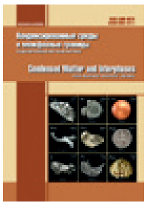

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Preparation of composite micro-nanofibers based on nano-sized magnetite by electrospinning

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Abstract

Composite materials with magnetic fillers play an important role in a number of industries, from functional coatings in electronics to electromagnetic wave absorption and microwave-shielding materials. An important feature is the selection of a magnetic nano-sized filler that does not cause increased degradation of the polymer binder, and the selection of a polymer that ensures the weather resistance of the nanocomposite material. In this study, composite samples of micro- and nanofibers based on fabricated particles of nanosized magnetite (Fe_3O_4) as a cheap electromagnetic wave absorption material were investigated.

Magnetic polymer-dielectric fibers polystyrene-Fe_3O_4 were obtained by electrospinning. The X-ray diffraction analysis showed that the synthesized Fe_3O_4 nanoparticles have a cubic space group structure *Fd3m* with crystal lattice parameter a = 8.422±0.026 Å. The analysis of the ferromagnetic resonance spectrum showed the ferromagnetic nature of the obtained magnetite nanoparticles. It has been shown that during the production of composite fibers by electrospinning, a dispersion of nano-sized magnetite powder can be included in the spinning solution, which, as a result of the electrospinning process, allows obtaining magnetic composite micro- and nanofibers. The average size of the included magnetite particles was 15±3 nm.

The resulting non-woven magnetic material is predominantly composed of two types of fibers with an average diameter of 680±280 nm and larger associated fibers with a diameter of 1500±300 nm. Based on a certain frequency dependence of losses upon reflection *RL* in the frequency range 15 MHz – 7.0 GHz, the synthesized fibrous material can be considered to be an effective electromagnetic wave absorption material.

Keywords: Nano-sized magnetite, Electrospinning, Composite fiber, Structural characteristics, Magnetic materials, Radio absorption

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1. Introduction

The diverse applications of magnetic nano-sized particles or materials are widely explored by scientists and researchers around the world for various industrial, engineering, structural, and biomedical applications. This interest is due to the exceptional physical and chemical properties of nanoscale objects, such as large specific surface area, small size, surface functionalization, and magnetism. Magnetic nanoparticles usually consist of pure metals (Fe, Co, Ni), metal alloys (CoPt, FePt) and metal oxides or ferrites [1]. In the last decade, magnetic nanoparticles have gained enormous interest due to their use in specialized areas such as medicine: as a carriers in targeted drug delivery [2, 3], cancer theranostics [4, 5], biosensors [6, 7], contrast agents for magnetic resonance imaging [8-10]; electromagnetic wave absorption and radio-shielding materials of electromagnetic radiation [11–14], fillers of composite materials for FDM printing [15, 16], production of magnetorheological fluids for systems of controlled hydraulic automation devices, in which such particles are a component of the complex dispersed phase [17], magnetic ink [18], etc. Magnetic nanoparticles of magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3) are of particular interest [19].

Nanoscale Fe_3O_4 is a cheap, effective magnetic, electromagnetic wave absorption and radio-shielding nanomaterial with a combination of unique magnetic, optical and photocatalytic properties [20–23]. Composite fibrous materials based on Fe_3O_4 are of particular interest due to the development of new materials with magnetic and conductive properties [24–26]. In [27], the authors obtained composite fibers by electrospinning based on a polyacrylonitrile/DMSO fiber-forming system with the inclusion of magnetite nanoparticles, in [28] the authors studied the effect of the concentration of magnetite nanoparticles in a colloidal solution on the process of their loading into calcium carbonate microparticles grown on polycaprolactone fibers; in [29] the authors obtained composite fibers by electrospinning based on the polyvinylpyrrolidone/water fiber-forming system containing magnetite nanoparticles. Composite fibrous materials based on nanosized magnetite can be used both for effective electromagnetic microwave absorption and for ensuring electromagnetic compatibility of radio-electronic equipment at ultrahigh frequencies [28–35]. From practical experience it is known that ultrafine Fe_3O_4 nanoparticles, which have strong catalytic properties, cause increased degradation of polymer binders, leading to poorly predictable changes in the properties of electromagnetic wave absorption and radio-shielding nanocomposite materials based on Fe_3O_4 on time and temperature. In addition, an important problem is the provision of the protection of nano-sized magnetic filler in a composite material from chemical leaching by precipitation.

