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تطوير مواد مركبة تعتمد على أكسيد المغنيسيوم باستخدام تقنية المحلول الغرواني
(سول-جيل)

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ABSTRACT

The study investigated the properties and efficiency of MgO:ZnO, MgO:Y₂O₃, MgO:Fe₂O₃ and MgO:FeZnOF composite materials for filtering oil-contaminated water and for using them as adsorbents for oil waste. The samples were heat-treated in air at 700°C for one hour. The MgO:ZnO composite demonstrated high efficiency in removing oil contaminants from water, achieving 85% removal due to the presence of active centers on the surface of the material that facilitate adsorption. MgO:Y₂O₃ demonstrated stability and a removal rate of 80% due to the improved

porosity provided by yttrium. MgO:Fe₂O₃ achieved 90% removal of oil contaminants due to its magnetic properties, which allows the use of magnetic methods for cleaning. The MgO:FeZnOF composition demonstrated unique adsorption properties, removing up to 88% of oil contaminants. All materials studied demonstrated high efficiency in filtering and adsorbing oil waste.

Keywords: Petroleum products, Water filtration, Composite materials, Magnesium oxide.

تطوير مواد مركبة تعتمد على أكسيد المغنيسيوم باستخدام تقنية المحلول الغرواني (سول-جيل)

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ملخص البحث:

خواصه المغناطيسية، مما يسمح باستخدام الطرق المغناطيسية للتنظيف .

اما التركيبية MgO:FeZnOF فقد أظهرت خصائص امتصاص فريدة، حيث تمكنت من إزالة ما يصل إلى 88% من ملوثات الزيت .

كما أظهرت جميع المواد التي تمت دراستها كفاءة عالية في ترشيح وامتصاص نفايات الزيت حيث تفتح النتائج التي تم الحصول عليها آفاقاً جديدة لاستخدامات هذه التركيبات كمرشحات للمياه من الشوائب للوصول لبثية نظيفة خالية من الملوثات.

الكلمات المفتاحية: المنتجات البترولية، تقنية المياه، المواد المركبة، أكسيد المغنيسيوم

تناولت الدراسة دراسة خصائص وكفاءة المواد المركبة MgO:Y₂O₃ ، MgO:ZnO و MgO:Fe₂O₃ في ترشيح المياه الملوثة بالنفط واستخدامها كمتصات للنفايات النفطية حيث تم معالجة العينات حرارياً في الهواء عند 700 درجة مئوية لمدة ساعة واحدة . لقد أظهر المركب MgO:ZnO كفاءة عالية في إزالة ملوثات النفط من الماء، حيث حقق إزالة بنسبة 85% ويعزي ذلك لوجود مراكز نشطة على سطح المادة تسهل الامتصاص.

كما أظهر المركب MgO:Y₂O₃ ثباتاً ومعدل إزالة بنسبة 80% بسبب المسامية المحسنة التي يوفرها الإيتريوم. أيضاً حقق المركب MgO:Fe₂O₃ إزالة 90% من ملوثات النفط بسبب

I. Introduction

Water is the main resource for life on Earth and plays a key role in maintaining ecosystems, agriculture, industry and everyday life of people. However, with the increase in anthropogenic load and pollution of water bodies, the problem of providing clean water is becoming one of the most pressing environmental challenges of our time [1]. According to the World Health Organization, billions of people around the world face a shortage of safe drinking water, which requires the development of effective technologies for purification and absorption of pollutants [2].

One of the promising areas in the field of water purification is the use of composite materials that can effectively remove a variety of pollutants, including heavy metals, organic compounds and microbial contaminants. Among such materials, magnesium oxide (MgO) attracts special attention, which has unique physicochemical properties, such as high thermal stability, low toxicity and good reactivity, making it an ideal candidate for use in water purification systems [3].

Sol-gel technology is one of the most effective methods for the synthesis of nanostructured materials, providing a high degree of control over their morphology and texture [4]. The essence of the sol-gel process is the hydrolysis and polycondensation of precursors, which leads to the formation of a three-dimensional network structure with high porosity and a large specific surface area [5]. These characteristics contribute to the creation of adsorbents that can effectively capture pollutants from the aquatic environment.

Studies show that MgO-based nanocomposites obtained using sol-gel technology exhibit outstanding adsorption properties. For example, they have demonstrated high efficiency in removing heavy metals such as lead and cadmium, as well as organic pollutants such as phenols and petroleum products [6]. In addition, the high adsorption characteristics of MgO are due to its ability to form active centers on the surface, which facilitates interaction with pollutants [7].

The introduction of various dopants into the MgO structure can significantly improve its adsorption properties. For example, the addition of transition metal oxides can not only increase the strength characteristics of composites, but also improve their catalytic properties, making them even more effective in the water purification process [8]. Studies show that combining MgO with other materials, such as iron or aluminum oxides, can lead to a synergistic effect that enhances the adsorption of various pollutants [9]. In addition to adsorption, MgO also exhibits antibacterial properties, which can be useful in the context of water purification from biological pollutants. Studies show that MgO nanoparticles promote the destruction of bacterial cell membranes and can effectively inhibit their growth [3]. This opens up additional opportunities for the use of MgO-based composites in water purification systems that require not only the removal of chemical pollutants, but also water disinfection. Thus, the development of MgO-based composite materials

using sol-gel technology is an urgent task that can significantly improve the efficiency of water purification. This study aims to investigate the properties and application possibilities of such composites for water absorption and purification, which is of great importance for ecology and public health. The results of this study can contribute to the creation of new, more efficient water purification systems that meet modern requirements for environmental protection and sustainable development.

