlayer space of the montmorillonite flakes and cause them to swell. It can be assumed that with an increase in the dispersion time of the composite, the epoxy resin molecules will penetrate more deeply between the montmorillonite flakes, with its subsequent swelling, affecting the viscosity and final properties of the composite. By the end of the study, the viscosity of the composite, compared to pure epoxy resin, increased by 170%. Montmorillonite significantly affects the viscosity of the base polymer, which can affect processability and cure, leading to changes in rheological and physical-mechanical properties [15–18].

The flexural test results for epoxy resin and epoxy-montmorillonite composite specimens are shown in Table 1. The cast pure epoxy resin has a flexural modulus of 2.8 GPa and a flexural strength of 83.0 MPa.

The composite containing 2 wt% montmorillonite showed a flexural modulus of 4.7 GPa and a flexural strength of 82.0 MPa. The elastic modulus of the composite increased by 25% compared to pure epoxy resin. The flexural strength of a composite with montmorillonite compared to pure epoxy resin is not significantly different. Thus, montmorillonite significantly affects the elastic modulus of the composite, which can have a significant impact on its technological and operational properties [19, 20].

Table 1. The modulus of elasticity and bending strength of cast and layered samples

Name of material	Elastic modulus, GPa	Flexural strength, MPa
Epoxy resin	2.8	83.0
Epoxy resin with montmorillonite	3.5	81.0

4. CONCLUSION

The work investigated the influence of montmorillonite on the rheological and physical-mechanical properties of composites of two compositions - two-component (from epoxy resin and hardener) and three-component (from epoxy resin, hardener and montmorillonite).

The scaly, branched surface of montmorillonite has a significant influence on the gelation and curing time of the composite. More research is needed on composites with different percentages of montmorillonite to obtain predictable properties of the final composites.

The results of the study showed that montmorillonite increases the viscosity of the composite by 170%, which affects its technological, rheological and physical-mechanical properties. The elastic modulus of the composite increased by 25% compared to pure epoxy resin. Montmorillonite significantly affects the elastic modulus of the composite, which can change its technological and operational properties. The flexural strength of a composite with montmorillonite compared to pure epoxy resin does not differ significantly.

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