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Preparation and Characterization of Polymer Composite Materials Containing Magnetite

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Abstract—In this work, magnetite nanoparticles are introduced into a polyethylene melt at the stage of a viscous-flow state by standard methods of polymer processing (extrusion), which makes it possible to obtain a nanocomposite with a uniform nanofiller distribution. The phase composition and structure of the nanocomposite were confirmed by XRF, electron microscopy, and IR spectroscopy. It is shown that the preparation of Fe_3O_4/LLDPE nanocomposites is not complicated by the appearance of unidentified phases and changes in the structure of the polymer matrix.

Keywords—Composites, polymers, nanoparticles, fillers.

I. Introduction

FUNCTIONAL nanocomposites with improved physical properties allow for a variety of applications (e.g. biomedicine, micro-optics, electronics, energy conversion or storage). In most cases, the change in the expected function is correlated with the loading of the filler [1]-[15]. The resulting composite flow behavior limits primarily huge solid loads and hence property adjustments due to molding or molding constraints. Therefore, a detailed description of the properties of the composite flow prior to shaping requires data on the shear rate and temperature dependence, as well as oscillating rheological studies [16]-[21]. In the case of nanoscale fillers, specific surface area and the resulting huge polymer-filler interfacial layer dominate rheological behavior.

Magnetic nanoparticles and nanocomposites have generated significant scientific and technological interest due to their potential applications in biomedicine, information technology, magnetic resonance imaging, catalysis, telecommunications, and environmental restoration [1]-[14].

Magnetic nanocomposites usually contain magnetic nanoparticles embedded in a non-magnetic or magnetic matrix. However, magnetic nanoparticles dispersed in composites usually have a strong tendency to form agglomerates to reduce the energy associated with the high surface area to volume ratio of nanosized particles [15-24]. To avoid aggregation of magnetic nanoparticles, protection strategies have been developed to chemically stabilize unprotected magnetic nanoparticles by grafting or coating with organic species, including surfactants or polymers, or coating with an inorganic layer such as silicon dioxide or carbon. Combining these functionalized magnetic nanoparticles in a polymer or other matrices to develop magnetic nanocomposite materials has proven to be more efficient [25]-[32]. There are mainly four types of magnetic nanocomposites, i.e. inorganic core nanocomposites, self-assembled nanocomposites, silica-based magnetic nanocomposites, and organic-inorganic nanocomposites [33]-[73]. Among them, organic inorganic magnetic nanocomposites have become more interesting due to the combination of the unique properties of the organic and inorganic components in one material. Hybrid organic inorganic magnetic nanocomposite materials can be obtained in situ, ex situ, microwave exposure, coprecipitation, melt mixing, ceramic glass treatment, and plasma polymerization methods.

Modern composites have not only a wide range of physical and mechanical properties, but are also capable of directionally changing them, for example, increasing fracture toughness, regulating rigidity, strength, and other properties. These possibilities are expanded when fibers of different nature and geometry are used in composites, i.e., when creating hybrid composites. In addition, these materials are characterized by the appearance of a synergistic effect

(coordinated joint action of several factors in one direction).

The properties of the interface or interfacial zone, first of all, the adhesive interaction between the fiber and the matrix, determine the level of properties of composites and their retention during operation. Local stresses in the composite reach their maximum values just near or directly at the interface, where material destruction usually begins. The interface must have certain properties to ensure efficient transfer of the mechanical load from the matrix to the fiber. The adhesion bond at the interface should not be destroyed under the action of thermal and shrinkage stresses arising from the difference in the temperature coefficients of linear expansion of the matrix and fiber or as a result of chemical shrinkage of the binder during its curing.

