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## SYNTHESIS OF OLIGOMER BASED ON SODIUM POLYSULFIDE AND HYDROLYZED ED-20 EPOXY RESIN: STRUCTURE AND PROPERTIES

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### Abstract

This article studies the process of oligomer synthesis based on sodium polysulfide, hydrolyzed ED-20 epoxy resin and 1,1,2-trichloroethane. The reaction mechanism, synthesis conditions and physicochemical properties of the product are analyzed. Based on the results of scientific research, the dependence of the oligomer formation process on temperature, reaction time, and reagent concentration was determined. UV-spectral analysis of the obtained sodium polysulfide oligomer (UV-Vis spectrometer range 350–800 nm, maximum reflectance – 67.02 %, light absorption – 323 nm), IR spectra, elemental analysis were conducted. The electron density surface (ESP) of the synthesized oligomer based on sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane optimized at the B3LYP/6-31G(d) level is presented in the GAUSSIAN 16 program. The stability, physicochemical properties, and functional capabilities of the synthesized oligomers indicate that they have high prospects for industrial production processes, especially for the preparation of hermetic materials, coatings, and composites, as well as for the creation of new materials in scientific research.

**Keywords:** oligomer synthesis; 1,1,2-trichloroethane; hydrolyzed ED-20 epoxy resin; sodium polysulfide; reaction kinetics; physicochemical properties.

## СИНТЕЗ ОЛІГОМЕРУ НА ОСНОВІ ПОЛІСУЛЬФІДУ НАТРІЮ ТА ГІДРОЛІЗОВАНОЇ ЕПОКСИДНОЇ СМОЛИ ЕД-20: СТРУКТУРА ТА ВЛАСТИВОСТІ

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### Анотація

У статті досліджений процес синтезу олігомеру на основі полісульфіду натрію, гідролізованої епоксидної смоли ЕД-20 та 1,1,2-трихлоретану. Проаналізований механізм реакції, умови синтезу та фізико-хімічні властивості продукту. За результатами наукових досліджень встановлена залежність процесу утворення олігомеру від температури, часу реакції та концентрації реагентів. Проведений УФ-спектральний аналіз отриманого олігомеру полісульфіду натрію (діапазон УФ-спектрометра 350–800 нм, максимальне відбивання – 67.02 %, світлопоглинання – 323 нм), отримані ІЧ-спектри, елементний аналіз. Поверхню електронної густини (ESP) синтезованого олігомеру на основі полісульфіду натрію, гідролізованого ЕД-20 та 1,1,2-трихлоретаном, оптимізовано на рівні B3LYP/6-31G(d), представлено в програмі GAUSSIAN 16. Стабільність, фізико-хімічні властивості та функціональні можливості синтезованих олігомерів свідчать про їх високу перспективність для промислового виробництва, особливо для одержання герметичних матеріалів, покриттів і композитів, а також для створення нових матеріалів у наукових дослідженнях.

**Ключові слова:** синтез олігомерів; 1,1,2-трихлоретан; гідролізована епоксидна смола ЕД-20; полісульфід натрію; кінетика реакції; фізико-хімічні властивості.

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## Introduction

In recent years, the demand for the production of high-performance polymer-based materials has been increasing in the chemical industry. In particular, oligomers – molecules with low molecular weight, consisting of repeating units – are of great importance as a transition stage for polymer materials. The physicochemical and structural properties of oligomers directly affect the functional properties of the final polymer synthesized from them. Therefore, the synthesis of oligomers of new composition, determination of their structure and optimization of reaction conditions are one of the current areas of scientific research [1].

R. Dallaev and his colleagues studied the main properties, chemical modification and application of epoxy resins [2]. The authors showed that epoxy resins, in particular ED-20 resins, are widely used for the synthesis of polymers with high reactivity and mechanical stability [3]. The hydrolyzed form of these resins, due to the presence of active hydroxyl groups in their composition, is prone to interaction with chemical compounds. Sodium polysulfide, due to the presence of reactive polysulfide groups, can enter into a wide range of reactions with organic and inorganic compounds [4].

The authors studied the degradation of epoxy resins under high electric field and thermal stress conditions using molecular dynamics. Gavrielides A. et al. presented an experimental and simulation-based model of the DGEBA-EDA epoxy polymer [5]. Patil Deepak M. and Phalak Ganesh A. studied the synthesis of bio-based epoxy resins derived from gallic acid and their effect on coating properties [6]. W. Gao and his colleagues studied the synthesis and properties of self-healing and recyclable sealants based on liquid polysulfide oligomers and epoxy resins [6]. C. Probst discussed the self-healing properties of polysulfide polymers due to their high sulfur content and their practical applications [7]. E. Edan studied the mechanical properties of composite materials reinforced with epoxy-polysulfide copolymers and silicon carbide powder [8].

