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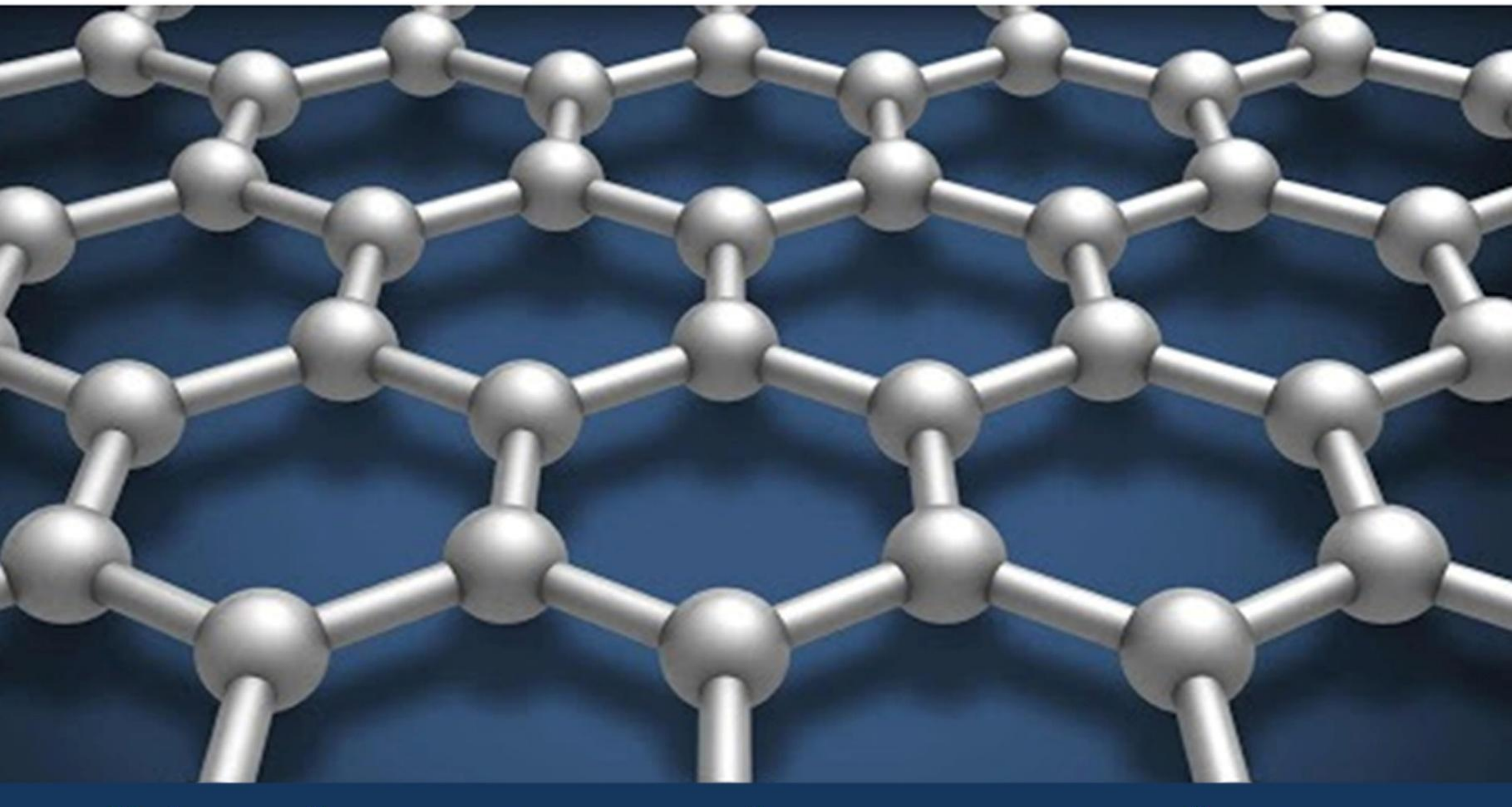