Magnetic nanoparticles can act as a new class of non-toxic and effective flame retardants. Fe_3O_4 enhanced both thermal stability and flame retardant properties of polyvinyl alcohol. Nanoparticles were synthesized by a simple precipitation reaction without using an inert atmosphere at room temperature. The nanoparticles exhibited ferromagnetic behavior at room temperature. To obtain a magnetic nanocomposite, Fe_3O_4 nanoparticles were added to polyvinyl alcohol. Dispersed nanoparticles play the role of a magnetic barrier layer, which slows down the volatilization of the product and prevents the penetration of oxygen into the sample during polymer decomposition.

The effectiveness of magnetic nanoparticles for specific biomedical applications depends on the magnetic properties of the particles, their size, and most importantly, the surface chemistry [1]-[9]. Of particular importance in the biomedical application of colloidal magnetic systems is their stability, which depends on the effective stabilization of nanoparticles, which would ensure the presence of individual nanoparticles rather than agglomerates in solutions, while not significantly affecting the magnetic properties of the material and preserving the particle surface suitable for further functionalization [10]-[18].

When creating nanocomposites, the key tasks are the development of efficient, reliable, and affordable production technologies for mass production, which make it possible to obtain materials with stable characteristics. The hand lay technique, also called wet lay, is the simplest and most widely used process for producing flat reinforced composites. The process consists of laying layers of a polymer in successive layering using an epoxy matrix. Wet-laying is a molding process that combines layers of reinforced carbon fiber with epoxy to create a high-quality laminate. Before starting the installation process, you must prepare the appropriate form. This preparation consists of cleaning the table and applying a release agent to the surface. The manual laying process can be divided into four main steps: mold preparation, epoxy coating, laying and curing. Form preparation is one of the most important steps in the installation process. This process requires dry reinforcement layers and the application of a wet epoxy matrix. They are connected together - reinforcing material, impregnated with a matrix

Nanoparticles, even with a very low volumetric content (less than 1%), are contained in such a fragment in a very large amount, and it is impossible to model their effect at this scale level. For example, a cubic fragment of a 1 μm matrix contains more than thousand nanoparticles for a given volumetric content. Therefore, in particular, the nano-modified binder is white, while the usual binder is yellow. To model such materials, it is necessary to resort to multiscale approaches and to carry out a consistent determination of effective properties at various scale levels. This task is greatly simplified if the properties of the nanomodified matrix are known from experiments. In particular, it is known that its Young's modulus is 2.5 GPa. The missing characteristic is Poisson's ratio, which can be approximately taken unchanged, or estimated on the basis of analytical calculations using the found value of the "effective" volumetric content of the filler, which was done. Further, it suffices to numerically solve the averaging problem on a representative fragment containing only nanoparticles.

At the moment, there are different methods for obtaining composite materials, but in the entire spectrum of literature, there is not enough research data on these materials. The aim of this work is to obtain a polymer nanocomposite based on linear low density polyethylene and magnetite nanoparticles on an extruder. At the moment, physical and the mechanical properties of such a composite have not been sufficiently studied, and the chemical ratio of substances in this material and the nature of the interaction of the filler of the composite material on the structure of the nanocomposite material have not been fully investigated. In addition, composites based on linear low density polyethylene are little known. The relevance of this work can also be justified by the fact that nanocomposite materials using magnetite nanoparticles are not enough to fully study their characteristics and the possibility of using them in medicine, as well as to study how the method of obtaining a nanocomposite affects its properties and structure [22]-[38].

The choice of just such a polymeric material is due to the fact that ultra-high molecular weight polyethylene is usually used for medical purposes, but since it is difficult to process under laboratory conditions, it is possible to replace such polyethylene with linear low density polyethylene. This substitution can be argued by the fact that linear polyethylene is the most optimal for work in laboratory conditions: it is much easier to obtain a nanocomposite on an extruder, and linear low-density polyethylene and ultra-high molecular weight polyethylene exhibit the same properties. In addition, linear polyethylene is biocompatible, which is further evidence of the possibility of its use in this work.