II. Materials and Methods

i. Materials

All chemicals listed below were procured and utilized in the form of magnesium oxide powder MgO (analytical grade, GOST 4526-75, 98%), zinc nitrate hexahydrate $Zn(NO_3)_2 \times 6H_2O$ (chemically pure, GOST 5106-77, 98%), yttrium chloride hexahydrate $YCl_3 \cdot 6H_2O$ (chemically pure, TU 6-09-4773-84, 99.9%), iron (III) chloride hexahydrate $FeCl_3 \cdot 6H_2O$ (analytical grade, GOST 4147-74, 97%), potassium ferricyanide (potassium hexacyanoferrate(III)) $K_3[Fe(CN)_6]$ (pure, GOST 4206-65, 97%), ammonium fluoride $NH_4F \cdot HF$ (analytical grade, GOST 9546-75, 97%), ammonium chloride NH_4Cl (analytical grade, GOST 3773-72, 98%), and polyvinyl alcohol $(-C_2H_4O)_n$. These chemicals were obtained from Vekton in Russia. Distilled water was employed to prepare the MgO sol, and all chemicals were used without any further purification.

ii. Synthesis Methodology and Preparation Technique

Xerogels composed of $MgO:Zn(NO_3)_2 \times 6H_2O$, $MgO:YCl_3 \times 6H_2O$, and $MgO:FeCl_3 \times 6H_2O$ were synthesized using the sol-gel method. Figure 1 illustrates a schematic of the sol-gel process, showing the precipitation of zinc nitrate, yttrium chloride, and ferric chloride after their addition to a pure magnesium oxide (MgO) sol.

The synthesis involved preparing xerogels at five different concentrations of the mentioned metal salts, resulting in various sizes and shapes. Key process parameters, such as reaction temperature and time, molar ratios of MgO to metal precursors, and initial concentrations of zinc, yttrium, and iron, were critical for successful synthesis (refer to Table 1).

Initially, a sol was prepared from pure MgO, to which zinc nitrate, yttrium chloride, and ferric chloride were added in the specified concentrations as water-soluble salts. This resulting sol, whether pure or containing metal salts, was allowed to gel in open plastic molds under ambient conditions.

The gels were subsequently dried in a ventilated oven at 80°C, producing xerogels of the compositions $MgO:Zn(NO_3)_2 \times 6H_2O$, $MgO:YCl_3 \times 6H_2O$, and $MgO:FeCl_3 \times 6H_2O$ at various concentrations. Table 1 summarizes the quantities of

zinc nitrate, yttrium chloride, and ferric chloride added to the aqueous MgO dispersion, along with the corresponding salt solutions.

Once the xerogels were formed, they were annealed in air at 700°C for one hour, facilitating the conversion of the xerogels into their respective metal oxides: zinc oxide (ZnO), yttrium oxide (Y₂O₃), and iron oxide (Fe₂O₃) [10–12]. The xerogels were then ground and sieved into fractions using a planetary mill and a vibrating sieve. If micro-powders of the compositions MgO:ZnO, MgO:Y₂O₃, and MgO:Fe₂O₃ containing reduced copper were needed, the xerogels were ground in an agate mortar and sieved through a 100 μm sieve.

The resulting micro-powders were used to create tablets of the compositions MgO:ZnO, MgO:Y₂O₃, and MgO:Fe₂O₃. These powders were compressed into tablet samples using a hydraulic press, forming disks with initial diameters of approximately 13 mm, 20 mm, and 40 mm. The pressing force applied during tablet formation was around 120-150 kg/cm². Finally, the tablets were calcined in air at 700°C for one hour to achieve the final configurations of MgO:ZnO, MgO:Y₂O₃, and MgO:Fe₂O₃ [13].

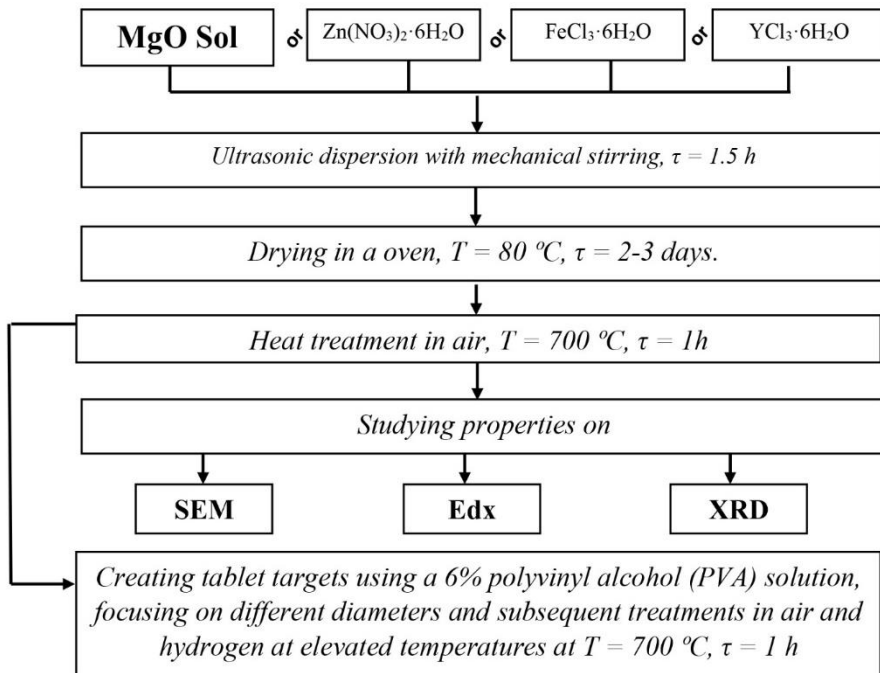


Figure 1 – Scheme for obtaining xerogel MgO matrices doped with Zn, Fe and Y.

