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SYNTHESIS OF ORGANOSILICINE OLIGOMER BASED ON SODIUM SILICATE

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Abstract

In this work, a study was conducted on the synthesis of high molecular weight organosilicon oligomers such as oligoethylsiloxane based on 1,2-dichloroethane and sodium metasilicate. The synthesis was carried out via a three-step nucleophilic displacement and condensation reaction in the presence of ethylene glycol, in a dimethylsulfoxide (DMSO) solvent, and in an autoclave using potassium hydroxide as a catalyst. The structure of the resulting oligomer was confirmed by FTIR analysis, and its thermal stability was determined by TGA/DTA. The resulting oligomer has hydrophobic properties and can be used in the separation of oil-water mixtures and in the biomedical field.

Keywords: *1,2-dichloroethane, sodium silicate, ethylene glycol, organosilicon oligomer, oligoethylsiloxane, nucleophilic substitution, condensation reaction, dimethylsulfoxide (DMSO), potassium hydroxide (KOH), autoclave synthesis, thermal stability, hydrophobicity, FTIR spectroscopy, TGA/DTA, environmental safety*

1. Introduction

In recent years, the separation of oil-water mixtures has become a pressing global issue due to the severe environmental damage caused by oil pollution. To address this problem, various functional materials, particularly polyurethane and silicon-based nanocomposites, have been successfully developed. The affordability, durability, and excellent mechanical properties of these materials have further increased interest in them (Liu, Lb & Guo, Gailan & Dang, Zhao & Wenyuan, Fang. 2017). Silicon (IV) oxide nanoparticles, synthesized from materials

such as sodium silicate or tetraethoxysilane (TEOS) using the sol-gel method, are widely used to create hydrophobic and superhydrophilic surfaces. In this process, the influence of electrolytes on particle morphology, as well as the roles of surfactants and solvents, are of significant importance (Valdez salas, Benjamin & Salazar Navarro, Alexis. 2022, 7). For instance, amorphous silicon nanoparticles have been synthesized from sodium silicate in a water-ethanol mixture with low ethanol concentration, controlling the condensation rate, and the process was stabilized by boiling at 95°C for one hour, which does not require high temperatures ((Valdez salas, Benjamin & Salazar Navarro, Alexis. 2022). Additionally, hydrophobic silicon nanoparticles have been synthesized using sodium silicate and trimethylchlorosilane, applied to filter paper and polyurethane foams via a dipping technique, resulting in superhydrophobic/superoleophilic surfaces with water contact angles of ~155° and <5°, demonstrating high efficiency in gravitational oil-water separation (Zulfiqar, Usama & Hussain, Syed Zajif & Khan, Sharjeel Ahmed. 2017).

Furthermore, polyurethane-based materials are gaining special attention in oil-water separation due to their affordability, durability, and mechanical properties (Liu, Lb & Guo, Gailan & Dang, Zhao & Wenyuan, Fang. 2017). Hyperbranched polyurethane (HBPU) and fluorine-modified silicon (F-SiO_2) composite membranes, produced using electrospinning techniques, can transition from superhydrophobicity to superhydrophilicity through plasma treatment, exhibiting variable wetting properties. These membranes have proven effective in separating water-oil and oil-water emulsions stabilized with surfactants (Liu, Lb & Wenyuan, Fang & Guo, Gailan. 2017; Wenyuan, Fang & Liu, Lb & Guo, Gailan. 2017). Additionally, fluorine-free, pH-sensitive polymers and silicon-based coatings have been used for the continuous separation of three-phase oil-water-oil mixtures and emulsion filtration. These coatings stand out for their cost-effective preparation process and environmental friendliness (Dang, Zhao & Liu, Lb & Li, Yan & Xiang, Yu & Guo, Gailan. 2016).