One of the defining factors that characterize the properties of polymer nanocomposites is the high proportion of the interface between the polymer matrix and the filler. The microstructure, namely the dispersed characteristics of nanoparticles, directly affect the interface and, thus, the

macroscopic properties of nanocomposites [39]-[67]. Therefore, the study of the dispersed characteristics of nanoparticles, the possibility of controlling their sizes and homogeneous distribution in the matrix space is a key task in the technology of metal-polymer nanocomposites. The fulfillment of this task requires the use of a certain set of studies, in particular, analysis of the phase composition, microstructure, magnetic and physical-mechanical properties, and IR spectroscopy.

II. Synthesis of Nanocomposites

A large number of works are underway to study the use of magnetic nanoparticles in various fields, including industry and medicine. Most of the applied magnetic nanoparticles are iron oxide nanoparticles, namely magnetite Fe_3O_4. Among the methods for obtaining magnetite nanoparticles, liquid-phase methods are mainly used, which are much easier to perform and require less energy and economic costs. Among them, the most common method: the method of coprecipitation of iron salts (II, III). The advantages of the method include the rapidity of synthesis due to the rapid nucleation of nanoparticles, the ease of synthesis due to one-stage mixing of salts in an alkaline medium, a high yield of the target product due to the complete conversion of the starting components, as well as the possibility of differentiation of the morphology and composition of nanoparticles at the synthesis stage [25]-[41]. However, despite the indicated advantages, this method also has a number of disadvantages. The main problems in the rapid nucleation of nanoparticles are the problem of separation of nucleation and growth of nanoparticles, as well as the problem of the need to control after nucleation over the growth processes of monodisperse nanoparticles, which are reflected in the uniformity of nanoparticle size distribution.

Highly dispersed nanoparticles iron oxide (II, III) Highly dispersed nanoparticles iron oxide (II, III) obtained by the Elmore reaction - rapid neutralization with constant stirring of chloride salts of ferrous and ferric iron with an excess of aqueous ammonia solution in the temperature range 20-60 °C.

$2FeCl_3 + FeCl_2 + 8NH_4OH = Fe_3O_4 + 8NH_4Cl + 4H_2O$

According to X-ray phase analysis, the product of the chemical deposition reaction is magnetite Fe_3O_4 (Table 1).

Table 1. XRF data of magnetite nanoparticles.

2Q/deg.	d/Å	I	I, rel. (%)	(hkl)
28.75	4.614	22	53	
45.65	2.953	13	31	220
53.80	2.532	42	100	311
66.60	2.086	11	25	400
90.95	1.607	11	25	511
101.80	1.476	26	62	440

Broadening of lines and a small number of intense reflections in the diffraction patterns of the samples are characteristic of nanoscale systems. The average size of magnetite nanocrystallites is 15 nm in accordance with the calculation of coherent scattering regions using the Debye-Scherrer formula, which agrees with the data of electron microscopy.

The matrix can be a thermosetting polymer - epoxy resin, which has already found many applications: from structural composites to adhesives and surface coatings. Epoxy resins already have a number of unique qualities among polymers: no shrinkage during curing, high adhesion to various substrates, good dielectric and other valuable properties [36]-[44]. Nanocomposites using thermoplastic polymers are well known and studied to improve mechanical, electrical, thermal and insulating properties. However, nanocomposites using thermosetting polymers have not been studied as widely, especially using TiO_2.

Nanoparticles are usually introduced into the polymer matrix using various methods. Dispersion processes are necessary in order to transfer nanoparticles from an agglomerated state to a uniformly dispersed state [26]-[34]. The most popular are live streaming with the use of chemical methods and the use of high shear forces in the process of mechanical dispersion of the powder. Chemical methods are capable of generating individual and non-agglomerated nanoparticles within a thermosetting or thermoplastic polymer. For mechanical dispersion, ultrasonic treatment is often used, which also improves the dispersion state of nanoparticles.