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Композиционные материалы

SYNTHESIS AND PHYSICOCHEMICAL CHARACTERIZATION OF HIGHLY ABSORBENT OLEOGELS

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Abstract - Oleogels were successfully prepared via radical polymerization and crosslinking of butadiene–styrene rubber (BSK), natural rubber (NR), and isoprene rubber (SKI) in a toluene medium. The synthesis was performed in a 250 mL round-bottom flask with a reflux condenser at 80–90 °C, employing ammonium persulfate as the initiator and glutaraldehyde as the crosslinker. After polymerization, the oleogels were dried and subsequently treated with maleic anhydride to enhance crosslink density and reduce solubility. FTIR analysis confirmed the presence of characteristic functional groups, including CH_3, CH_2, C=N, and C–O–C, indicating the formation of a stable gel network.

Keywords: rubber, toluene, organic compounds, FTIR spectroscopy, DTG analysis.

Introduction. Currently, significant research efforts worldwide focus on developing novel, environmentally friendly, and economically feasible methods for synthesizing oleogels from hydrophobic compounds, as well as investigating their potential applications in industrial, agricultural, and domestic contexts. Originally regarded as cost-effective synthetic materials, oleogels have been widely applied for oil spill recovery. However, the gradual discontinuation of certain dispersants-some of which pose even greater risks to aquatic environments than petroleum itself-has intensified the search for alternative oil recovery strategies. Collecting spilled oil from water bodies continues to represent a major environmental challenge [1, 2].

Oily wastewater is increasingly recognized as a critical global environmental issue. The severity of oil contamination is closely linked to the worldwide expansion of oil exploration and extraction, coupled with rapid industrial growth. For example, global annual consumption of petroleum-derived products and vegetable oils exceeds approximately 100 million and 92 million metric tons, respectively [3].

Between 2000 and 2011 alone, oil tanker spills released an estimated 224,000 tons of petroleum into the oceans [4–6]. This work investigates the formation of EC–MGc oleogels by examining interactions between low molecular weight ethyl cellulose (19 kDa) and a commercial monoglyceride. Oleogels were prepared from vegetable oil solutions of EC at different concentrations (7%, 8%, and 10%) with MGc levels below its minimal gelling concentration (0–1%). Rheological, DSC, and FTIR analyses indicated that at 0.1% MGc, hydrogen bonding between EC and MGc enhanced oleogel elasticity, whereas higher MGc concentrations (≥0.25%) promoted EC–EC interactions due to increased oil polarity. At temperatures below 10 °C, MGc crystallized within the EC network, acting as an active filler [6].

Various structuring agents-including fatty acids (e.g., 12-HSA), fatty alcohols, ceramides, lecithins, sterols, cellulose fibers, and fumed silica-have been extensively studied for their molecular characteristics, gelling abilities, and oleogelation mechanisms, alongside the thermal, rheological, and stability properties of the resulting gels. These materials exhibit potential applications in the food and cosmetic industries [7].

In this study, oleogel emulsions composed of monoglycerides (MG), hardfat (HF), and lecithin (LE) were examined, with and without high-intensity ultrasound (HIU). MG alone produced stable emulsions, whereas MG:HF combinations demonstrated the strongest synergistic effects across all water contents. Application of HIU improved droplet organization, stability, and functional properties such as hardness, elasticity, and oil-binding capacity, making these systems promising for food-related applications [8, 9].

Materials and Methods. The functional groups present in the composition were identified by Fourier-transform infrared spectroscopy (FTIR) using a Shimadzu IRTracer-100 spectrometer (Shimadzu, Japan), based on the characteristic absorption bands in the infrared region. Thermal stability and decomposition behavior of the composition were evaluated by thermogravimetric analysis (TGA) and differential thermal analysis (DTA) using a DTG-60 thermal analyzer (Shimadzu, Japan) under an argon atmosphere with a flow rate of 80 mL min^{-1} and a constant heating rate of 10 °C min^{-1}.