Table 1 - Amount of dissolved zinc nitrates, ferric chlorides and yttrium chlorides in 200 ml of magnesium oxide-based sol.

Composition	MgO: Zn(NO ₃) ₂ ×6H ₂ O				
Mole concentration	(1:0.05)	(1:0.10)	(1:0.15)	(1:0.20)	(1:0.25)
Concentration in grams per 200 ml of MgO sol	6.8	13.6	20.4	27.2	34
Composition	MgO: YCl ₃ ×6H ₂ O				
Mole concentration	(1:0.05)	(1:0.10)	(1:0.15)	(1:0.20)	(1:0.25)
Concentration in grams per 200 ml of MgO sol	6.9	13.8	20.7	27.6	34.5
Composition	MgO: FeCl ₃ ×6H ₂ O				
Mole concentration	(1:0.05)	(1:0.10)	(1:0.15)	(1:0.20)	(1:0.25)
Concentration in grams per 200 ml of MgO sol	6.15	12.3	18.45	24.6	30.75
Composition	MgO:FeZnOF (1:1)				
	K ₃ [Fe(CN) ₆]	NH ₄ Cl	NH ₄ F.HF	Zn(NO ₃) ₂ ×6H ₂ O	
Concentration in grams per 200 ml of MgO sol	52	9.72	10.40	54.16	

iii. Characterization

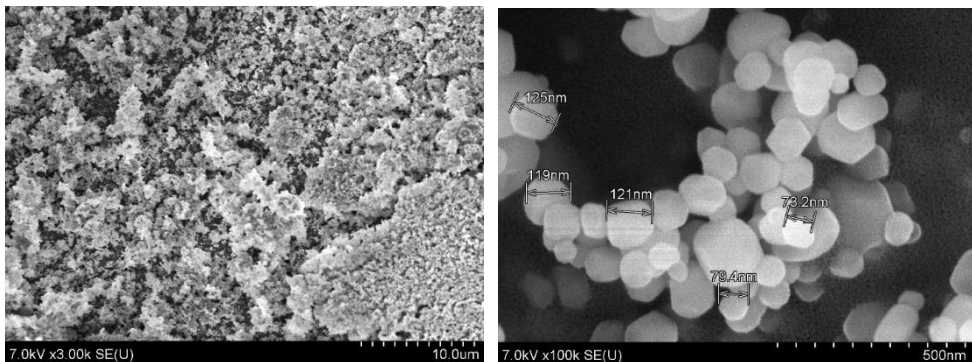
The surface morphology of the synthesized samples was examined on the central part of the fractured MgO xerogel, as depicted in the figures, utilizing a scanning electron microscope model S-4800 (Hitachi, Japan) with a resolution of 1 nm. The concentrations of various elements were assessed using the EDX (EDS) method with a Quantex 200 energy-dispersive microanalyzer (without a nitrogen microanalyzer and equipped with an XFlash Detector 5030), featuring a resolution of 125 eV (Bruker, Germany). These studies were conducted by staff from the STC "Belmicroanalysis," part of the "Belmicrosystems" branch of JSC "INTEGRAL," the management company for the holding "INTEGRAL." To analyze the phase composition of the synthesized samples' surfaces, the central part of the MgO xerogels was also examined. Nanostructured micropowders were analyzed using X-ray diffraction on a multifunctional diffractometer GNR APD 2000 PRO, with the

studies performed by personnel from the BelCZM Institute of Mechanics of Metal-Polymer Systems named after V. A. Bely.

III. Results & Discussion

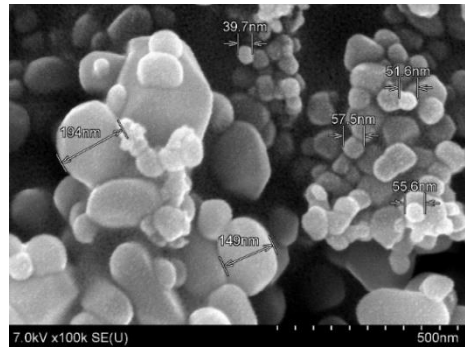
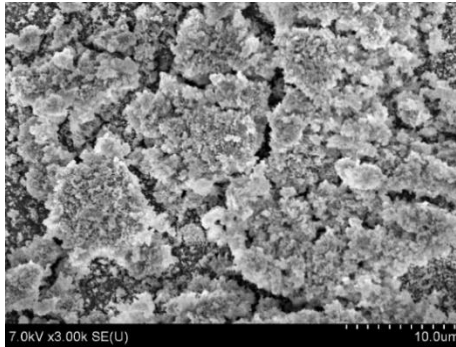
To investigate the interaction of various materials with the surfaces of spheres within the MgO xerogel, five types of samples were prepared: MgO, MgO:ZnO, MgO:Y₂O₃, MgO:Fe₂O₃, and MgO:FeZnOF. These samples were analyzed using a scanning electron microscope (SEM) (see Figures 2 and 3). The dried gels exhibited a distinct spherical structure and high morphological homogeneity. According to the data sheets, the large spherical aggregates forming the xerogel framework consist of magnesium oxide particles approximately 5–75 nanometers in size.