A solution to this problem may be the creation of fibrous composite materials in which Fe_3O_4 nanoparticles are “encapsulated” in a weather-resistant polymer binder (polystyrene or acrylate-styrene copolymer) using fiber electrospinning technology. This approach fundamentally allows reducing the temporary degradation of the operational properties of electromagnetic wave absorption and radio-shielding nanocomposite materials based on Fe_3O_4 under atmospheric conditions.

The purpose of this study was the creation and investigation of the characteristics of a fibrous composite material based on nano-sized magnetite in a polystyrene matrix using electrospinning.

2. Experimental

A sample of nanosized magnetite was obtained using the ammonium hydroxide method. As iron salts $FeSO_4 \cdot 7H_2O$ (chemically pure) and $Fe_2(SO_4)_3$ (reagent grade) which were dissolved in double-distilled water at a concentration of 0.05 M were used. Next, the salt solution in the required proportions was heated on a laboratory electric stove with a power stirrer to a temperature of 65 °C and the calculated amount of a 25% ammonium hydroxide solution ($NH_3 \cdot H_2O$ with density ρ = 0.9070 g/ml) with a 1% excess was poured drop by drop with constant stirring at a slow rate upon reaching pH = 8.5. The formation of magnetite took place in accordance with the ionic equation:

$$Fe^{2+} + 2Fe^{3+} + 8OH^- \rightarrow Fe_3O_4\downarrow + 4H_2O.$$

After pouring in the ammonia precipitant, the solution was kept for 20 min at a temperature of 65 °C for the formation of the magnetite nanoparticles. The resulting magnetite nanoparticles were separated from the resulting solution using magnetic decantation with a permanent magnet. The powder was thoroughly washed four times with bidistilled water. The resulting black wet powder was dried in air for 3-4 days. The dried magnetite powder was then ground in a ceramic mortar until homogeneity was achieved.

The microstructure of the synthesized magnetite powder was analyzed using a JEOL JSM-7500F electron scanning microscope. The microstructure was studied using the secondary electron registration mode. The advantage of using the secondary electron registration mode is the ability to study the surface morphology, taking into account the dependence of the contrast on the relief [36]. Elemental analysis was performed using an Inca X Sight EDX Spectrometer X-ray energy dispersive microanalysis attachment. The X-ray spectral analysis method allows both qualitative and quantitative analysis of samples without compromising their integrity [37]. Laser granulometric analysis was performed using laser particle size analyzer Analysette 22 of JEOL JES-FA300X ESR/FMR X-ray spectrometer. X-ray phase analysis of a sample of nanosized magnetite powder was carried out using a powder diffractometer D2 Phaser. The sample was examined at room temperature in the 2θ angle range from 10° to 70° with a scanning step of 0.02°.

The synthesis of individual and composite polystyrene nano- and microfibers was carried out using an independently developed installation for needle-free electrospinning. Emulsion-type polystyrene was dissolved in toluene (chemically pure) until the mass fraction of polystyrene in the solution reached 18%. To obtain nanocomposite fibers based on nanomagnetite and polystyrene, a concentrated aqueous dispersion of purified magnetite nanoparticles was used. Magnetite nanoparticles were removed from the aqueous dispersion by magnetic decantation using a permanent magnet. The solution for electrospinning fibers was prepared in order to obtain a composite fiber with a mass content of nanosized magnetite of 25%. The electroforming process was carried out at a electric potential difference between the electrodes of 18 kV and an interelectrode distance of 10 cm.

For the determination of the electromagnetic wave absorption properties of the fabricated fibrous composite based on polystyrene fiber with nano-sized magnetite, the reflection loss characteristics of its compressed layer with the thickness of 2.54 mm were measured in a 10-cm HP-11566A coaxial cell with toroid dimensions of 7.0 × 3.05 mm. A KC901V Deepace vector network analyzer was used in the operating frequency range from 15 MHz to 7.0 GHz. Losses upon reflection *RL* for the nanocomposite was determined experimentally by measuring the complex transmission coefficient S_{11} in a short-circuited line.