Experimental Procedure for Synthesis. To obtain the super oleogel we synthesized, we need a 250 ml high-temperature resistant flask, a 200°C thermometer to monitor the temperature, an oil bath and an oven to maintain the temperature constant, and a reflux condenser to maintain the reaction. A 10% solution of styrene butadiene rubber (BSK), natural rubber (TK), and isoprene rubber (SKI) was prepared. First, the rubbers were dissolved in toluene. The mixture was stirred with a glass rod at room temperature for two days. The resulting solution was placed in a 250 ml flask and, after installing a reflux condenser on the flask containing the solution, it was placed in an oil bath in the oven, then the temperature was maintained at 80°C for 40 minutes, and then 0.3% ammonium persulfate was added to it as an initiator, and the reaction temperature was slowly increased. After the reaction temperature reached 90°C, 3% glutaraldehyde was added as a mordant. The reaction process was carried out at 80-90°C for 6 hours, and it was dried in a User Manual oven at 75°C for 12 hours. *IR-analysis.* FTIR spectrum analysis of the synthesized oleogel (Figure 1).

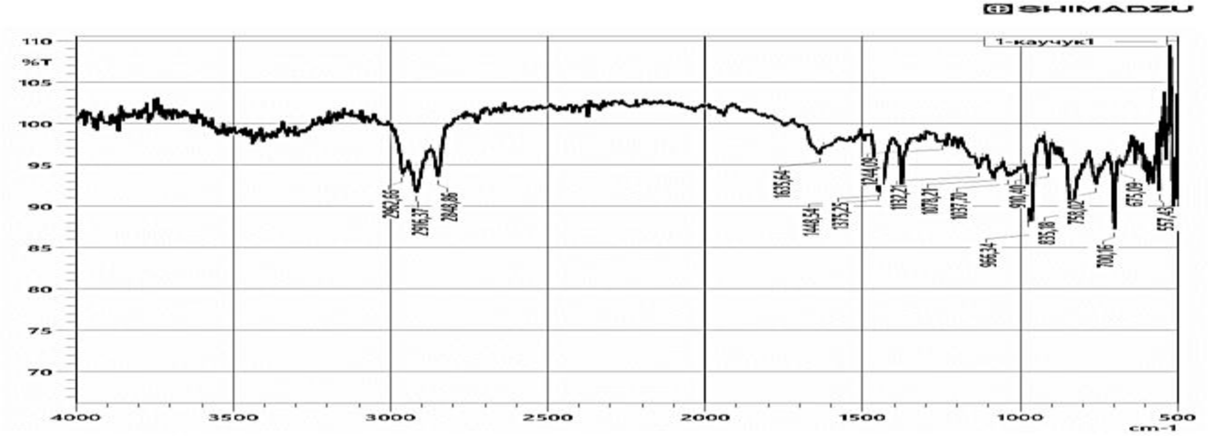

Fig. 1. FTIR spectrum of the synthesized super oleogel

When super oleogels (gel substances capable of absorbing water-insoluble oils and petroleum products) used in wastewater treatment are analyzed by infrared (IR) spectroscopy, the functional groups and structural properties of their composition are determined. The IR spectrum of our resulting Super oleogels was analyzed. The presence of highly intense vibrational asymmetric valence bonds of CH_3 in the 2962.66 cm^{-1} region, highly intense vibrational asymmetric valence bonds of CH_2 in the 2916.37-2848.86 cm-1 region, intensive valence vibrational bonds of ¬C=N¬ in the 1635 cm^{-1} region, asymmetric deformation vibrational bonds of ¬CH_2 in the 1448 cm-1 region, and highly intense, asymmetric valence vibrational bonds of ¬C¬O¬C¬ in the 1244.09 cm^{-1}, 1132.21 cm^{-1}, 1078.21 cm^{-1}, 1037.70 cm^{-1} regions was detected.

Results and Discussion. ***DTA and TGA Analysis.*** To evaluate the thermal stability and decomposition behavior of the PF-1 composite corrosion inhibitor, simultaneous thermogravimetric (TGA) and differential thermal (DTA) analyses were conducted within a temperature range of 10–600 °C using a SHIMADZU DTG-60 instrument (Figure 1). During the measurements, each sample was continuously exposed to a dynamic flow of argon gas (80 mL min^{-1}) to maintain an inert atmosphere. The heating process was performed in an aluminum crucible equipped with a furfuric alumina pan, with a constant heating rate of 10 °C min^{-1}.