Silicon nanoparticles are also widely applied in the medical field. For example, amorphous silicon nanoparticles have been synthesized through the hydrolysis of TEOS in ethanol, with their properties studied using surfactants such as cetyltrimethylammonium bromide (STAB) and polyvinylpyrrolidone (PVP). These particles, measuring 198 nm, have demonstrated biocompatibility and drug-carrier properties confirmed by MTT assays (Ashour, Mohamed & Soliman, Islam & Mabrouk, Mostafa & Beherei, Hanan & tohamy, Khairy. 2020). Furthermore, chitosan and its oligomer (COS) have been functionalized with silicon nanoparticles to synthesize nanosystems with antibacterial properties. COS-SiNPs systems, with sizes of 139–251 nm and zeta potentials of 30–34 mV, exhibited high antibacterial activity against Escherichia coli and Staphylococcus aureus (Salazar Navarro, Alexis & Rivera-Reyna, Nallely & gonzalez-mendoza, Daniel. 2023). Chitosan oligomers (OCS) were synthesized via microwave oxidation with hydrogen peroxide (H_2O_2), optimizing their molecular weight and deacetylation degree. The minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC) of OCS ranged from 3.75––15 mg/ml, confirming their potential use in wound healing (Doan, Vinh & Ly, Loan Khanh & Tran, Nam & Ho, Trinh & Ho, Minh & Ngoc, Nhi & Chang, Cheng-Chung & Nguyen, Hoai & Phuong Thu, Ha & Tran, Quyen & Tran, Lam & Vo, Toi & Nguyen, Thi. 2021). These studies highlight the broad application of silicon-based materials in oil-water separation and biomedical fields, emphasizing their importance in ensuring ecological sustainability. The aim of this study was to synthesize organosilicon compounds, such as ethylsiloxane, using 1,2-dichloroethane ($C_2H_4Cl_2$) and sodium silicate (Na_2SiO_3).

2. Materials and methods

The study utilized 1,2-dichloroethane (99%), anhydrous sodium silicate (99%), ethylene glycol (99%), anhydrous dimethyl sulfoxide (DMSO, 99.8%) as a solvent, and copper (I) iodide and potassium hydroxide as catalysts. FTIR spectroscopy and thermogravimetric/differential thermal analysis methods were employed.

Experimental part. The synthesis of an organosilicon oligomer, specifically oligoethylsiloxane, was carried out via a nucleophilic substitution reaction between 1,2-dichloroethane ($C_2H_4Cl_2$) and anhydrous sodium silicate (Na_2SiO_3), with ethylene glycol as an additional monomer. The experiment was conducted in a 150 ml stainless steel autoclave capable of withstanding 10 atm pressure. Reagents were used in a 1,2-dichloroethane:sodium silicate:ethylene glycol molar ratio of 1:2:1. Initially, the reagents were dissolved in DMSO solvent and mixed for 15 minutes at 600 rpm using a magnetic stirrer to ensure a homogeneous reaction environment. The resulting mixture was placed in the autoclave, securely sealed, and maintained at 100 ± 5 °C under 3–5 atm pressure for 6–8 hours. After the reaction, the autoclave was cooled to room temperature, and the mixture was filtered using a vacuum filter, with sodium chloride residues washed away. The obtained product was dried in an oven at 80 °C for 12 hours. The bonds and functional groups in the oligomer product were analyzed using infrared spectroscopy, while its thermal stability was assessed with thermogravimetric/differential thermal analysis. The synthesis yielded oligoethylsiloxane with a 75–90% yield, exhibiting hydrophobic properties (contact angle >90°).

3. Results

The synthesis of oligoethylsiloxane resulted in a yield of 75–90%, exhibiting hydrophobic properties with a water contact angle of 92–110°. Table 1 presents the reaction conditions and results, with experiments conducted at 80 °C and 100 °C temperatures, using molar ratios of $C_2H_4Cl_2$:Na_2SiO_3:HO-CH_2CH_2-OH at 1:1:1 and 1:2:1, and 0.5% CuI as a catalyst. The optimal conditions, achieved at 100 °C with a 1:2:1 ratio over 6–8 hours, provided a 90% yield. FTIR spectroscopy revealed Si-O-C and Si-O-Si (1000–1050 cm^{-1}) bonds in the 1080––1100 cm^{-1} region. TGA/DTA analysis indicated a mass loss of less than 8% up to 300 °C, confirming high thermal stability (Figure 1). Hydrophobicity tests demonstrated that the oligomer achieved 95% efficiency in separating oil-water mixtures.