Due to the high degree of hydration, zinc nitrate salts, along with iron and yttrium chlorides, occupy the interspherical and porous spaces of the xerogel. The formation of matrices from these dry gels (MgO, MgO:ZnO, MgO:Y₂O₃, MgO:Fe₂O₃, and MgO:FeZnOF) restores the structure of the dry gel, indicating the mutual chemical inertness of the phases involved. It was observed that the incorporation of zinc, yttrium, and iron alters the internal structure of the diluted gel, creating a unique layer on the surfaces of the MgO granules. This effect is likely attributed to the high concentrations of zinc nitrate and yttrium chloride in the initial MgO solution (with the ratio of Mg:Zn/Y/Fe atoms being 1:0.05, 1:0.10, etc.), along with the significant absorption capacity of the xerogel matrix itself.

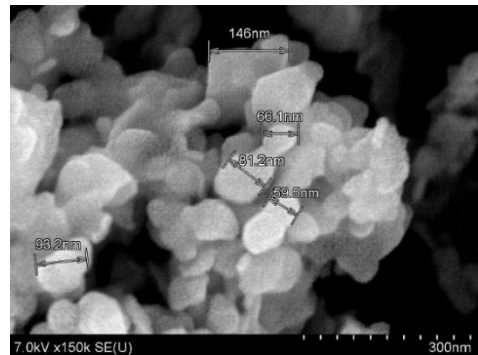
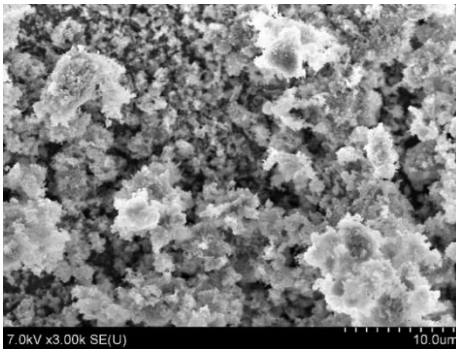


a) annealing in air at $T=700\text{ }^{\circ}\text{C}$ for 1 hour. Phase composition of the xerogel is MgO.

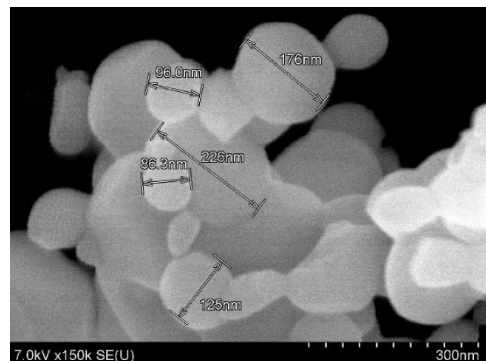
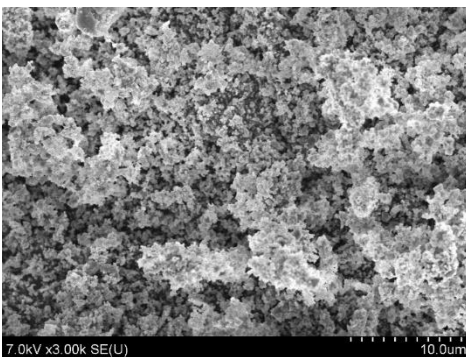
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b) annealing in air at $T=700\text{ }^{\circ}\text{C}$ for 1 hour. Phase composition of the xerogel is $\text{MgO}:\text{ZnO}$.



c) annealing in air at $T=700\text{ }^{\circ}\text{C}$ for 1 hour. Phase composition of the xerogel is $\text{MgO}:\text{Y}_2\text{O}_3$.



d) annealing in air at $T=700\text{ }^{\circ}\text{C}$ for 1 hour. Phase composition of the xerogel is $\text{MgO}:\text{Fe}_2\text{O}_3$.

Figure 2 – SEM image illustrating the morphological features of sorption materials based on magnesium oxide. (The concentration of zinc nitrate/yttrium chloride/iron chloride is relative to 1 mol of MgO in 200 ml of a 0.05 mol.% solution of zinc nitrate/yttrium chloride/iron chloride.)

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During the study, comprehensive measurements of the synthesized samples (MgO, MgO:ZnO, MgO:Y₂O₃, MgO:Fe₂O₃, and MgO:FeZnOF) were conducted using SEM at 700 °C. The results enabled an analysis of the morphological and structural characteristics of the samples. No distinct formation of micro- and nanoparticles was observed, suggesting the presence of two-dimensional layers composed of zinc, iron, and yttrium covering the inner surface of the xerogel. The composites exhibited a uniform distribution of particles ranging from 5 to 319 nm, indicating a high level of dispersion and a spherical particle shape (see Figures 2 and 3). Additionally, the SEM images revealed a porous structure within the synthesized composites, which is crucial for their application in filters and sorbents. The pore sizes ranged from 5 to 75 nanometers, enhancing the adhesion and mechanical properties of the materials.