M. Arslan studied the synthesis and properties of sulfur-rich polymers based on polysulfide salts and bisepoxides derived from elemental sulfur [9]. K.-X. Chen and co-workers analyzed the synthesis and dynamic properties of sulfur-rich polymers based on elemental sulfur and epoxides [10]. S. Guchhait performed kinetic modeling and

degradation analysis based on liquid polysulfide resin and clay nanoparticles [11]. V. Burkhardt analyzed the application of liquid polysulfide polymers as chemical and solvent-resistant sealants [12]. D. K. Pradhan studied the thermophysical and mechanical properties of epoxy polysulfide nanocomposites [13]. US Patent No. 5,610,243 describes the synthesis and properties of thermoplastic elastomers based on epoxy resins and liquid mercapto-unfilled polysulfides [14].

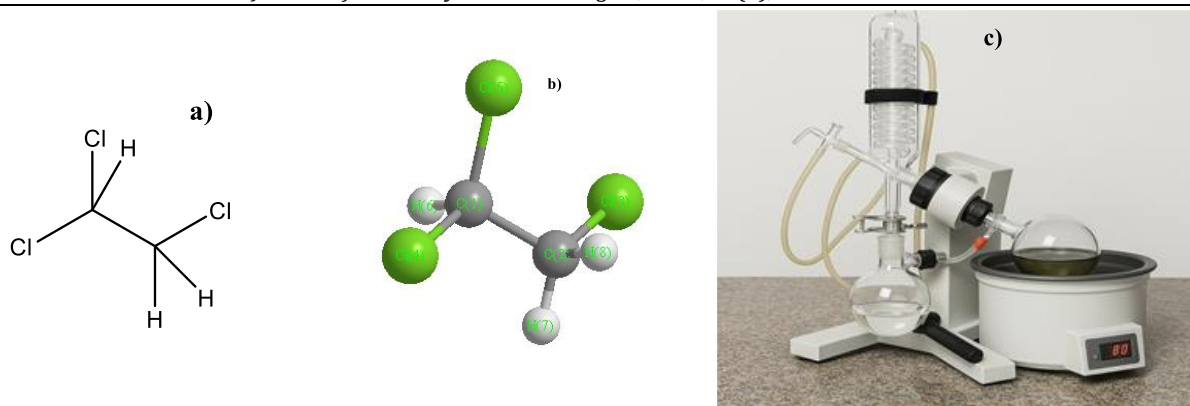
The research on the self-healing ability of epoxy polymers is reviewed. The article discusses the mechanisms of recovery through addition to the material and energy impact [15]. Recent advances in self-healing composite materials are reviewed. Sandra Milev studied the application of self-healing sealants based on liquid polysulfide and epoxy resin in transportation infrastructure. The recovery ability and mechanical properties of the materials are reviewed [16]. Paolillo S. et al. analyzed the mechanisms of internal self-healing based on epoxy in polymer matrix composites. They discussed suitable formulations for the design of high-performance composites [17]. Zhang, F. in their scientific works provided information on the self-healing properties, mechanisms and practical applications of epoxy composites [18].

There are scientific works on the synthesis of oligomers based on sodium polysulfide and epichlorohydrin, the research in this area is mainly focused on the synthesis of polysulfide sealant components. An in-depth study of the physical and chemical properties of hydrolyzed ED-20 epoxy resin and oligomers based on sodium polysulfide allows for the development of new approaches science in the field of materials science [19]. To date, the synthesis and structure of oligomers have been studied, as well as their application as anti-corrosion agents, sorbents, thickeners for the textile industry, and adhesive materials [20]. However, unlike previous studies, the present research focuses on the structure and properties of a novel oligomer synthesized from hydrolyzed ED-20 epoxy resin and 1,1,2-trichloroethane. The possibility of using this oligomer as an effective coagulant in the future is also being considered.

## Materials and Methods

### *Materials and Reagents*

1,1,2-Trichloroethane ( $C_2H_3Cl_3$ ) is the main starting material for the reaction (isolation process) (Fig. 1).



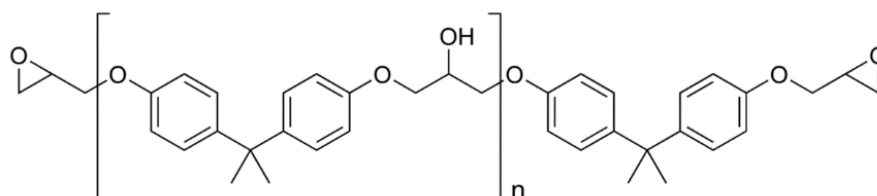
**Fig. 1. Structural models and laboratory equipment related to 1,1,2-Trichloroethane. a) Lewis's structure of 1,1,2-Trichloroethane; b) Space-filling molecular model; c) Rotary evaporator used in the isolation process**

Table 1

| Selected physical properties of 1,1,2-Trichloroethane |                                   |
|-------------------------------------------------------|-----------------------------------|
| Indicator                                             | Value                             |
| Aggregate Status                                      | Colorless liquid                  |
| Molar mass                                            | 133.4 g/mol                       |
| Density                                               | $1.44 \pm 0.01$ g/sm <sup>3</sup> |
| Surface tension                                       | 0.03357 N/m                       |
| Boiling temperature                                   | 113.9 °C                          |
| Flash point                                           | 29 °C                             |

Secondary products formed during the production of polyvinyl chloride at Navoiazot JSC were isolated from a mixture of chlorinated organic compounds.