Table 1. *Reaction conditions and results*

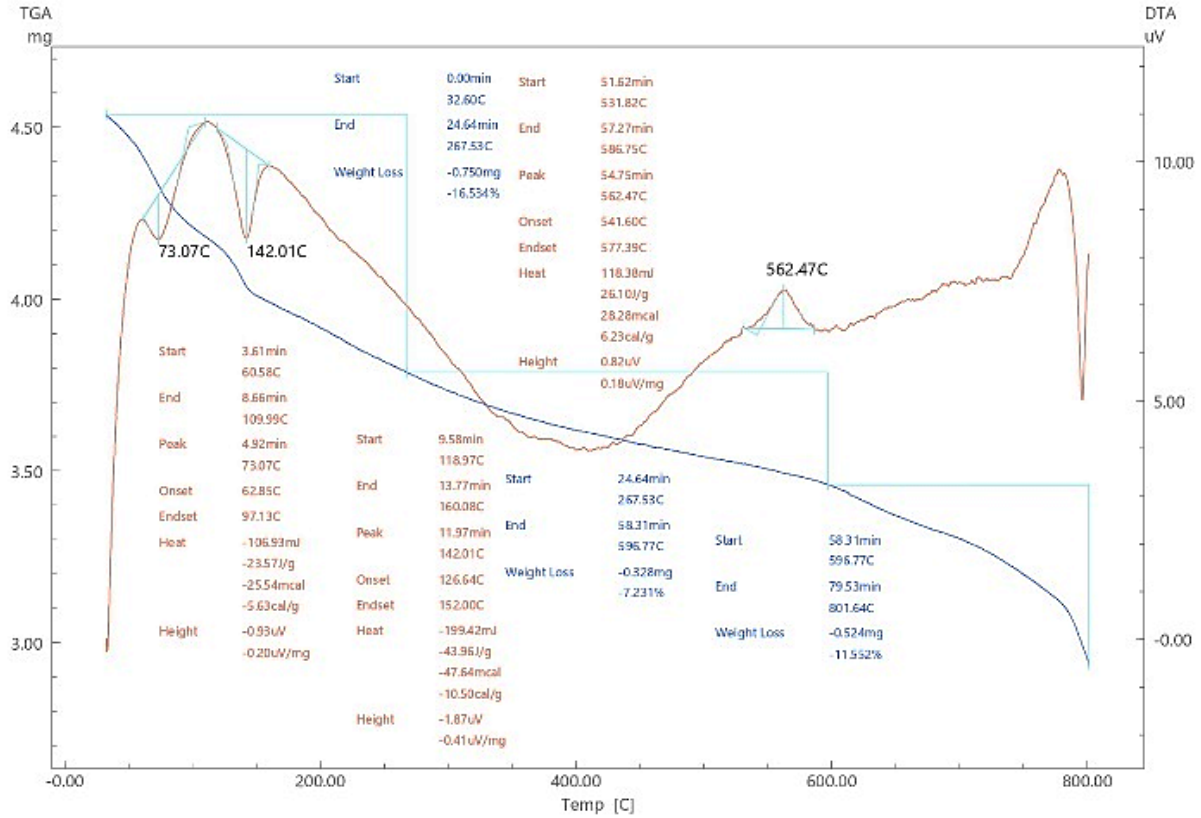
Experiments	Mol ratio*	Temperature (°C)	Catalyst (CuI, %)	Reaction time (hours)	Yield (%)
1	1:1:1	80	0.5	6	75
2	1:1:1	100	0.5	6	80
3	1:2:1	80	0.5	6	82
4	1:2:1	100	0.5	8	90
5	1:2:1	80	0.3	8	78
6	1:2:1	100	0.3	8	85

** – ($C_2H_4Cl_2$:Na_2SiO_3:HO-CH_2CH_2-OH)*

As evident from Table 1, the 1:2:1 molar ratio at 100 °C with CuI catalyst over an 8-hour reaction provided the highest yield (90%). Lower temperature (80 °C) and reduced catalyst amount decreased the yield.

Figure 1 shows the TGA/DTA curves of oligoethylsiloxane. Three main temperature ranges were identified by TGA: a 16% mass loss at 25–267 °C, attributed to the evaporation of residual DMSO, moisture, and ethylene glycol; a 7.2% loss at 267–596°C, indicating the complete degradation of Si-O-C bonds and organic chains (1,2-dichloroethane and ethylene glycol residues); and a minimal 11.5% loss above 596°C, with 50–55% stable SiO_2 residues retained. These results confirm the oligomer's high stability up to 267 °C, making it suitable for use in coatings, adhesives, and composite materials.

Figure 1. *Thermal analysis of the synthesized organosilicon oligomer*



4. Discussion

The obtained oligoethylsiloxane exhibits high thermal stability and hydrophobic properties, achieving 95% efficiency in separating oil-water mixtures. These results indicate slightly lower hydrophobicity compared to silicon nanoparticles synthesized via the sol-gel method using sodium silicate (contact angle ~155°), but the absence of fluorine in this study provides an ecological advantage. The high yield (90%) of the nucleophilic substitution reaction is attributed to the effectiveness of CuI and KOH catalysts, enhanced by the high polarity of the DMSO solvent. FTIR analysis confirmed the presence of Si-O-C and Si-O-Si bonds, indicating the oligomer's siloxane chain structure.

5. Conclusion

An effective method for synthesizing oligoethylsiloxane based on 1,2-dichloroethane and sodium silicate was developed, involving nucleophilic substitution and condensation reactions conducted in an autoclave with CuI and KOH catalysts in DMSO. The resulting compound exhibits thermal stability up to 300°C and hydrophobic properties, achieving 95% efficiency in separating oil-water mixtures. FTIR analysis confirmed the siloxane structure of the new organosilicon compound.

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