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# Synthesis of polyether monomer-based demulsifiers and their efficiency in breaking up oil emulsions

## Poliefir monomerlari asosidagi demulgatorlar sintezi va ularning neft emulsiyalarini parchalashdagi samaradorligi

## Синтез деэмульгаторов на основе полиэфирных мономеров и их эффективность при разрушении нефтяных эмульсий

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**Abstract.** In this article, experiments were conducted on methods for synthesizing demulsifiers with high-performance polymer properties based on hydroxy polyethylene glycol (HPEG). The effectiveness of the synthesized demulsifier in breaking oil-water emulsions was studied. Nowadays, breaking emulsions formed during oil production and refining is a very complex process. As part of scientific research, surfactants were synthesized by modifying the hydrophilic chains of HPEG with a number of hydrophobic components. The synthesized demulsifier was applied to an oil sample and its effectiveness in separating water from the oil was studied. In addition, the chemical composition and molecular structure of the demulsifier were studied and analyzed using IR spectroscopy. SEM analysis methods.

**Keywords:** oil; surfactants; demulsifier; emulsion; hydrophob

### O'ZBEKCHA

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**Annotatsiya.** Ushbu maqolada gidroksi polietilenglikol (HPEG) asosida yuqori samarali polimer xossalariga ega demulgatorlarni sintez qilish usullari bo'yicha tajribalar o'tkazilgan. Sintez qilingan demulgatorning neft-suv emulsiyalarini parchalashdagi samaradorligi o'rganilgan. Bugungi kunda neftni qazib olish va qayta ishlash jarayonida hosil bo'ladigan emulsiyalarni parchalash juda murakkab jarayon hisoblanadi. Ilmiy tadqiqot doirasida HPEG ning gidrofil zanjirlarini bir qator gidrofob komponentlar bilan modifikatsiyalash orqali sirt-faol moddalar sintez qilingan. Sintez qilingan demulgator neft namunasiga qo'llanilib, uning neftdan suvni ajratishdagi samaradorligi o'rganilgan. Bundan tashqari, demulgatorning kimyoviy tarkibi va molekulyar tuzilishi IQ-spektroskopiya hamda SEM tahlil usullari yordamida o'rganilib, tahlil qilingan.

**Kalit so'zlar:** neft; sirt-faol moddalar; demulgator; emulsiya; gidrofoblik

### РУССКИЙ

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**Аннотация.** В настоящей статье проведены эксперименты по методам синтеза деэмульгаторов с высокоэффективными полимерными свойствами на основе гидроксиполиэтиленгликоля (HPEG). Изучена эффективность синтезированного деэмульгатора при разрушении нефтеводяных эмульсий. В настоящее время разрушение эмульсий, образующихся при добыче и переработке нефти, представляет собой весьма сложный процесс. В рамках научного исследования поверхностно-активные вещества были синтезированы путём модификации гидрофильных цепей HPEG рядом гидрофобных компонентов. Синтезированный деэмульгатор был применён к образцу нефти, и изучена его эффективность в отделении воды от нефти. Кроме того, химический состав и молекулярная структура деэмульгатора были изучены и проанализированы методами ИК-спектроскопии и СЭМ-анализа.

**Ключевые слова:** нефть; поверхностно-активные вещества; деэмульгатор; эмульсия; гидрофобность

## 1. INTRODUCTION

The oil extracted from oil fields contains various amounts of water droplets. The amount of water in oil varies depending on the place of its extraction and the extraction methods. This is because oil is formed under different conditions underground. Some layers of the earth have a high water content and some layers have a much lower water content. From this point of view, the amount of water in the oil also differs accordingly [1].

Water in oil fields mainly takes the following forms according to the causes of its formation. In the case of groundwater, the water layer is located around or below the oil reservoir. As oil production increases, such water replaces the oil layer underground as a result of a decrease in pressure and is extracted partially mixed with oil during the extraction process. Also, water on the surface of mineral particles in small pores and capillaries of rocks and earth layers does not move together with oil underground. However, under the influence of high pressure and strong currents in the underground, it is partially separated and sometimes joins the oil flow. According to the methods of technological water extraction, oil initially comes out of the ground under its own pressure. As the well surface deepens and the amount of oil in the well decreases, the pressure also decreases. In such a case, water or other chemical reagents are injected into the underground oil layer to maintain the formation pressure and increase the volume of oil extraction. The water or chemical reagents injected into the well act as a natural emulsifier. This actively participates in the formation of a stable emulsion in the oil composition.[2] Water and oil do not naturally mix with each other due to the difference in density. However, during the extraction process, when they rise from the bottom of the well to the surface, they are subjected to strong mechanical action. As a result, water partially mixes with the oil composition and oil-water emulsions are formed [3].