*ED-20 epoxy resin* was used as a source of functional groups.



*ED-20* consists of one bisphenol molecule and two epoxide groups. One epoxide group has a molecular mass of 45 g/mol, and two epoxide

groups have a molecular mass of 90 g/mol. The total molecular mass is 350 g/mol (Table 2).

Table 2

| Physicochemical properties of ED-20 epoxy resin         |                      |                                 |
|---------------------------------------------------------|----------------------|---------------------------------|
| Indicator                                               | Top grade            | First grade                     |
| Appearance Viscous                                      | Viscous, transparent | Malleable, yielding transparent |
| Mass fraction of epoxide groups, %                      | 20–22.5              | 20.2–22.5                       |
| Mass fraction of chlorine ion, %, max.                  | 0.001                | 0.005                           |
| Mass fraction of esterifiable chlorine, %, max.         | 0.3                  | 0.8                             |
| Mass fraction of hydroxyl groups, %, max.               | 1.7                  | -                               |
| Mass fraction of volatile substances, %, max.           | 0.2                  | 0.8                             |
| Dynamic viscosity, Pa*s, at (25±0.1) °C and (50±0.1) °C | 13–20                | 12–25                           |
| Gelatinization time, hours, at least                    | 8                    | 4                               |

*Sodium polysulfide* ( $\text{Na}_2\text{S}_x$ ) is used to form sulfide bonds. Sodium polysulfide ( $\text{Na}_2\text{S}_x$ ) was synthesized in the scientific laboratory of Bukhara State University based on  $\text{S}_x$ , a by-product of the Mubarak Gas Processing Plant, and NaOH produced at Navoiazot JSC. The procedure was as

follows: 60 ml of distilled water and 60 g of NaOH were placed in a 500-ml flask at a temperature of 40 °C. Then, with vigorous stirring, 144 g of  $\text{S}_x$  was added. Then the temperature was raised to 85–90 °C, and after 1–1.5 hours, a homogeneous solution was formed (Fig. 2).



Fig. 2. Equipment and reaction mixture during the synthesis of the resin:

a) Mechanical stirrer with temperature controller; b) Reaction flask containing the dark brown resin solution.

The value of  $x$  in the  $S_x^{2-}$  ion is 4-6, UV-Vis Spectroscopy – Electronic transitions in delocalized  $S_6^{2-}$  chains have intense absorption in the range of 520–550 nm.

### Experimental methods

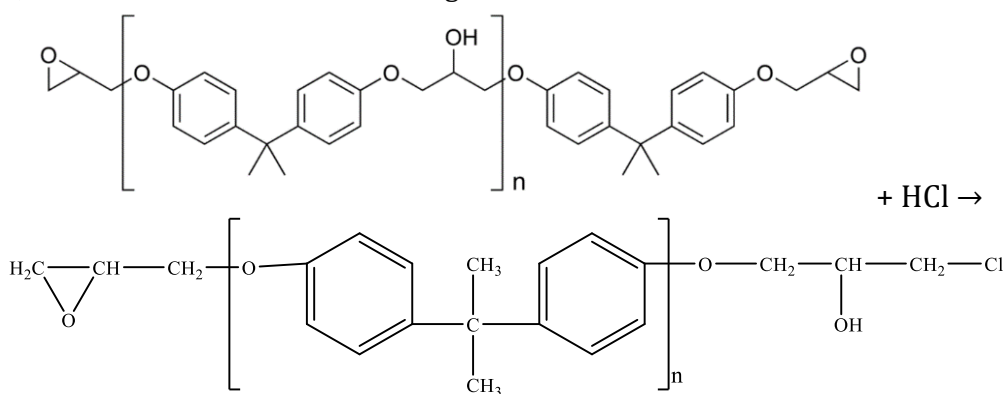
A rotary evaporator (IKA RV 10, Germany), a magnetic stirrer (VELP Scientifica, Italy), and a laboratory hot plate (Heidolph, Germany) were used during the synthesis process. Experimental studies were carried out in laboratory conditions. Different ratios of 1,1,2-trichloroethane, ED-20 epoxy resin and sodium polysulfide were selected for the oligomer synthesis process. The synthesis conditions, including temperature, reaction time and pH of the medium, were controlled. The resulting

products were characterized by infrared spectroscopy using a SHIMADZU Fure-10 infrared spectrometer (Japan) in the range of 600–4000  $\text{cm}^{-1}$ .

Oligomer synthesis: Oligomer synthesis was carried out in four stages.