3. Results and discussion

Based on the data obtained by processing photographs of the microstructure at high

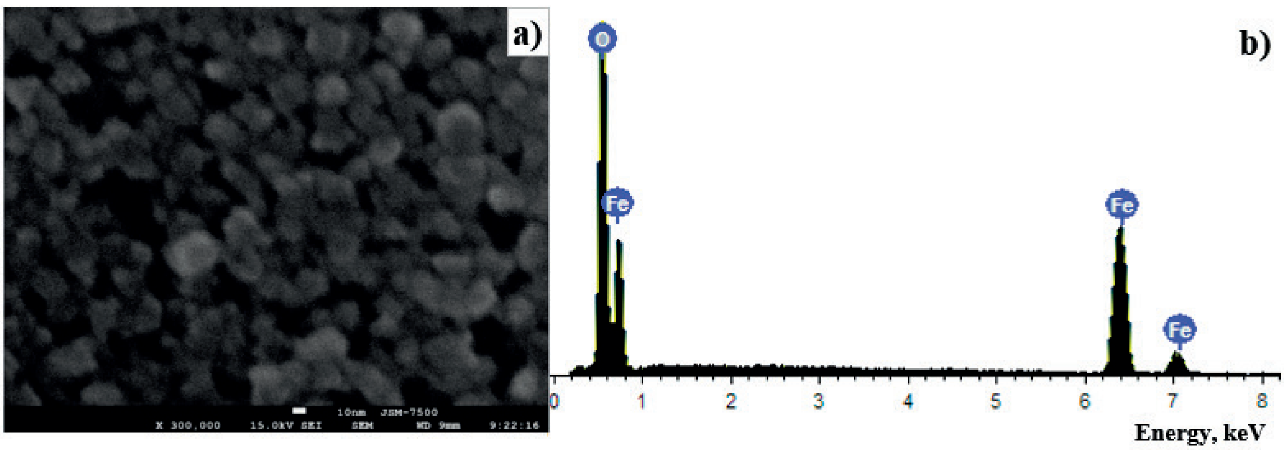

Fig. 1. Photograph of nanoparticles (a) and EDA spectrum (b) of the resulting nano-sized magnetite powder

resolution (Fig. 1), the size of magnetite nanoparticles in the synthesized sample was 15 ± 3 nm. Our results are consistent with the results of [38], in which a similar synthesis method was used, but with iron chlorides and low temperature, short exposure of the resulting nanomagnetite in the mother solution, and are in good agreement with the data of [39]. In this case, the synthesis product, according to energy-dispersive microanalysis, in terms of the percentage of Fe and O atoms corresponded to the expected composition of Fe_3O_4 without impurities in significant quantities.

Laser granulometric analysis of the synthesized magnetite powder showed (Fig. 2a) significant agglomeration of particles in it; therefore, an aqueous dispersion of purified magnetite nanoparticles was used to obtain nanocomposite fibers based on magnetite and polystyrene. Before adding the magnetite dispersion to the polymer molding solution, the nanoparticles were dispersed using an AG SONIC TC-50 ultrasonic bath for 20 min at room temperature.

FMR spectrum of synthesized nanosized Fe_3O_4, magnetite powder is shown in Fig. 2b. Based on the shape of the FMR spectrum, the studied sample of Fe_3O_4 nano-sized magnetite powder is a typical ferromagnetic material with a highly symmetrical nanoparticle shape.

The powder X-ray diffraction pattern of the studied sample of synthesized nanosized magnetite is shown in Fig. 3. Based on X-ray diffraction analysis, it was found that Fe_3O_4 nanopowder has a typical cubic space group structure *Fd*3*m* with crystal lattice parameter *a* = 8.422±0.026 Å and an average Fe-O distance of 2.55 Å, which correlates well with known literature data for Fe_3O_4 (*a* = 8.407-8.414 Å [40], *a* = 8.40-8.42 Å [41], *a* = 8.397 Å [42] or JCPDS19–0629 *a* = 8.396 Å [43]). This confirms that the sample was composed of Fe_3O_4 without possible traces of γ-Fe_2O_3.