Methods for the synthesis of nanocomposite materials (or nanocomposites) are divided into two main large approaches: in situ and ex situ. The creation of nanocomposites by the in situ method makes it possible to obtain simultaneously (in one stage) both a matrix and nanoparticles, obtaining a nanocomposite at the output. The advantage of the method is that it prevents particle agglomeration while maintaining a good spatial distribution in the polymer matrix. This is due to the disjoining forces caused by the molecules of polymer matrix. The main disadvantage of the method is that all the products including side products of the synthesis of nanoparticles remain in the nanocomposite, which can deteriorate the quality and purity of the obtained material.

When using the ex situ method, each stage of nanocomposite creation is brought into a separate process: from the synthesis of nanoparticles to the preparation of a nanocomposite. This method is more energy and labor intensive compared to in situ, and requires much more time. Also, special attention should be paid to the dispersion of the nanocomponent in the matrix, since during long-term storage nanoparticles are collected into larger ones and they must be dispersed by various methods, for example, by ultrasonic treatment [1]-[8]. The ex situ synthesis method is more suitable for large scale industrial applications than the in situ method.

The ex situ method, despite some drawbacks, is used more often than the more technologically advanced in situ method. There are several reasons of it. First of all, this is due to the simplicity of the approach based on the fact that in such

reactions the synthesis of nanoparticles is not complicated by additional reactions with the polymer matrix, the resulting nanocomposite will not contain by-products associated with the formation of nanoparticles in the polymer matrix, etc.

III. Studying the Properties of Nanocomposites

LLDPE was used as a polymer matrix, matrix-stabilized iron (III, II) oxide nanoparticles served as a filler. The crystalline phases of the nanocomposites were identified using X-ray phase analysis. The diffractogram shown in Fig. 1, there are diffraction peaks corresponding to interplanar the distances of the crystal lattices of PE and magnetite.

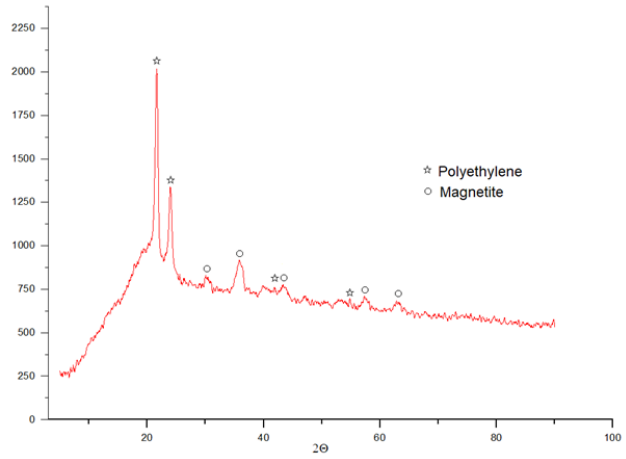

Fig. 1 Diffraction pattern of the obtained Fe_3O_4/ LLDPE nanocomposite.

Broadened peaks indicate at the presence of very small particles. No other diffraction peaks are observed on the diffraction pattern, which indicates the occurrence of the dispersion process in the polymer matrix without additional phase transformations.

Table 2. XRF data for the 10Fe3O4/ LLDPE nanocomposite.

2θ/ deg	d	Iimp/s	Irel (%)	Phase
21,65	4,105	1007	100	LLDPE
23,95	3,715	477	43	LLDPE
30,05	2,974	74	7	Fe_3O_4
35,85	2,505	193	17	Fe_3O_4
41,95	2,154	77	7	LLDPE
43,35	2,087	94	9	Fe_3O_4
54,85	1,674	59	5	LLDPE
57,30	1,608	71	6	Fe_3O_4
62,85	1,479	74	7	Fe_3O_4

To determine the structure of the obtained nanocomposite, the method of IR spectroscopy was used. IR spectroscopy allows first to get evidences of the interaction between nanoparticles and polymer matrix and, second, get the information about the structure of the resulting nanocomposite. For the details of the IR characterization of the polymer nanocomposites, it is referred to the literature [48]-[50]. Fig. 2 gives the overview of IR spectra of LLDPE and the resulting nanocomposite is shown. As can be seen from the low-frequency region of the spectrum (Fig. 3), the absorption bands of the Fe-O bond in the IR spectrum of pure magnetite are shifted in comparison with the corresponding stretching vibrations of Fe-O in the nanocomposite, which indicates that during the mixing of the polymer and the nanofiller between the functional groups (mainly OH groups) on the surface of nanoparticles and the polyethylene macromolecule, a chemical interaction occurs.