*First stage:* Sodium polysulfide was synthesized in alkaline medium from plastic sulfur, a by-product of the Mubarak gas processing plant.

*Second stage:* ED-20 epoxide and hydrochloric acid were mixed in a 1 : 1 molar ratio (temperature: 40–60 °C, solution pH  $\approx$  4–5, reaction time 40 minutes). The reaction equation can be expressed as follows (Scheme 1):



Scheme 1. Synthesis pathway of epoxy-based oligomers

In this process, some of the epoxy rings are opened and chlorohydrin is formed.

*Third stage:* 100 g of the polysulfide solution was poured into a 3-necked flask equipped with special devices (Libex refrigerator, thermometer, hot plate with

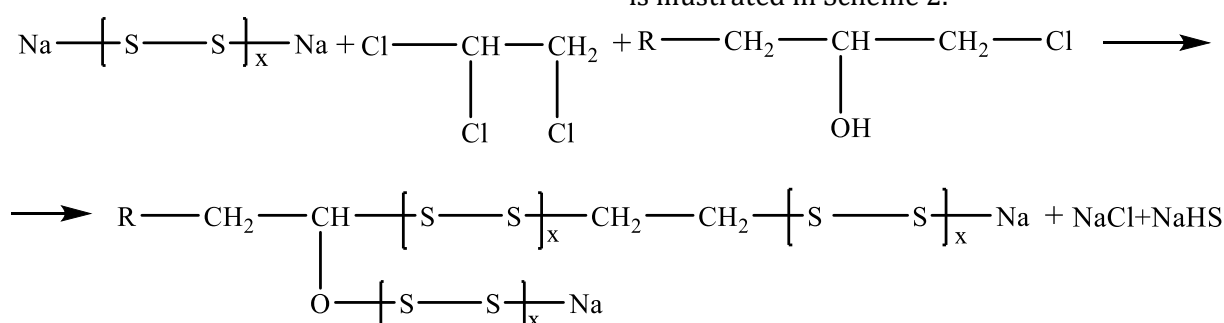
magnetic stirrer, dispenser), the resulting mixture was heated to 40–50 °C and stirred for 15 minutes. The stirring process ensures the homogeneity of the medium and serves as a preparation for subsequent processes (Fig. 3).



**Fig. 3. Experimental setup used during the third stage of the synthesis process: a three-necked flask equipped with a magnetic stirrer, thermometer, and reflux condenser**

*Fourth stage:* In the third stage, 20 g of a mixture of 1,1,2-trichloroethane and hydrolyzed ED-20 (taken in a 1 : 2 mass ratio) was added dropwise to the sodium polysulfide solution. The temperature was maintained at 80–90 °C for 1 hour. At this stage, the hydrolyzed ED-20 and 1,1,2-trichloroethane interact with sodium polysulfide, and the oligomerization process begins.

*Reaction mechanism.* During the reaction, 1,1,2-trichloroethane and ED-20 form polysulfide chains through epoxy-like halogen exchange and activation of functional groups. As a result, the bonds are mainly in the form of C–S, C–O, and S–S, which increases the mechanical properties of the oligomer. The interaction of sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane can be described as follows. The proposed mechanism is illustrated in Scheme 2.



**Scheme 2. Proposed reaction mechanism between sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane leading to the formation of a polysulfide-based oligomer**

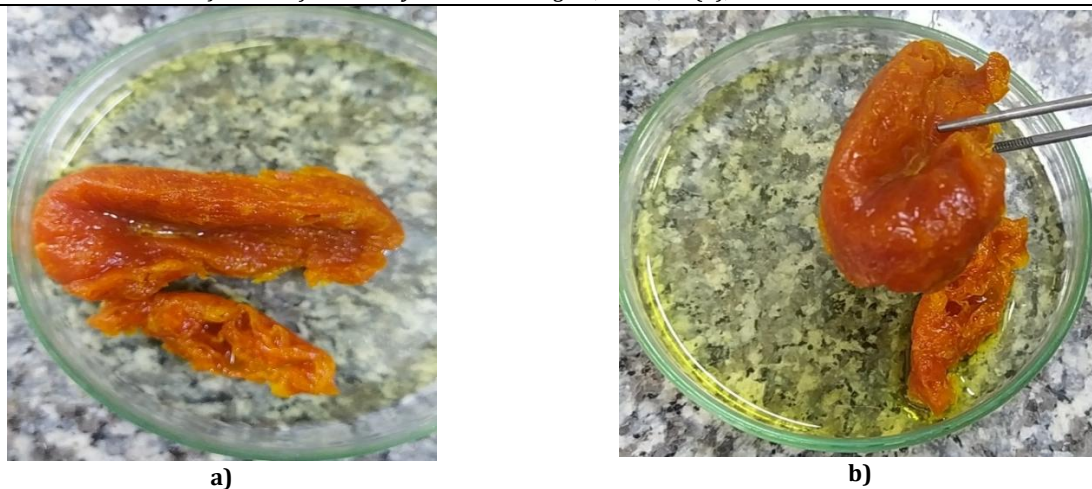
A substance synthesized on the basis of sodium polysulfide, hydrolyzed ED-20 and 1,1,2-trichloroethane