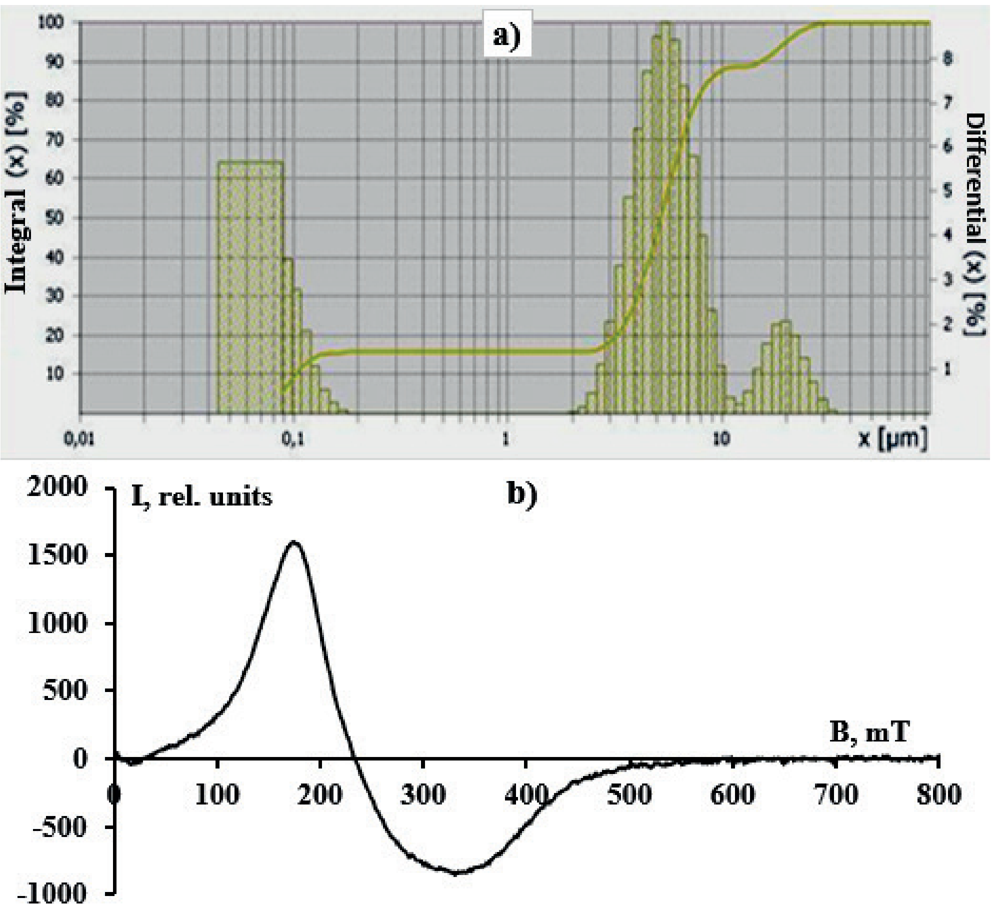

Fig. 2. Laser granulometric analysis (a) and FMR spectrum (b) of the resulting nano-sized magnetite powder

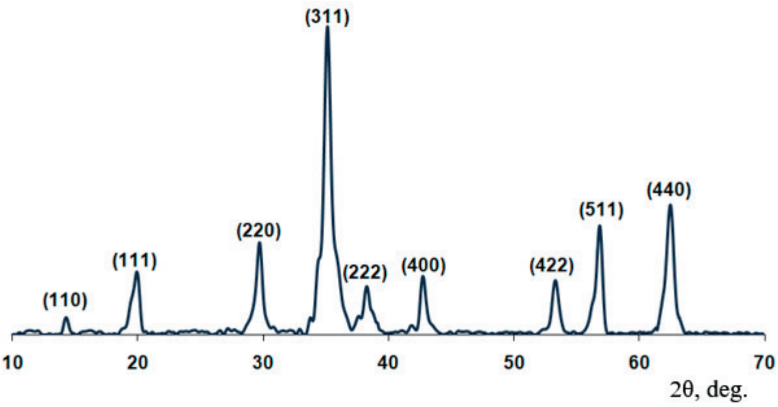

Fig. 3. Powder diffraction pattern of a sample of nano-sized magnetite

Average size of coherent scattering regions (CSR) - *D* for a sample of nano-sized magnetite was calculated based on X-ray diffraction data for all peaks using the Scherrer formula:

$$D = \frac{k \times \lambda}{\beta \times \cos\theta}$$

where $k = 0.9$ – for spherical particles; λ – wavelength of the x-ray radiation used ($\lambda = 0.15405$ nm), nm; θ – Bragg angle, rad; β – half-width of integral peaks at half-maximum, rad.