As we know, oil consists of several components, which often include natural emulsifiers such as paraffins, naphthenic and aromatic hydrocarbons, tar-asphaltenes, acidic compounds (naphthenic acids, fatty acids, aromatic acids) and resins. In addition, oil contains more than 20 elements such as vanadium, nickel, calcium, magnesium, iron, aluminum, silicon, sodium, hydrogen sulfide, thiophene, disulfides, pyridines and a number of other emulsifiers [4]. In particular, a large part of the currently produced oils is classified as heavy and high-viscosity oils. The content of asphaltenes and resins in such oils is quite high. Asphaltenes and resins play an important role in the formation of emulsions. The structure of asphaltenes and resins is quite complex and is the heaviest and most aromatic fraction of oil. It is soluble in aromatic solvents such as benzene, toluene, and xylene, but insoluble in light alkanes such as n-pentane and n-heptane[5]. The difficulty in solubility increases the activity in forming stable emulsions. Asphaltenes usually contain carbon (C), hydrogen (H), oxygen (O), nitrogen (N), sulfur (S), and small amounts of iron (Fe), vanadium (V), and nickel (Ni) as well as several other natural surfactants. These substances also serve to significantly strengthen the surface tension of the emulsion.

From the point of view of physical and colloidal chemistry, oil-water emulsions are hydrophobic dispersed systems. The state of water in them is described as follows, depending on the size of the droplets (d) and the thermodynamic stability of the system. Free and fast-moving water ($d > 100\mu m$). These water droplets are dispersed into the oil for a short time as a result of mechanical mixing. After the system is hydrodynamically stabilized, they quickly sink to the bottom under the influence of gravity in accordance with Stokes' law. A humic shell does not have time to form around the oil and water. As a result, water and oil separate into two phases. Finely dispersed, i.e. water that forms a stable emulsion ($0.1\ \mu m < d < 100\mu m$) Such emulsions are considered the most complex type of emulsion, and their decomposition requires high energy in oil refineries. Here, water droplets are covered with natural surfactants in the oil layer - asphaltenes, resins and high molecular acids [6,7,8,9].

The main emulsion-forming substances in the oil, namely tar and asphaltene fractions, as well as high-paraffin hydrocarbons, form a "reticular layer". It is this reticular layer that serves to form a stable emulsion with water droplets. The task of demulsifiers is to disrupt this layer, lose its elasticity and bring water droplets closer together (coagulate). When oil emerges from the bottom of the well, water is not only in the form of droplets in the oil. According to the dynamics of the process, water appears in the following technological situations. If the water content in the oil exceeds a certain critical point (usually 60-70%), the type of emulsion changes. The "water-oil emulsion" (O/W) system turns into an "oil-water emulsion" (W/O) system. From this point of view, the structure of oil emulsions is mainly of 4 types. These are oil-in-water (W/O), water-in-oil (O/W) and, in more complex forms, emulsions in the W/O/W and O/W/O states. (Figure 1) Due to their structural complexity, the separation of such emulsions is a rather difficult process. In this case, the viscosity of the system changes dramatically. In low-temperature climates or in transport pipelines, free and partially emulsified water can form hydrates or turn into ice crystals. This makes it difficult to transport oil in pipelines [10,11,12].