### Results and their analysis

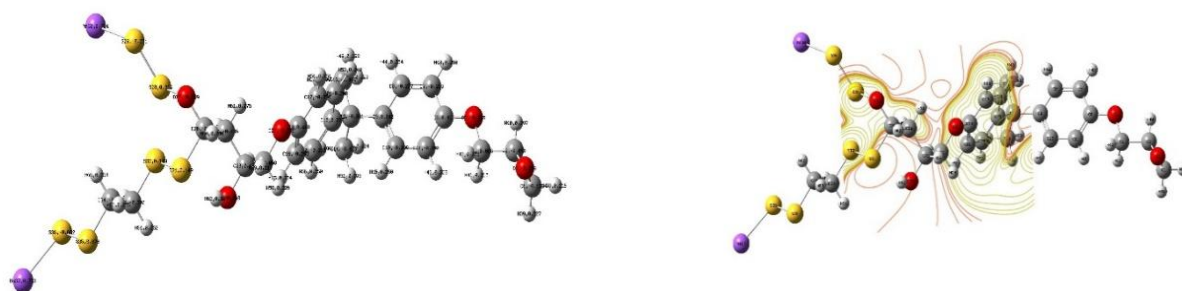
A new type of oligomer was formed as a result of the synthesis based on sodium polysulfide and hydrolyzed ED-20 epoxy resin. The optimal reaction conditions during the synthesis process, including temperature, duration and sodium

polysulfide concentration, were determined (Fig. 4).

The model in this image is a 3D visualization of the electron density distribution of the substance formed as a result of the oligomerization reaction based on sodium polysulfide and hydrolyzed ED-20 resin. This visualization displays the electron density calculated using the GAUSSIAN 16 program through a color gradient (Fig. 5).



**Fig. 4.** Synthesized oligomer based on sodium polysulfide, hydrolyzed ED-20 and 1,1,2-trichloroethane (a- reaction at 90 °C for 30 minutes, b- reaction at 90 °C for 60 minutes)



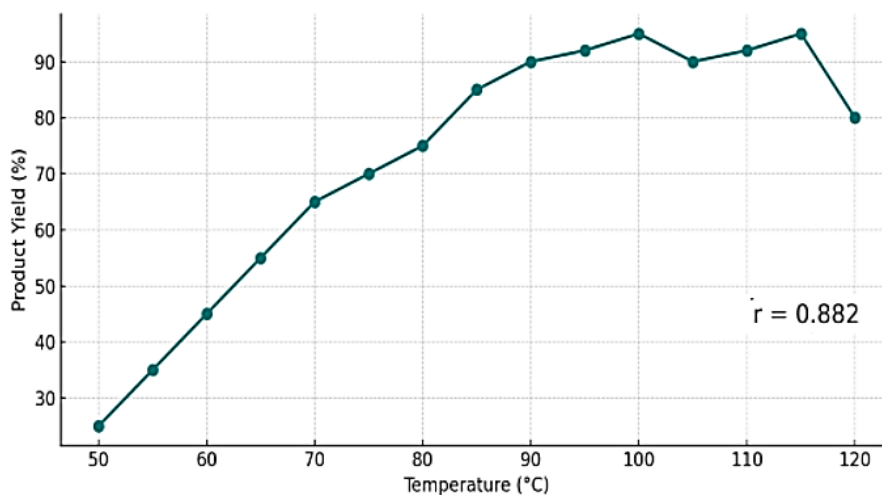
**Fig. 5.** Electron density surface (ESP) image of the synthesized oligomer based on sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane, optimized at the B3LYP/6-31G(d) level

This figure represents the electron density surface (ESP) of an oligomer synthesized from sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane, optimized at the B3LYP/6-31G(d) level in the GAUSSIAN 16 program. The oligomer consists of rigid and stretchable segments, which give it flexibility and thermal stability. The red areas with high electron density are usually found around oxygen atoms (e.g., OH, O-Na). They indicate areas where electrons are densely packed due to high electronegativity. The yellow-green (medium density) areas are often observed around C-C bonds or C-S bonds. These segments act as the backbone of the molecule and provide structural stability (Fig. 5).

The blue (low electron density) regions are often located around hydrogen atoms or less

reactive sites. Such regions are located on the periphery of the molecule and have low reactivity. The chemically active part, which contains the -O-Na and -O-H groups, has high density, indicating a high tendency for nucleophilic reactions. -S-S- bonds also have strong electron clouds and are actively involved in oxidation or other substitution reactions.