The calculated CSR value for magnetite crystallites using the Scherrer method for the main diffraction peak was $D = 15.1$ nm, which was consistent with the results of electron microscopy, and according to all observed diffraction peaks $D = 19.5 \pm 6$ nm. Our results are in good agreement with the data of [44], in which the size of synthesized magnetite nanoparticles based on electron microscopy data of 15 nm was lower than the size calculated based on powder X-ray diffractometry data of 19.4 nm.

Calculation of CSR sizes and microstresses for a sample of the studied Fe_3O_4 nanopowder using the Williamson-Hall method, provided the following results: CSR sizes $D = 17.2$ nm, which agreed with the value obtained using Scherrer's formula, the microstresses value $\varepsilon = 4.6 \cdot 10^{-4}$.

It should be noted that a electrospinning spinneret in the form of a hollow needle is usually used to produce non-woven materials by electrospinning. However, the use of a hollow needle has the following limitations and disadvantages: clogging of the needle channel with a dispersion of filler particles of the spinning solution due to the narrow internal diameter of the hole, which may not allow encapsulation of particles that can improve the properties of the resulting fibers and/or functionalize the resulting non-woven material; limited productivity (up to 0.1 grams per hour), nonlinear scaling [45]; a needle-down spinneret placement can result in droplets forming at the needle tip, which can fall onto the collector, preventing the formation of uniform fibers [46]. To overcome these disadvantages, needleless electrospinning units can be used to produce polymer nano- and microfibers filled with nanoparticles. Needleless electrospinning is the process of producing nanofibers by electrospinning of a polymer solution directly from the exposed surface of a liquid/liquid dispersion of a spinning solution with nanoparticles using various structural elements as a spinning electrode [46], such as a conical wire supported by gravity [47], metal plate [48], rotating cone [49], gear [50], spinneret with a mechanical shift [51], etc. Such structural elements are partially immersed and rotated in the polymer molding solution, resulting in the formation of a thin polymer solution layer on their surface and thus from the surface of the thin polymer layer, multiple cones are formed, which, after applying an electric field, initiate electrospinning. In our unit the fibers were formed from a polymer solution flowing under the influence of gravitational force along a vertically oriented spinning electrode. The forming electrode consisted of a metal rod made of surgical stainless steel with a diameter of 1 mm, on top of which a wire with a diameter of 0.2 mm was wound as a spiral.

The microstructure of the resulting polystyrene micro- and nanofibers according to scanning electron microscopy data are shown in Fig. 4. According to the studies, the average thickness of the obtained polystyrene microfibers was 910 ± 160 nm (Fig. 4a). At the same time, the resulting fibrous material also contained a small fraction of thin nanofibers with a thickness of 89 ± 7 nm (Fig. 4b).

The results of studying the microstructure of the obtained composite polystyrene fibers with included magnetite nanoparticles are shown in Fig. 5. According to studies conducted in the resulting fibrous material, polystyrene-Fe_3O_4 the fraction of submicron fibers with a thickness of 680 ± 280 nm predominated. At the same time, the discussed material also contained a small fraction of large microfibers with a thickness of 1500 ± 300 nm, probably being pairs of submicron fibers. We concluded that the obtained composite fibers based on nanosized magnetite had an average diameter almost 2-3 times higher compared to the results for composite nanofibers based on nanosized magnetite from [8] with a diameter of 200-350 nm and [13] with a diameter of 200-320 nm. This was due to the use of low potential difference of 18 kV in the electrospinning process, compared to the electrospinning process carried out at 30 kV in [8] and at 65 kV in [13].