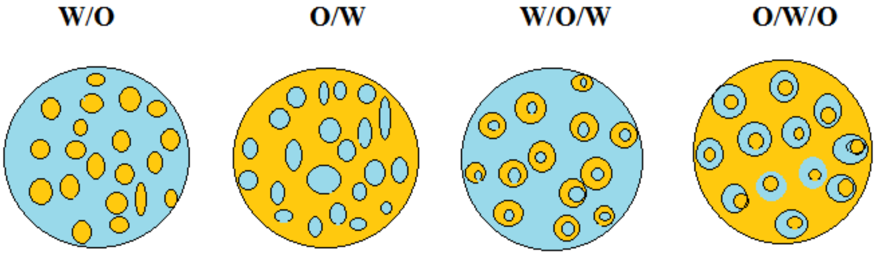


**Fig. 1.** Neft emulsiyalarining tashqi ko‘rinishi / **Fig. 1.** Appearance of oil emulsions

Today, several different methods of breaking oil emulsions are used in the oil refining industry. These are: Thermal method - in this case, the oil emulsion is heated to a certain temperature. (50-80 0C) The mechanical strength of natural emulsifiers in the oil, i.e. asphaltene-resins, decreases and, as a result, the emulsions break down [16,17].

The mechanical method is based on the initial settling of oil in oil tanks and then the separation of water-oil emulsions by centrifugation or filtration [18]. Electrical method - this method is carried out in Electrical Desalting and Dehydration Unit devices at oil refineries. The oil is treated with an electric current to remove water and dissolved mineral salts [19,20].

The chemical method is a unique method for breaking down oil emulsions, which is considered the most widely used and highly efficient method in the oil refining industry. In this case, oil emulsions are broken down using chemical reagents, i.e. demulsifiers[21,22,23]. Chemical surfactants are usually composed of amphiphilic molecules consisting of hydrophilic and hydrophobic groups. These amphiphilic molecules are well adsorbed at the water-oil interface. Hydrophilic groups move towards water, and hydrophobic groups towards oil. This allows for a significant reduction in the surface tension of the emulsion [24,25,26].

2. Methodological part

## 2. MATERIALS AND METHODS

### 2.1. Materials

HPEG was selected for synthesis due to its water-viscosity-reducing properties, obtained from the chemical reagent supplier “Diag Lab Trade”. The presence of carboxyl groups (-COOH) in the acrylic acid (CH2 = CH - COOH) molecule indicates its high water-absorbing ability, and this substance was obtained from the chemical reagent supplier “Xim lab pribor”. 1.3-Mercaptopropionic acid is important in the synthesis of surfactants due to the presence of two different functional groups (-SH and COOH) at both ends, and it was also obtained from the laboratory chemical reagent supplier “Diag Lab Trade”. Hydrogen peroxide (H2O2) is important in the synthesis of SFM because it has the ability to introduce hydroxyl (-OH) and carboxyl (-COOH) groups into its molecule, and these groups form hydrogen bonds with water droplets in the oil, which is important in breaking up oil emulsions, and it was also obtained from the company “Diag Lab Trade”.

### 2.2. Demulsifier synthesis

The synthesis process was carried out in a 500 ml three-necked, flat-bottomed flask equipped with an automatic stirrer, thermometer, dropwise addition device and other standard equipment. Initially, 30 grams of HPEG were weighed into the flask, 3 grams of acrylic acid and 0.3 grams of MPA-3 and 60 grams of water were added to the three-necked flask, and the reaction process was started. The temperature was gradually increased to 20 Co with an automatic rotation speed of 50 s/min. The reaction mass was continuously stirred using an automatic stirrer, which ensured a homogeneous reaction of the components and the effective development of polymer chains. The reaction temperature was maintained at 20 Co in an inert environment, which guaranteed the complete completion of the polymerization process and the yield of the resulting product. After 2 hours, 0.6 grams of hydrogen peroxide was added dropwise to the reaction process without stopping the reaction. The total reaction time was 4 hours. At the end of the reaction, a light reddish liquid was formed (Figure 2).

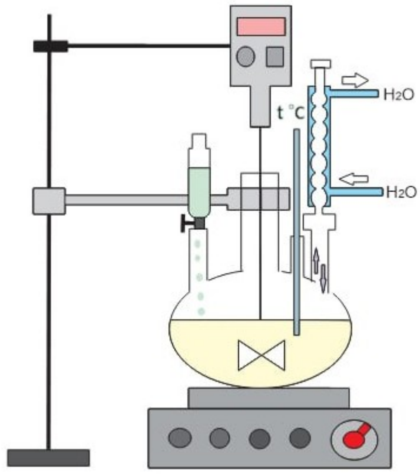


**Fig. 2.** Demulgator sintezi qurilmasining chizmasi / **Fig. 2.** Drawing of the demulsifier synthesis equipment

The process of obtaining a highly effective defoamer based on HPEG was carried out based on the following reaction mechanism: (Figure 3).