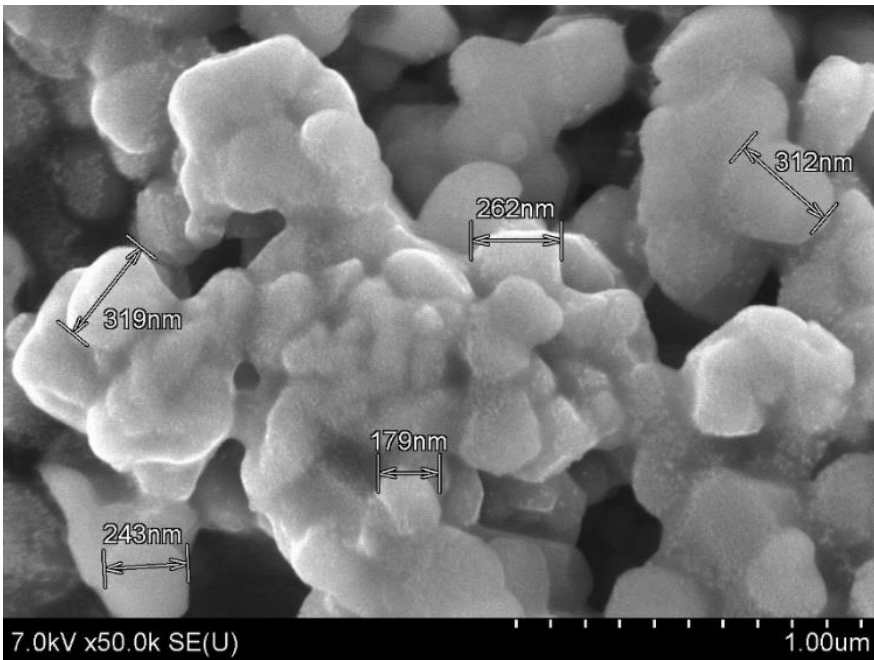




Figure 3 – SEM image of the morphology features of sorption materials of the composition MgO: FeZnOF at a concentration of (1:1) in moles, annealing in air at T=700 °C for 1 hour.

In MgO:ZnO, a uniform distribution of particles was observed, with sizes ranging from 39 to 194 nm, confirming a high level of dispersion and a spherical particle shape. For MgO:Y₂O₃, particle sizes varied from 59 to 146 nm; in MgO:Fe₂O₃, they ranged from 86 to 226 nm; and in MgO:FeZnOF, sizes spanned from 5 to 319 nm. This indicates that the pores in the MgO matrix were effectively filled. Additionally, heat treatment at 700 °C resulted in phase transformations, evident by changes in particle morphology and the formation of new structures typical of composite materials.

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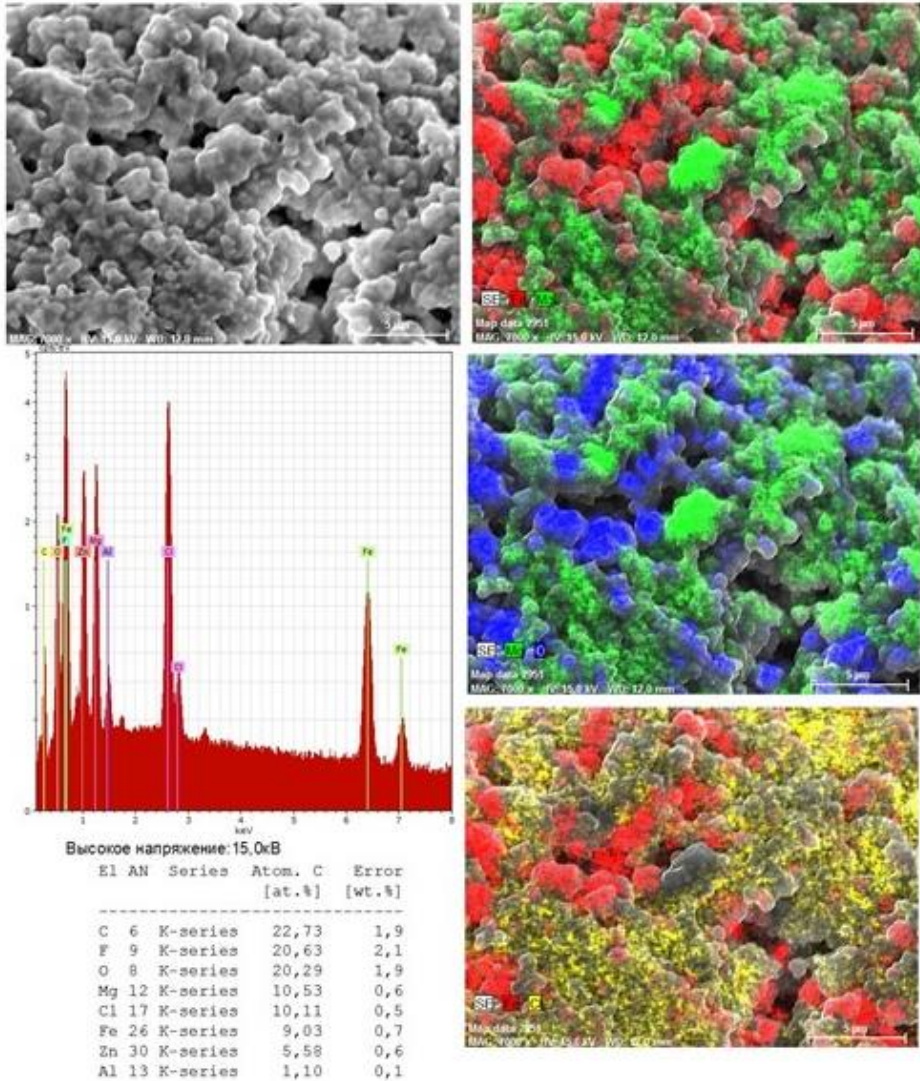


Figure 4. EDX analysis of MgO:FeZnOF composites after heat treatment in air at 700 °C.

The elemental composition and distribution of dopants within the magnesium oxide (MgO) matrix were examined using energy dispersive X-ray spectroscopy (EDX). The MgO:FeZnOF compound was prepared and characterized with a dopant concentration of 1:1 in moles (see Fig. 4). The analysis confirmed the presence of magnesium (Mg), oxygen (O), iron (Fe), zinc (Zn), and fluorine (F)

within the matrix, all of which were uniformly distributed, indicating successful incorporation of the dopants into the MgO host lattice. It was observed that chlorine (Cl) remained in the sample, likely due to inadequate annealing temperature during synthesis, which prevented the complete removal of chlorine residues. The presence of carbon (C) in the EDX spectrum can be attributed to two factors: (i) the high sensitivity of the measuring instrument to carbon contamination, potentially stemming from the instrument itself or the sample preparation process, and (ii) the high concentration and complex nature of the synthesized compound, which can retain carbon-based impurities [14-18].