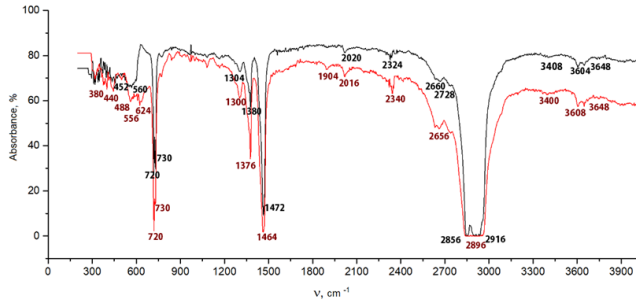

Fig. 2. ir spectrum of the obtained nanocomposite Fe3O4/LLDPE and LLDPE.

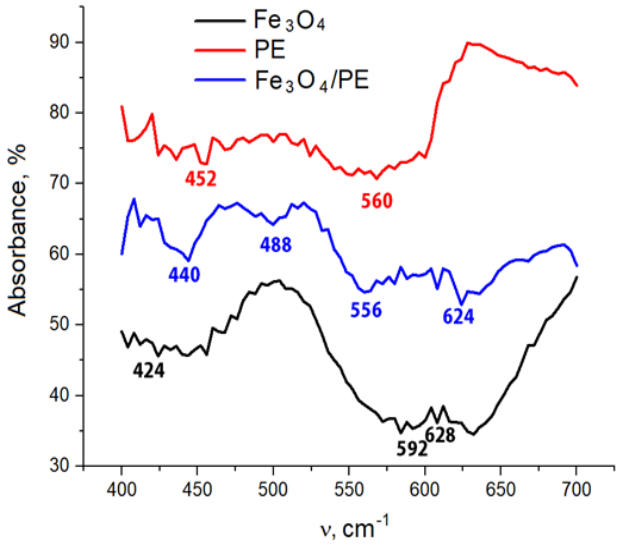

Fig. 3 ir spectrum of the nanocomposite Fe_3O_4/LLDPE, LLDPE and Fe_3O_4.

It can be seen from the survey spectrum (see Fig. 2) that all characteristic LLDPE absorption bands are present in the composite. The spectra contain intense absorption bands of stretching vibrations (n) >C-H at frequencies of 2856-3648 cm^{-1} and 2896-3648 cm^{-1}, respectively, and a series of bands corresponding to bending vibrations dCH_2: 1472, 1464, 1376, 730 and 720 cm^{-1}. Although the introduction of nanoparticles does not have a significant effect on the LLDPE structure, nevertheless, certain changes in the region of the amorphous and crystalline phases take place, as can be seen from Fig. 3, in particular, the ratio of the peaks changes, this is noticeable at 720/730 cm^{-1} and 1464/1472 cm^{-1}, doublets, which indicates some amorphization of the LLDPE structure.

IV. Conclusion

As a result of this study, it was shown that the introduction of magnetite nanoparticles into the LLDPE melt at the stage of the viscous-flow state by standard methods of polymer processing (by extrusion) makes it possible to obtain a nanocomposite with a uniform distribution of the nanofiller. The phase composition and structure of the nanocomposite were confirmed by XRF, electron microscopy, and IR spectroscopy. The preparation of Fe_3O_4/ LLDPE nanocomposites is not complicated by the appearance of unidentified phases and changes in the structure of the polymer matrix. This opens new prospective for tailored fabrication of polymer nanocomposites with desired structure and properties. Further research can be aimed toward the development of nanocomposites with advanced mechanical properties.

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