

STUDY OF THE STRUCTURE OF OLIGOMERS OBTAINED FROM SODIUM POLYSULFIDE EPICHLOROHYDRIN USING SPECTRAL METHODS

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ANNOTATION

The study explores the synthesis and structural characterization of oligomers obtained based on sodium polysulfide and epichlorohydrin, using modern spectral analysis methods. The main goal is to determine the molecular composition, functional group transformations, and structural features of synthesized oligomers through IR spectroscopy, UV-Vis spectroscopy, and NMR analysis. The results confirm that the polymerization reaction proceeds through nucleophilic substitution, resulting in the formation of flexible oligomeric chains containing polysulfide and ether fragments. The obtained data contribute to the understanding of the structure–property relationship in sulfur-containing oligomers and provide a foundation for their application in corrosion protection and composite materials.

KEYWORDS: sodium polysulfide, epichlorohydrin, oligomer, IR spectroscopy, NMR, spectral analysis, polymer structure, sulfur-containing polymer.

АННОТАЦИЯ

В работе изучены синтез и структурная характеристика олигомеров на основе полисульфида натрия и эпихлоргидрина с использованием современных методов спектрального анализа. Основная цель исследования – определение молекулярного состава, трансформаций функциональных групп и структурных особенностей синтезированных олигомеров методами ИК-спектроскопии, УФ-видимой спектроскопии и ЯМР-анализа. Результаты подтверждают, что реакция полимеризации протекает по механизму нуклеофильного замещения, приводящего к образованию гибких олигомерных цепей, содержащих полисульфидные и эфирные фрагменты. Полученные данные способствуют пониманию взаимосвязи между структурой и свойствами серосодержащих олигомеров и создают основу для их применения в антикоррозионной защите и композиционных материалах.

КЛЮЧЕВЫЕ СЛОВА: полисульфид натрия, эпихлоргидрин, олигомер, ИК-спектроскопия, ЯМР, спектральный анализ, структура полимера, серосодержащий полимер.

INTRODUCTION

Modern polymer chemistry increasingly focuses on the synthesis of functionalized sulfur-containing oligomers with unique physical and chemical properties. Such oligomers exhibit high **thermal stability, chemical resistance, and dielectric characteristics**, making them valuable in various industrial sectors, including **corrosion protection, composite materials, and adhesives**.

Among these, **oligomers synthesized from sodium polysulfide and epichlorohydrin** attract attention because the combination of **–S–S– bonds** with **ether linkages (–O–)** gives rise to flexible, chemically resistant polymeric structures. The formation of such compounds occurs via nucleophilic substitution reactions between the chloromethyl group of epichlorohydrin and the polysulfide anion.

However, understanding their **structural composition** is crucial for predicting performance. Therefore, this study utilizes **spectral analytical methods (IR, NMR, UV-Vis)** to reveal molecular features and confirm the chemical structure of the obtained oligomers.

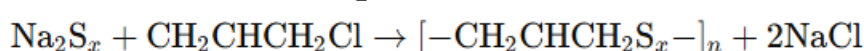
MAIN PART

1. Materials and Methods

- **Reagents:** Sodium polysulfide (Na_2S_x , $x=2-4$), epichlorohydrin ($\text{C}_3\text{H}_5\text{ClO}$), distilled water, and ethanol as solvent.
- **Reaction Conditions:** The reaction was carried out at $60-70^\circ\text{C}$ for 3 hours with continuous stirring under nitrogen atmosphere. The resulting viscous product was washed, dried, and purified via ethanol extraction.
- **Analytical Methods:**
 - **IR spectroscopy** ($4000-400\text{ cm}^{-1}$) – identification of functional groups.
 - **^1H NMR spectroscopy** – determination of hydrogen environments and polymer chain formation.
 - **UV-Vis spectroscopy** – evaluation of electronic transitions and conjugation effects.

2. Reaction Mechanism and Formation of Oligomers

The synthesis follows a **nucleophilic substitution mechanism**:



This reaction leads to the incorporation of **polysulfide linkages** and ether bridges into the polymer backbone. The flexibility and elasticity of the resulting product depend on the length of the polysulfide chain (x value).

3. Spectral Analysis Results

Table 1. IR spectral data of synthesized oligomer

Wavenumber (cm ⁻¹)	Functional group	Interpretation
3430–3450	–OH stretching (terminal)	Residual hydroxyl groups
2920–2850	C–H (aliphatic)	Backbone hydrocarbon vibrations
1120–1080	C–O–C stretching	Ether linkage formation
650–550	S–S and C–S stretching	Polysulfide chains

IR spectra confirm the presence of ether (C–O–C) and polysulfide (S–S, C–S) bonds, indicating the success of the nucleophilic substitution reaction.

Table 2. ¹H NMR spectral data of synthesized oligomer

Chemical shift (δ, ppm)	Proton type	Assignment
3.4–3.8	–CH ₂ –O–	Ether protons
2.5–2.8	–CH ₂ –S–	Sulfide-bound methylene protons
1.2–1.5	–CH ₂ –CH ₂ –	Aliphatic chain protons

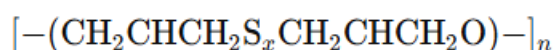
¹H NMR analysis confirms that the reaction leads to the substitution of chlorine atoms by polysulfide anions, forming –CH₂–S– linkages. The broadening of peaks indicates polymer chain formation.

UV-Vis spectra reveal weak absorption bands around 290–320 nm, corresponding to n→σ* transitions typical of sulfur–oxygen bonds. This confirms partial delocalization and the conjugation effect of S–S and C–O linkages in the polymer chain.

4. Structural Interpretation

The combined spectral data indicate the formation of an **oligomeric polysulfide-ether structure**.

The general structural formula can be represented as:



This structure combines flexibility (due to ether groups) and chemical resistance (due to polysulfide bonds). The materials exhibit good solubility in polar solvents and potential for crosslinking under thermal curing.

CONCLUSION

The conducted study demonstrates that **sodium polysulfide and epichlorohydrin** can effectively form sulfur-containing oligomers with a combination of ether and polysulfide fragments.

Spectral analyses (IR, NMR, UV-Vis) confirm the **formation of –C–S–S– and –C–O–C– linkages**, indicating successful nucleophilic substitution and oligomerization. The synthesized oligomers possess structural flexibility and chemical stability, making them promising materials for **anticorrosion coatings, sealing compounds, and composites**.

Future work should include thermal analysis (TGA, DSC) and molecular weight determination (GPC) to further elucidate physical characteristics.

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