It was found that the yield of oligomer synthesis depends on the reaction conditions (Fig. 6). With increasing temperature, a change in molecular mass and an increase in the degree of polysulfide bonding were observed. Changes in concentration and pH level affected the physic mechanical properties of the product. The results in the form of graphs and tables showed that the composition and properties of the product depend on the reaction conditions.

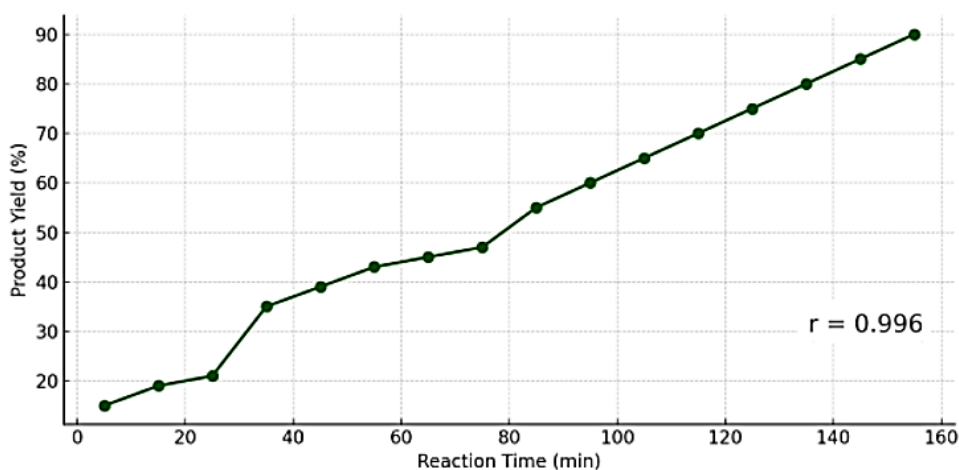


**Fig. 6. Effect of temperature on product yield**

(20 g of a mixture of 1,1,2-trichloroethane and hydrolyzed ED-20 (1 : 2 mass ratio) was added dropwise to 100 g of sodium polysulfide solution)

As can be seen from the figure, the product yield increases with increasing temperature. It shows that the yield of oligomer formation at 50 °C increased by 65 %, and at 80 °C by 90 %. At temperatures above 90 °C, a

decrease in product yield was observed, indicating a correlation with the boiling point of the chlorinated organic compound at high temperatures (Fig. 7).



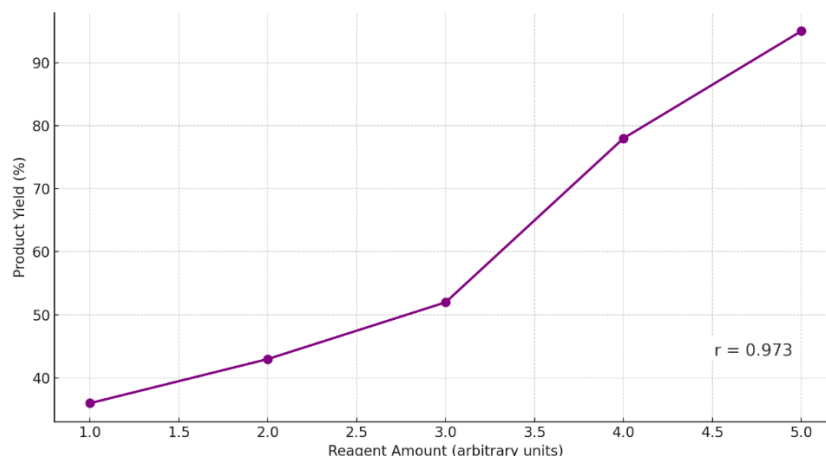
**Fig. 7. Effect of time on product yield**

(20 g of a mixture of 1,1,2-trichloroethane and hydrolyzed ED-20 (1:2 mass ratio) was added dropwise to 100 g of sodium polysulfide solution. The temperature was 100°C)

The figure shows how the reaction time affects the product yield. The reaction rate slows down after 155 minutes and does not change at the yield level after 160 minutes. This helps to determine the optimal range for the reaction.

As the reaction time increases, the product yield increases almost linearly and steadily. This

high correlation  $r = 0.996$  indicates that reaction time is one of the main factors affecting the yield. 90 % yield is achieved in ~155 minutes — this may be the time required for the reaction to complete (Fig. 8).