X-ray diffraction patterns of the starting materials and composite materials synthesized from an aqueous dispersion of magnesium oxide, ferric chloride, ammonium fluoride, and zinc nitrate were analyzed to determine their phase composition. The samples underwent heat treatment in air at 700 °C for 1 hour during synthesis. The diffraction patterns of the micro-powders obtained from the xerogel synthesized from these reagents and annealed under the same conditions are displayed in Figure 5.

Figure 5 presents the X-ray diffraction patterns of the as-synthesized powders of the MgO:FeZnOF composite materials, produced by heat treatment of the starting materials at 700 °C for 1 hour. Analysis of the X-ray diffraction data using the JCPDS database (Card No. 45-0946) indicated that the synthesized powders correspond to the cubic structure of MgO:FeZnOF, consistent with previously published data [18-21]. The high intensity of the peaks in the X-ray diffraction patterns reflects a significant degree of crystallinity in the synthesized fine powders. The X-ray diffraction analysis revealed that the complexity of the synthesized compound, along with the preparation conditions and high concentration of additives in the magnesium oxide-based sol, resulted in the formation of iron oxide in three distinct forms. As shown in Figure 5, peaks corresponding to Fe₂O₃, Fe₃O₄, and FeO were identified, alongside peaks for zinc oxide (ZnO) and potassium. These findings corroborate the initial compositional measurements obtained through energy-dispersive X-ray spectroscopy (EDX). Additionally, magnesium oxide was found to be in a non-crystalline state, which can be attributed to insufficient processing temperature and the presence of small amounts of chlorine that were not eliminated due to low thermal conditions. This compound exhibited high porosity, facilitating the absorption of waste oil while allowing only clean water to pass through. The material's effectiveness as a water purification filter was further validated by practical results demonstrating its ability to separate waste oil and permit only clean water to flow through.

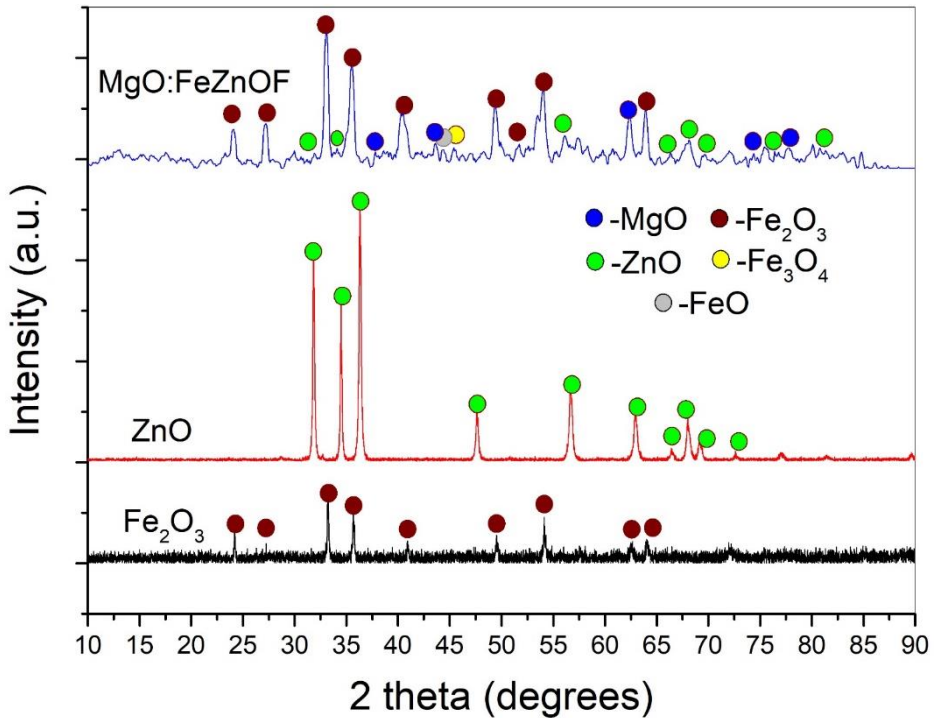


Figure 5 – Diffraction patterns of a xerogel formed on the basis of an aqueous dispersion of magnesium oxide (heat treatment in air at $T=700\text{ }^{\circ}\text{C}$ for 1 hour), the composition of the xerogel is MgO:FeZnOF at a concentration of (1:1) in moles

To determine the sorption capacity, the volume of the dispersion medium that passed through a ceramic filter of specified area over a designated time period during the filtration of an aqueous solution mixed with oil was measured. For this study, ceramic tablets with a diameter of 12.5 mm and a thickness of 5 mm (varying between 3 and 10 mm) were synthesized (see Fig. 6). Figure 6 illustrates the final appearance of the tablets used in the experiment after being exposed to air at a temperature of $700\text{ }^{\circ}\text{C}$.

The results shown in Table 1 indicate the ability of the synthesized composite materials to absorb oil products. To assess this, the tablets were placed in transparent plastic cups containing crude oil and machine oil, allowing for a comparison of their sorption properties. The samples were immersed in the liquids for one day. Figure 6 also provides an example of a sample used to evaluate the sorption capacity in both crude oil and machine oil.

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a) tablets before immersion in crude oil and machine oil



b) tablets immersed in machine oil



c) tablets immersed in crude oil

Figure 6 – Image of tablet after immersion in crude oil and machine oil
Figure (7) shows the manufactured tablets after they were immersed in crude oil and machine oil.