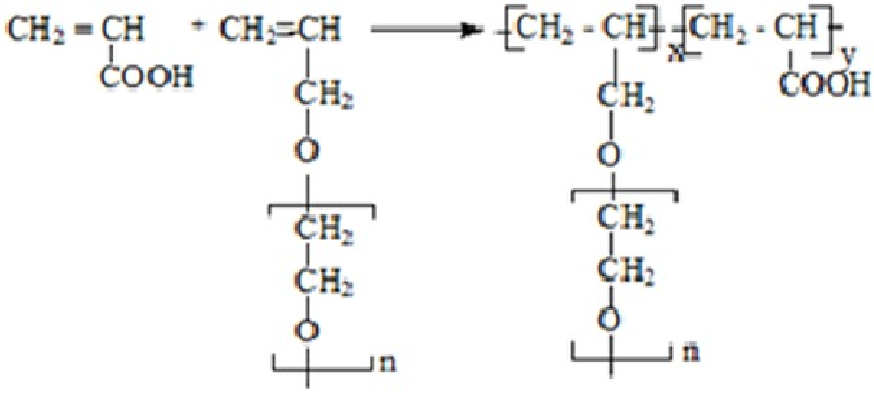

**Fig. 3.** Demulgator sintezi reaksiyasining tenglamasi / **Fig. 3.** Demulsifier synthesis reaction equation

## 3. RESULTS AND DISCUSSION

Several analytical methods were used to study the effectiveness of the HPEG-based demulsifier in separating oil-water emulsions. First, it was tested on an oil sample brought from the “Kakaydi” oil field. It was determined how much water the synthesized demulsifier was able to separate from the oil. It was previously determined that the oil sample contained 20% water. The process of testing the demulsifier on oil was as follows. For this, we needed three glass test tubes, a water bath to create a sufficient temperature for the oil sample, and a 100°C thermometer to constantly monitor the temperature. The same amount of oil sample was placed in three glass test tubes and lowered into a water bath. The temperature was gradually increased. Initially, when the temperature reached 40-50 °C, the demulsifier was added to the oil. If the temperature is not brought to this level, the oil will not change its solid state and the demulsifier will not be effective when applied. The glass test tubes were closed with a cylinder stopper and mixed well by hand before being put back into the water bath. This is a good effect for the uniform distribution of the demulsifier over the entire surface of the oil. Initially, the demulsifier was added at 10 ppm and water separation was observed every 10 minutes. After the demulsifier was added, the temperature was increased to 80-90 °C. However, at each temperature change, the appearance of the separated water was monitored using an electron microscope. Over time, the increase in the size of the water droplets and their sinking to the bottom of the oil were observed (Figure 4-a). The results obtained every 10 minutes were recorded. This helps to choose the most effective method for demulsifying the oil. This experiment is a very convenient way to observe the water content of the oil under an electron microscope (Figure 4-b).

In our next experiment, we used a flask with the ability to remove water from the bottom to accurately measure the amount of separated water in the oil (Figure 4-c). In this case, we performed the same work as above, except that in our previous work, we observed the state of the water in the oil through an electron microscope, which made it possible to directly measure the separated water through this experiment. The same experiments were carried out several times. In this case, the effectiveness of the synthesized demulsifier was tested at each stage at different concentrations, different temperatures, and different time intervals. At the end of the experiments, the most acceptable indicators5 for the separation of the synthesized demulsifier oil-water emulsions were determined (Figure 5).