**Fig. 8. Effect of concentration on product yield**

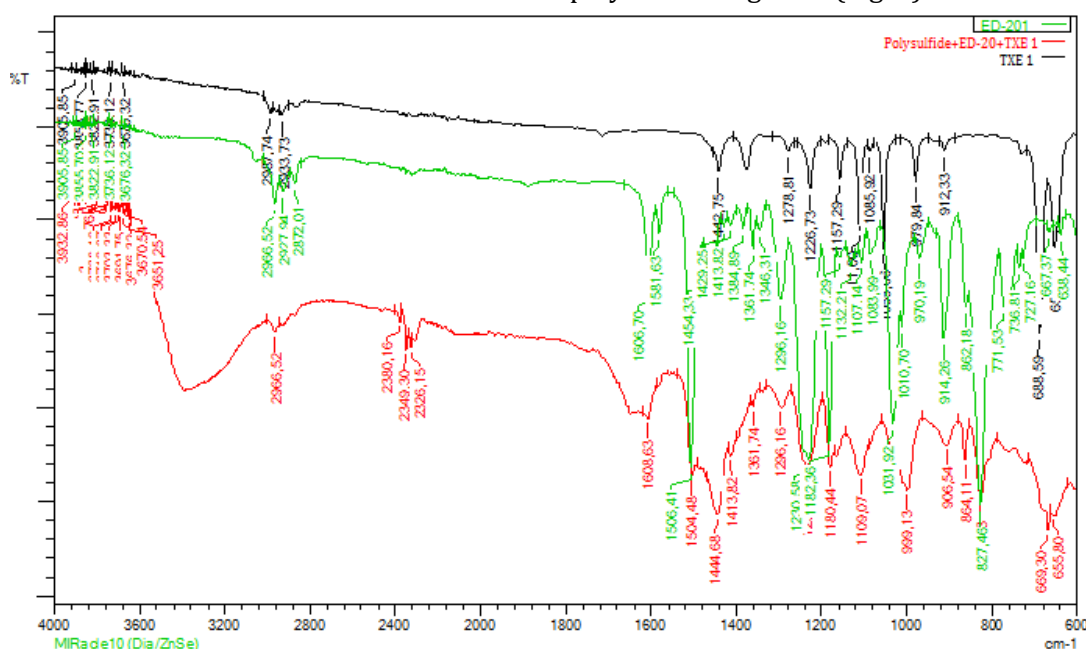
(20 g of a mixture of 1,1,2-trichloroethane and hydrolyzed ED-20 (taken in a 1:2 mass ratio) was added dropwise to 100 g of sodium polysulfide solution. The temperature was maintained at 100 °C for 1 hour)

This figure shows how the concentration of reagents affects the yield of the product during the oligomer synthesis process. It shows the effect of the amount of reagent on the yield of the oligomer product in the synthesis based on sodium polysulfide, 1,1,2-tetrachloroethane and hydrolyzed ED-20 epoxy resin. As the amount of reagent increased from 1 to 5 units, the yield of the product increased from 36 % to 95 %. The Pearson correlation coefficient ( $r = 0.973$ ) shows the statistical reliability of this relationship. This means that the formation of oligomers was more efficient as the amount of reagent increased. It also indicates that this is an important factor in optimizing the efficiency of the reaction.

IR spectroscopy, NMR spectroscopy and elemental analysis methods were used to determine the chemical structure of the obtained

oligomers. According to the results of IR spectroscopy, absorption lines characteristic of hydroxyl (-OH), ether (C-O-C) and sulfide (S-S) groups were detected in the spectra of the oligomers.

In the IR spectra of the synthesized oligomer, characteristic absorption lines were observed in the following regions.  $2966.52\text{ cm}^{-1}$  – CH- group,  $1608.63\text{--}1504.48\text{ cm}^{-1}$  – Aromatic group,  $1444.66\text{ cm}^{-1}$  –  $\text{CH}_2$  group,  $1361.74\text{ cm}^{-1}$  – C-O-C group,  $1296.16\text{--}1244.44\text{ cm}^{-1}$  – epoxy group vibrations,  $1180.47\text{--}1108.07\text{ cm}^{-1}$  – C-O-S-,  $999.13\text{--}906.54\text{ cm}^{-1}$  –  $\text{CH}_2$  bonds,  $825.53\text{--}841.11\text{ cm}^{-1}$  – Polysulfide group (-S-S-),  $669.30\text{--}655.80\text{ cm}^{-1}$  – valence vibrations of ring sulfur groups are observed. Analysis of the obtained results confirms the formation of a sulfur-based polysulfide oligomer (Fig. 9).



**Fig. 9. IR spectra of the oligomer based on sodium polysulfide (Polysulfide), hydrolyzed ED-20(ED-20) and 1,1,2-trichloroethane (TXE).**

The elemental analysis of the synthesized oligomer was carried out on a Rigaku NexDE VS (Rigaku, Japan). Using this instrument, it is important to determine the chemical elements

and their quantitative composition in the oligomer. The results of the analysis are presented in Fig. 10.