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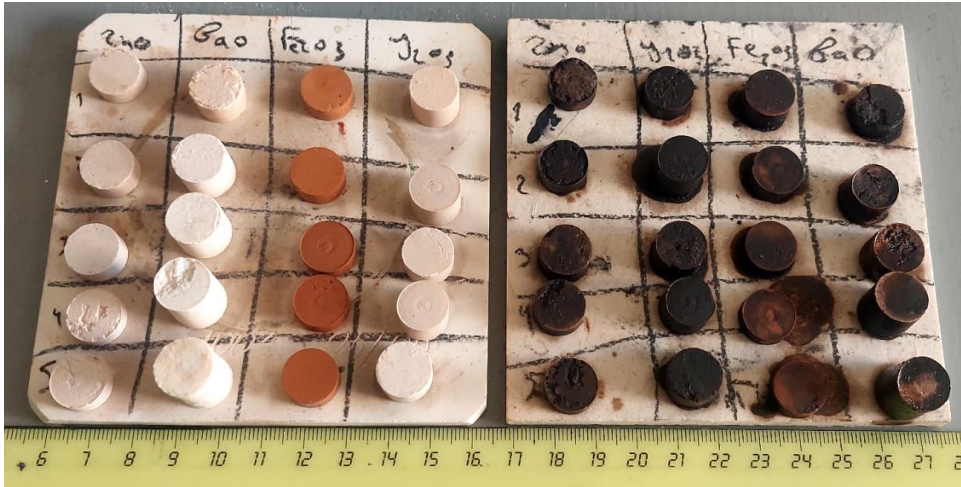
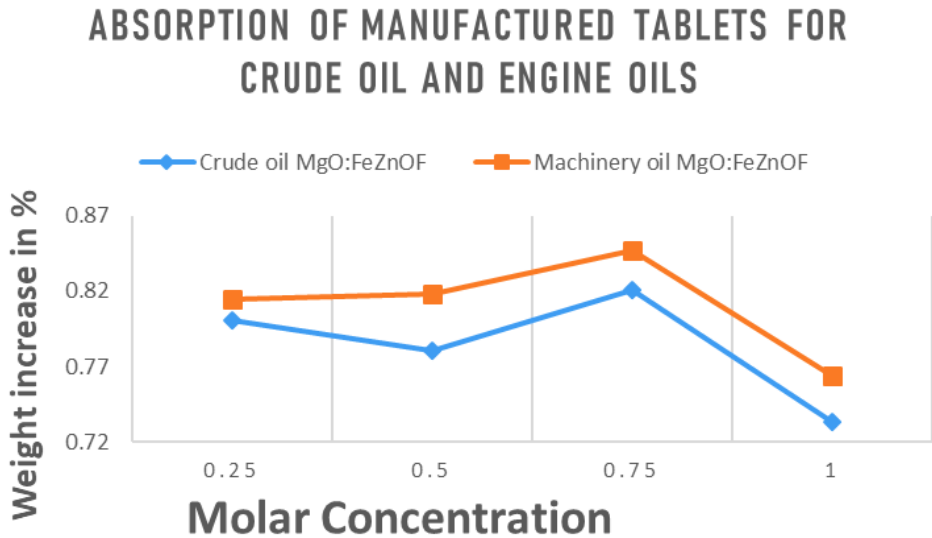


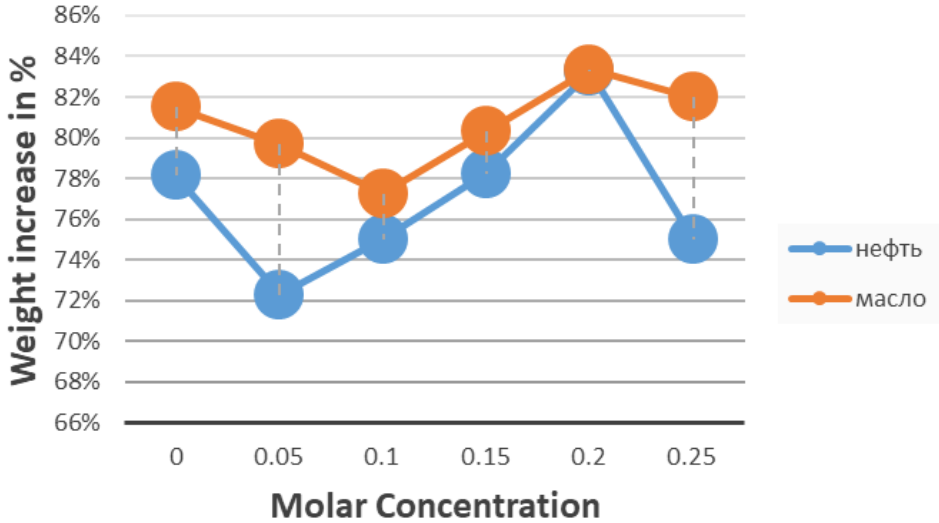
Figure 7 – Image of tablet after immersion in crude oil and machine oil

After the tablets were soaked overnight in crude oil or machine oil, their percentage absorption was determined by calculating the difference in mass before and after immersion. The results of these measurements are presented in Figure 7.