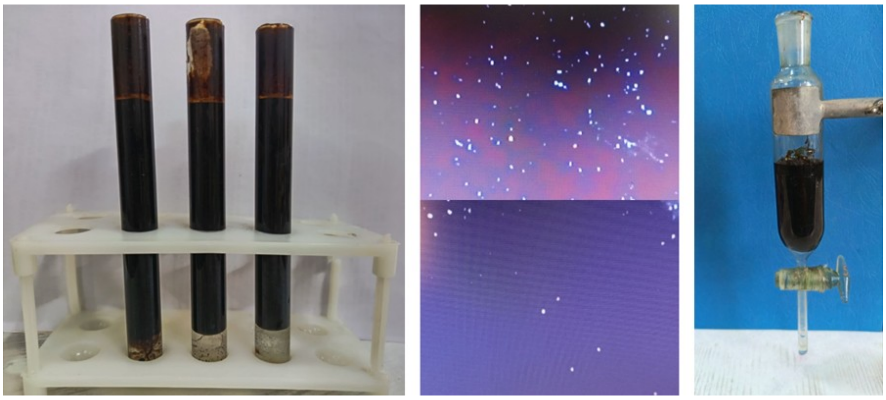

**Fig. 4.** Demulgator qo‘llanilgan neftning tashqi ko‘rinishi: A — sinov probirkalari; B — emulsiya namunasi; C — suvni ajratish kolbasi / **Fig. 4.** Appearance of oil with the demulsifier applied: A — test tubes; B — emulsion sample; C — water-separating flask

From this graph, it can be seen that for the most effective effect of the demulsifier applied to the oil, the following factors affecting the demulsification process can be considered: (Table 1).

**Table 1**

Demulgator samaradorligi / **Table 1.** Demulsifier efficiency

| Deemulsification time (min) | Concentration (ppm) | Temperatyre(C°) | Separated water(%) | Deemulsification time (min) | Concentration (ppm) | Temperatyre(C°) | Separated water(%) |
|---|---|---|---|---|---|---|---|
| 70 | 5 | 50 | 20 | 90 | 5 | 50 | 35 |
| 70 | 10 | 60 | 25 | 90 | 10 | 60 | 40 |
| 70 | 15 | 70 | 30 | 90 | 15 | 70 | 60 |

| Deemulsification time (min) | Concentration (ppm) | Temperatyre(C°) | Separated water(%) | Deemulsification time (min) | Concentration (ppm) | Temperatyre(C°) | Separated water(%) |
|---|---|---|---|---|---|---|---|
| 70 | 20 | 80 | 35 | 90 | 20 | 80 | 70 |
| 70 | 30 | 90 | 40 | 90 | 30 | 90 | 80 |
| Deemulsification time (min) | Concentration (ppm) | Temperatyre(C°) | Separated water(%) | Deemulsification time (min) | Concentration (ppm) | Temperatyre(C°) | Separated water(%) |
| 80 | 5 | 50 | 25 | 100 | 5 | 50 | 20 |
| 80 | 10 | 60 | 37 | 100 | 10 | 60 | 40 |
| 80 | 15 | 70 | 40 | 100 | 15 | 70 | 60 |
| 80 | 20 | 80 | 45 | 100 | 20 | 80 | 80 |
| 80 | 30 | 90 | 50 | 100 | 30 | 90 | 100 |

When applying a demulsifier to oil, the dependence of the demulsifier efficiency on the dynamics of water separation (time), the process temperature and the concentration of the demulsifier used was studied (Table 1). As can be seen from the table, the time taken to reach the optimal demulsification conditions was 100 minutes, and the process temperature was 90°C, and the demulsifier concentration used was 30 ppm. This led to the active adsorption of demulsifier molecules in the stable film layer at the oil-water interface, which led to the destruction of the protective shell created by natural stabilizers (asphaltenes and resins). However, when the concentration exceeded 30 ppm, the emulsions were restabilized. Thus, it was determined that 30 ppm was the optimal demulsifier condition.

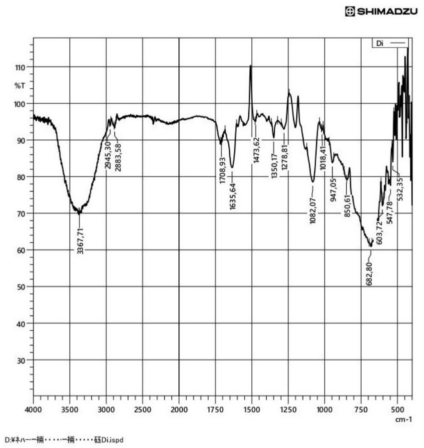


**Fig. 5.** Demulgatorning IQ-spektri / **Fig. 5.** IR spectrum of the demulsifier

The broad and intense absorption band observed at wavenumber 3367.71 cm−1 in the spectrum of the demulsifier sample corresponds to the stretching vibrations of hydroxyl (-OH) groups. The peak at 1708.93 cm−1 indicates the presence of a carbonyl (C=O) group. The signals at wavenumbers 2945.30 cm−1 and 2883.58 cm−1 represent the stretching vibrations of C-H, while the absorption around 1082.07 cm−1 is characteristic of the C-O bond. The peaks around 682.80 cm−1 and 603.72 cm−1 reflect the skeletal vibrations of the molecule.