The obtained thermograms clearly indicated both endothermic and exothermic transitions of the oleogel sample. The detailed results of the DTA and TGA measurements for the synthesized oleogel are illustrated in Figure 3.

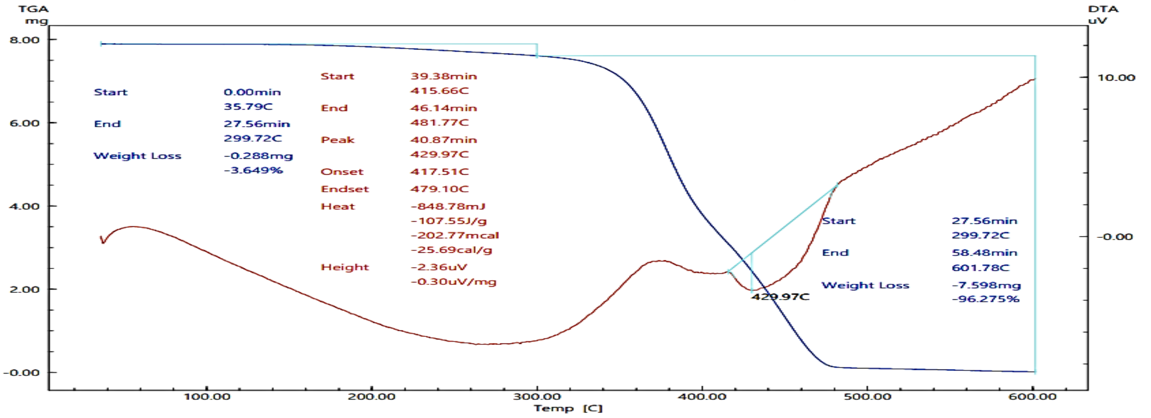

Fig. 2. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) curves of the synthesized oleogel

Figure 2 presents the thermal analysis of the synthesized oleogel in its dry form, conducted up to a maximum temperature of 600 °C. Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) results were interpreted based on the corresponding curves. An endothermic peak was observed at 429.97 °C, indicating the temperature at which the material absorbs heat, resulting in a change in its internal energy. The specific variations in energy state are summarized in Table 1.

Table 1. Differential Thermal Analysis (DTA) profile of the synthesized oleogel, showing characteristic endothermic and exothermic transitions

Temperature	Total energy absorption, µV	Per unit mass uV/mg	Heat, m/cal	Specific heat, cal/g
429.97 °C	-2.36	-0.30	-202.77	-25.69

The first mass loss occurred between 35.75 and 299.72 °C, corresponding to a reduction of 0.288 mg, which is attributed to the evaporation of residual glutaraldehyde and unreacted toluene from the oleogel synthesis. A second, more significant mass loss of 7.886 mg was observed between 299.72 and 601.78 °C, reflecting the complete thermal decomposition of the oleogel and the release of gaseous products [13-18]. The thermogravimetric analysis (TGA) curve (blue line) shows that these two temperature ranges account for the majority of the observed mass losses, as summarized in Table 2.

Table 2. Thermogravimetric analysis (TGA) profile of the synthesized oleogel

Temperature	Mass loss, mg	Mass loss, %
35.79-299.72°C	0,288	3.649
299.72-601.78°C	7.598	96.275
Total	7.886	99.924

The synthesized oleogel undergoes thermal decomposition, producing gaseous products and simultaneously releasing a certain amount of energy to the surroundings. The agreement between the decomposition range and the corresponding temperature interval confirms this conclusion.

Conflicts of Interest. This section is compulsory. A competing interest exists when professional judgment concerning the validity of research is influenced by a secondary interest, such as financial gain. We require that our authors reveal any possible conflict of interest in their submitted manuscripts. If there is no conflict of interest, authors should state that The author(s) An Acknowledgements section is optional and may recognise those individuals who provided help during the research and preparation of the manuscript. Other references to the title/authors can also appear here, such as Khusanova M. F and Djalilov A. T contributed equally to this work.

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СОДЕРЖАНИЕ

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