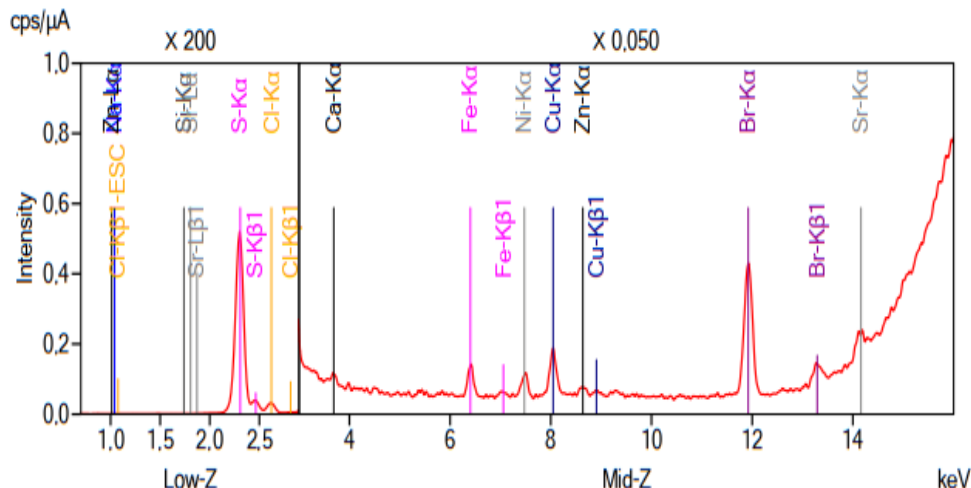


Fig. 10. Elemental analysis of an oligomer based on sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane

The results of the analysis show that the sulfur content in the sample obtained is higher than 78.5 %, which confirms the presence of a large proportion of sodium polysulfide oligomer. Polysulfides are usually linked through S-S bonds and have hydrophobic and elastic properties. The fact that the sodium content in the oligomer is 14.6 % indicates the presence of -S-S-Na bonds in the substance. The aggregate state of sodium polysulfide in most cases forms a polymer in the form of a liquid or gel. The chlorine content in its

composition is 6.7 %, which indicates that the chlorine atoms contained in 1,1,2-trichloroethane are preserved in the oligomer. The importance of chlorine in the oligomer is that it acts as a stabilizer that ensures the thermal stability of the substance during polymerization. The presence of the following elements in the oligomer composition: Si (silicon), Fe (iron), Ni (nickel), Br (bromine), Cu (copper), Zn (zinc), Sr (strontium) occurs in the sulfur compound due to the catalysts and reagents used in the gas processing process.

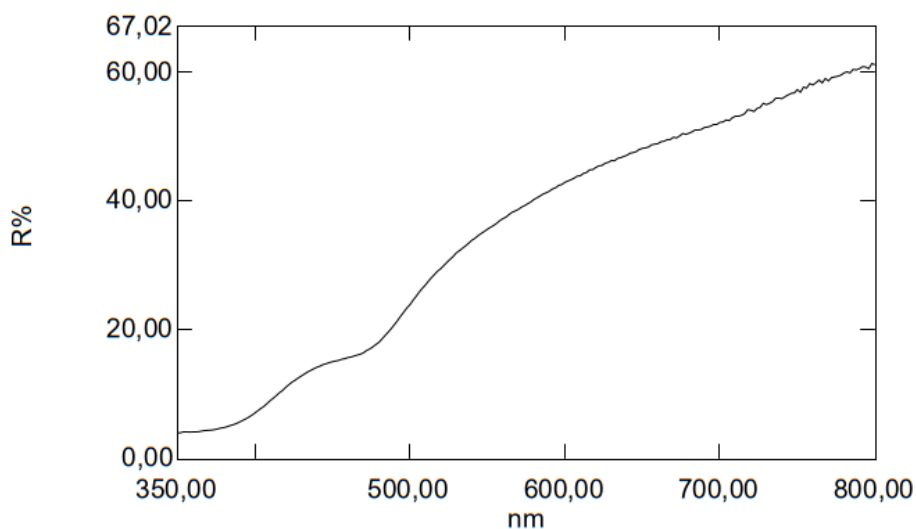


Fig. 11. UV spectrum of an oligomer based on sodium polysulfide, hydrolyzed ED-20, and 1,1,2-trichloroethane

UV-spectral analysis of the obtained sodium polysulfide oligomer (UV-Vis spectrometer range 350–800 nm, maximum reflectance – 67.02 %, light absorption – 323 nm) was performed (Fig. 11). The obtained spectral analysis results show that the oligomer has a maximum absorption

region of light, which indicates that the polymer structure is formed correctly. The absorption region of 280–350 nm confirms the formation of sulfide groups (-S-S- bonds) in sodium polysulfide. The absorption region of 300–400 nm indicates

the formation of derivatives containing aromatic rings in the oligomer.

## Conclusion

In this study, a new oligomer based on sodium polysulfide, hydrolyzed ED-20 epoxy resin and 1,1,2-trichloroethane was successfully synthesized. The effects of temperature, time, pH environment and reagent concentration on the oligomer synthesis process were studied, and

optimal technological conditions were determined. The physicochemical properties of the synthesized oligomer were characterized by UV-Vis spectroscopy, IR-spectral analysis, elemental analysis and electron density surface (ESP) using the GAUSSIAN 16 program. The oligomer exhibited high elasticity, chemical stability, good mechanical strength and self-healing ability.

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