The surface morphology of the synthesized sample was studied using a scanning electron microscope (SEM) (Figure 7). As can be seen from the SEM image, the sample consists of particles of irregular shape and different sizes, which aggregated to form large structures. The surface of the particles is rough and uneven, and the absence of clear crystalline forms indicates that an amorphous or partially disordered polymer structure was formed during the synthesis process.

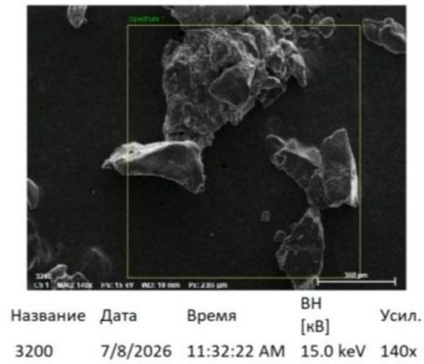


**Fig. 6.** Demulgatorning SEM tahlili / **Fig. 6.** SEM analysis of the demulsifier

The SEM image shows that the particles have a jagged and plate-like appearance and their sizes vary significantly. This indicates that the particles adhered to each other and formed aggregates as a result of intermolecular interactions during the synthesis process. Such a porous and uneven surface structure increases the specific surface area of the material and facilitates the adsorption of molecules at the phase boundary. The SEM method allows for a high-precision assessment of the surface morphology and particle structure of materials and, together with EDS, is widely used in a comprehensive characterization of the morphology and elemental composition of the material.

Such morphology is important from the point of view of breaking oil emulsions. The rough surface and the developed interface area contribute to the faster diffusion of demulsifier molecules to the water-oil boundary. As a result, the destruction of the asphaltene-resin layer stabilizing the emulsion is accelerated and the coalescence of water droplets is facilitated. Therefore, the observed morphological features can serve to increase the demulsification efficiency of the synthesized substance. It has also been noted in the literature that for polymer demulsifiers, it is the surface morphology and interfacial activity that significantly affect their practical effectiveness. In general, the SEM results confirm that the synthesized substance is a heterogeneous polymer material with a developed surface structure.

The elemental composition of the sample was analyzed using energy dispersive X-ray spectroscopy (EDS) (Figure 8). According to the spectral results, it was determined that the sample mainly contains carbon (C), nitrogen (N), oxygen (O), sulfur (S) and silicon (Si) elements.