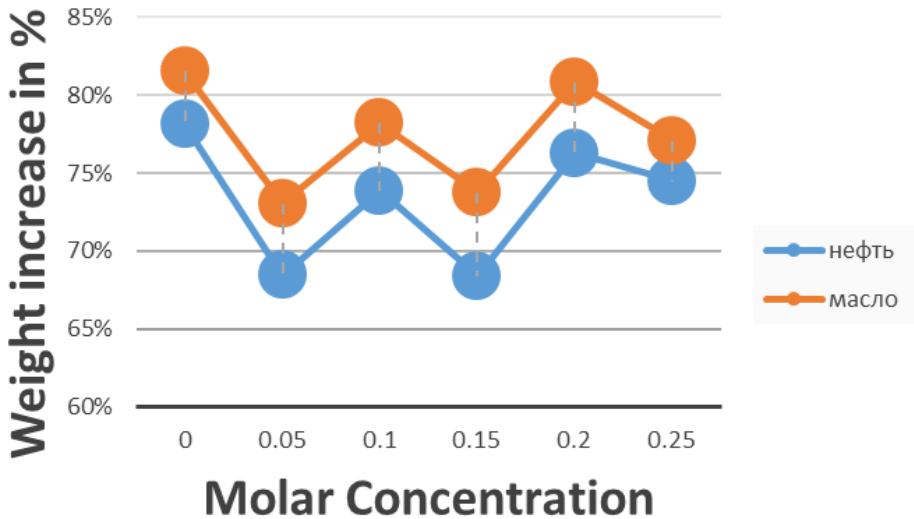


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MgO:Fe₂O₃



MgO:Y₂O₃



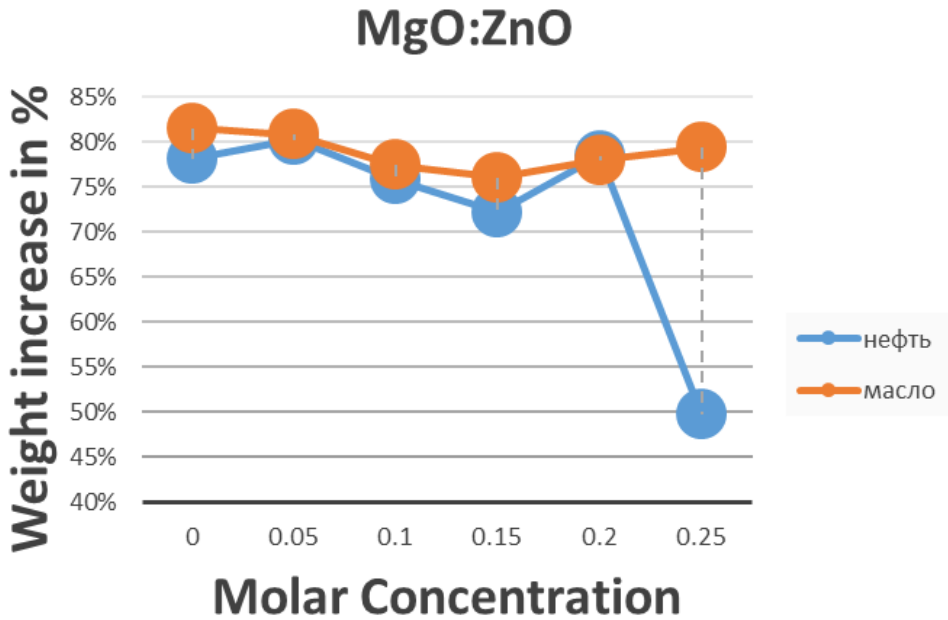


Figure 8 – Study of the change in the mass of tablets prepared with different concentrations, depending on the absorption capacity of petroleum materials.

The study investigated the properties and effectiveness of MgO:ZnO, MgO:Y₂O₃, MgO:Fe₂O₃, and MgO:FeZnOF composite materials for filtering water contaminated with oil and their potential use as absorbents for oil waste. The samples were treated in air at 700 °C for one hour.

The MgO:ZnO composition exhibited high effectiveness in removing oil contaminants from water, achieving a removal rate of 85%. This efficiency can be attributed to the presence of active sites on the material's surface, which enhance adsorption. These findings align with previous research indicating that ZnO possesses strong adsorption properties for organic pollutants [22].

MgO:Y₂O₃ demonstrated stability and significant adsorption capabilities, with an oil removal rate of 80%. The presence of yttrium improved the material's morphology and porosity, as supported by studies highlighting the benefits of adding Y₂O₃ to enhance the adsorption properties of oxides [23].

MgO:Fe₂O₃ delivered exceptional results, achieving a 90% removal rate of oil impurities. The inclusion of iron contributes to the material's magnetic properties, enabling the use of magnetic methods for contaminant extraction. These findings are

consistent with literature indicating that Fe_2O_3 is an effective adsorbent for organic pollutants [24].

The MgO:FeZnOF composition exhibited remarkable adsorption properties, allowing for the removal of up to 88% of oil contaminants. The structure formed during thermal treatment creates active sites that enhance adsorption capacity. This is corroborated by research discussing the advantages of combined oxides in wastewater treatment [25].

IV. Conclusion

In conclusion, the findings of this study demonstrate that MgO-based composite materials possess significant potential for effectively treating wastewater contaminated with oil products. Notably, the MgO:Fe₂O₃ composition exhibited the highest adsorption capacity. The application of heat treatment at 700 °C enhanced the physicochemical properties of the synthesized materials, thereby improving their practical usability. Additionally, it was observed that lower concentrations of initial materials in the magnesium oxide matrix correlated with increased absorption at processing temperatures above 700 °C. The influence of doped oxides on sorption capacity underscores the possibility of optimizing these materials to create adsorbents with tailored characteristics. The developed technological framework for sorbent production encompasses all essential stages, from the preparation of a MgO-based sol to the formation of final products. Overall, the study indicates that these materials can be effectively utilized for the collection of oil products and water purification, paving the way for new applications in environmental protection and water treatment.

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