The frequency dependence of losses upon *RL* reflection for a manufactured fiber composite with nanosized magnetite particles in the frequency range from 15 MHz to 7.0 GHz is shown in Fig. 6. According to the data in Fig. 6, the resulting fibrous nanocomposite material in compressed form had wide-range radio absorption and electromagnetic wave absorption properties in the microwave range acceptable for practical use, taking into account its microporosity and the low

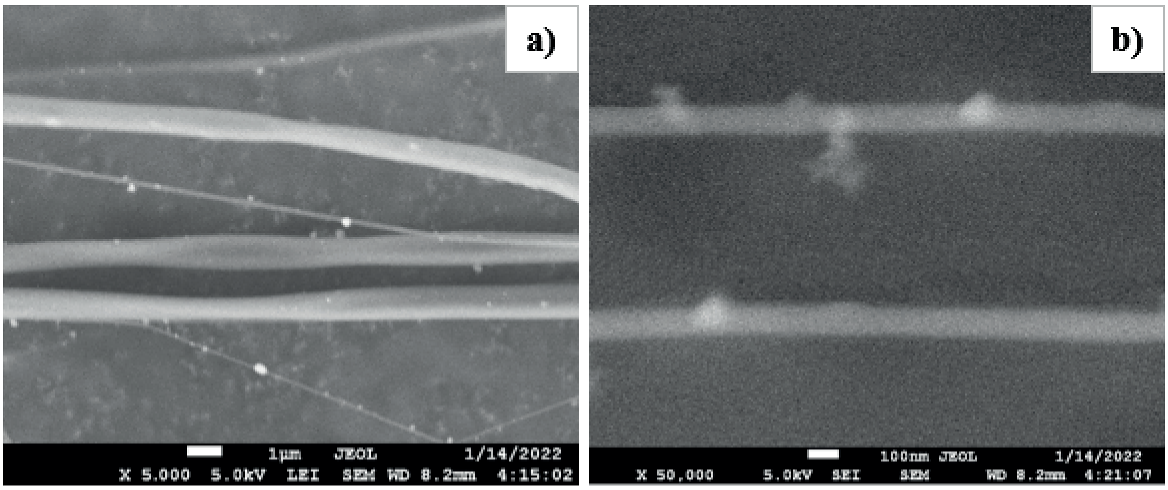

Fig. 4. Structure of polystyrene microfibers obtained at a magnification of 5000x (a) and 50,000x (b)

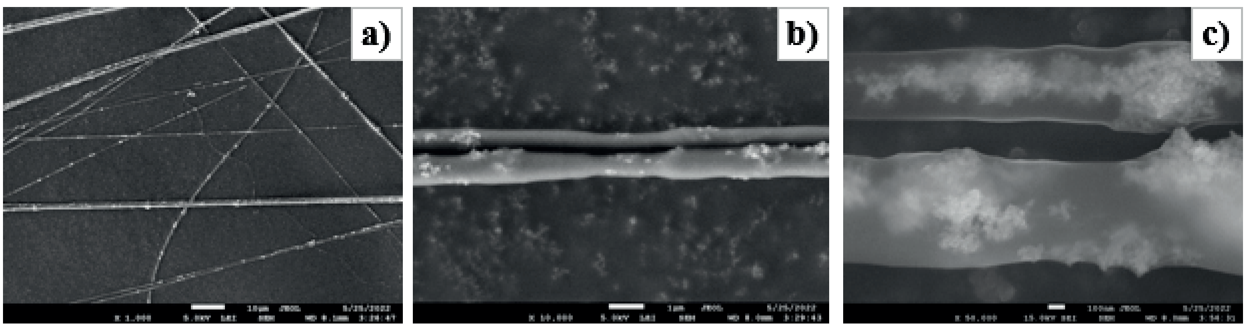

Fig. 5. Photographs of the structure of synthesized composite fibers, obtained at magnifications of 1000 (a), 10,000 (b) and 50,000x (c)