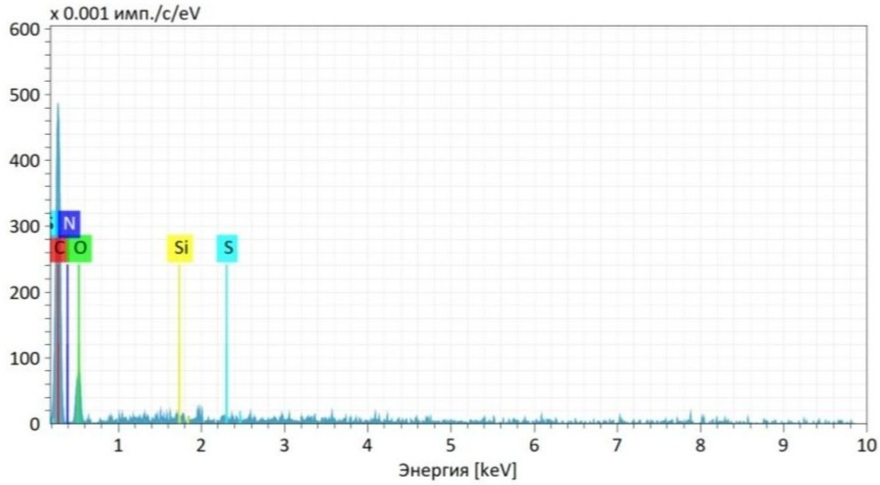


**Fig. 7.** Demulgatorning EDS tahlili / **Fig. 7.** EDS analysis of the demulsifier

The highest intensity of the carbon signal indicates that the synthesized product has an organic polymer skeleton. The presence of oxygen confirms the presence of oxygen-containing functional groups such as hydroxyl, ether, ester or carbonyl in the molecule. The detection of the nitrogen element in the spectrum indicates the successful introduction of nitrogen-containing functional groups of the amine or amide type during the synthesis process. Such groups increase the hydrophilicity of the substance and enhance its interaction with the water phase. As a result, demulsifier molecules are adsorbed more quickly at the water-oil interface, accelerating the breakdown of the emulsion film.

The presence of the sulfur element indicates the presence of sulfur-containing functional groups in the substance. Sulfur atoms can increase the polarity of the molecule and enhance the interaction with asphaltene and resin components that stabilize the oil emulsion. This accelerates the coalescence of water droplets and increases the efficiency of the demulsification process. It has been reported in many studies that heteroatoms (O, N, S) in polymer demulsifiers play an important role in enhancing the interfacial activity.

The silicon (Si) peak observed in the spectrum has a relatively low intensity. This element may originate from the silicon-containing components used in the synthesis, residual mineral impurities, or the substrate used in the SEM sample preparation process. Due to its low intensity, no significant effect of silicon on the main physicochemical properties of the material is observed. The absence of significant peaks related to iron, calcium, or other heavy metals in the EDS spectrum indicates that the synthesized product has a relatively high chemical purity.

Thus, EDS analysis confirms the successful incorporation of carbon, oxygen, nitrogen, and sulfur atoms into the synthesized substance. These elements play an important role in shaping the surface-active properties of the demulsifier and reducing the stability of the water-oil emulsion. The results of SEM and EDS analyses confirmed that the synthesized substance is a heterogeneous polymer material with an uneven and rough surface morphology and contains elements that form surface-active functional groups, such as carbon, oxygen, nitrogen and sulfur. Such morphological and elemental compositional properties can enhance the adsorption of demulsifier molecules to the water-oil interface, accelerate the degradation of the asphaltene-resin film and accelerate the coalescence of water droplets. Therefore, the synthesized substance can be evaluated as a promising demulsifier for the effective breakdown of oil emulsions.

## 4. CONCLUSION

In this scientific work, the high efficiency of polymeric demulsifiers synthesized on the basis of HPEG (hydroxy polyethylene glycol) and acrylic acid in breaking oil-water emulsions was experimentally confirmed. The modified molecular structure of HPEG sharply reduced the surface tension at the oil-water interface, demonstrating higher performance compared to traditional demulsifiers in disrupting the stability of the emulsion. Experiments have shown that the optimal concentration of the synthesized demulsifier is 30 ppm, the time required for complete demulsification is 100 minutes and the temperature is 80-90 0 C. These parameters significantly accelerate the process of oil dehydration and reduce energy consumption. The demulsifier synthesized on the basis of HPEG and acrylic acid is considered a new generation of demulsifiers, increasing technological efficiency, significantly reducing energy and time consumption at oil refineries, and at the same time fully meeting the requirements of environmental safety. This indicates their potential for widespread use in the processing of heavy and highly viscous oil types, as well as in primary preparation processes in mining conditions.

## DECLARATIONS

### Author contributions *CRediT*

Conceptualization; Methodology; Investigation; Data curation; Writing – original draft; Writing – review & editing. All authors have read and agreed to the published version of the manuscript.

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### Ethics approval

Not applicable. This study did not involve humans, animals or other regulated materials.

### Informed consent

All authors have read and approved the final version of the manuscript and have given their consent to its submission and publication. The study involved no human participants and no personal data.

### Data availability statement

All data supporting the findings of this study are presented in the article and are available from the corresponding author upon reasonable request.

### Conflict of interest

The authors declare no conflict of interest.

### Generative AI disclosure

Generative AI was used only partially. The authors used Claude (Anthropic, Opus 5) for language editing, formatting of the reference list and preparation of the English and Russian metadata. No text, data, figures or conclusions were generated by AI. The authors reviewed and verified all output and take full responsibility for the content of the publication.

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*References are numbered in order of first appearance in the text. Give all authors up to six; a DOI must be provided where one exists.*

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