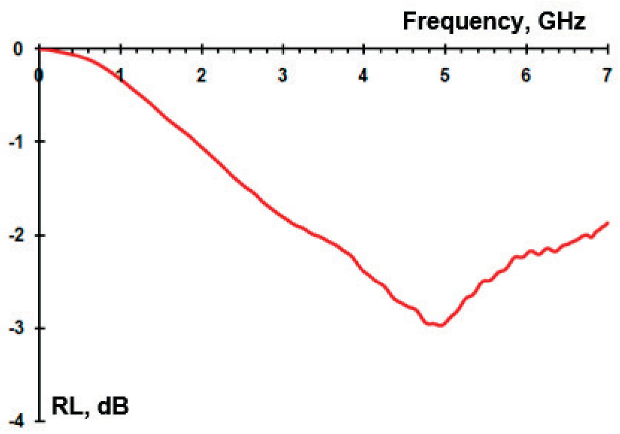

Fig. 6. Frequency dependence of reflection loss *RL* for a fabricated fiber composite based on polystyrene fiber with nanosized magnetite

proportion of magneto-dielectric filler in the form of nano-sized magnetite in it.

Previously published data on the electromagnetic wave absorption properties of composites with different thicknesses and concentrations of magnetite particles are shown in Table 1. The size of magnetite particles used in various studies ranged from 15 nm to 1000 nm. As can be seen from the data in Table 1, the material made of Fe_3O_4 nanoparticles had the highest radio absorption of – 8.2 d, with a diameter of 30 nm in a silicone binder [57], however, it should be noted that the thickness of this sample was 4 mm, and the percentage of magnetite was 30% by weight. Our sample had a microwave absorption of −2.97 dB with a thickness of 2.54 mm and a magnetite concentration of 25% in polystyrene. Taking into account the thickness of the studied materials, the proportion and size of filler particles, and the used polymer binder, we can suggest the using the material of submicron polystyrene fibers with included magnetite nanoparticles, as a cheap non-woven electromagnetic wave absorption material.

4. Conclusions

Thus, we can conclude that the combination of a simple solution method for the synthesis of magnetite nanoparticles without the use of expensive stabilizing polymers or surfactants in combination with the encapsulation technique of Fe_3O_4 nanoparticles into polystyrene submicron fibers during electrospinning allowed to develop elements of the technology for creating fibrous magnetic and electromagnetic wave absorption nanocomposite materials based on magnetic Fe_3O_4 nanoparticles. According to their characteristics, the resulting micro- and nanofibers with nanosized magnetite particles we can suggest the promise of the obtained material for use as a cheap non-woven electromagnetic wave absorption material.

Table 1. Radio absorption properties of various composites based on magnetite particles of various natures

Material	Filler (Fe_3O_4), %	Sample thickness, mm	Reflection loss, dB	Reference
Fe_3O_4 nanoparticles 15 nm in submicron polystyrene fibers	25	2.54	−2.97 at 4.96 GHz	this article
Fe_3O_4 nanoparticles 20–30 nm in submicron polyvinyl chloride fibers	40	2.4	−6.6 at 9.7 GHz	[52]
natural Fe_3O_4 in paraffin	50	5	−5.47 at 7.44 GHz	[53]
cubic Fe_3O_4 nanoparticles 15–20 nm in paraffin	40	5.5	−7.6 at 5.1 GHz	[54]
Fe_3O_4 microspheres 300 nm in paraffin	50	2	−1.0 at 5.6 GHz	[55]
hedgehog-like microspheres Fe_3O_4 500–1000 nm in paraffin	50	5	−4.1 at 8.4 GHz	[56]
Fe_3O_4 nanoparticles 30 nm in silicone polymer	30	4	−8.2 at 6.7 GHz	[57]
Fe_3O_4 microspheres 200–1000 nm in paraffin	20	4	−7.5 at 7.6 GHz	[58]

Author contributions

Yakupov R. P. – synthesis of polystyrene fibers and polystyrene-Fe_3O_4, discussion of the results, c. Buzko V. Yu. – experimental planning, synthesis of Fe_3O_4 dispersion, organization of measurements, analysis of powder diffraction results, analysis of electron microscopy data, discussion of results, writing an article. Ivanin S. N. – planning of the experiment, scanning electron microscopy of samples, measurement of reflection losses, discussion of results, design and editing of the text. Papezhuk M. V. – organization of measurements, discussion of results.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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