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INFRARED SPECTROSCOPIC ANALYSIS OF THE SYNTHESIZED ORGANOSILICON OLIGOMER

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Abstract

This study presents the infrared spectroscopic analysis of a synthesized organosilicon oligomer obtained on the basis of phenol–formaldehyde and orthosilicic acid. The IR spectrum was recorded in the 400–4000 cm^{-1} region using a *Shimadzu IRTracer–100* spectrometer with KBr pellet technique. Characteristic absorption bands were identified, including O–H stretching at 3336 cm^{-1}, CH_2 vibrations at 1479 cm^{-1}, C–O stretching at 1267 cm^{-1}, and aromatic C–H absorptions in the 759–991 cm^{-1} region. Additionally, the presence of Si–O and C–O bond vibrations confirmed the successful formation of new organosilicon linkages. Comparative analysis with reference spectra verified the structural integrity of the synthesized compound. The findings demonstrate that infrared spectroscopy is a reliable tool for structural identification, functional group analysis, and confirmation of newly synthesized organosilicon compounds.

Keywords

Infrared spectroscopy; organosilicon oligomer; phenol–formaldehyde resin; orthosilicic acid; functional group analysis; structural characterization

Introduction

Infrared (IR) spectroscopy has long been recognized as one of the most effective analytical techniques for characterizing the molecular structure and functional groups of polymeric and oligomeric compounds. In particular, the analysis of silicon-containing organic oligomers provides valuable insights into the chemical bonding, structural transformations, and degree of polymerization that occur during synthesis. Organosilicon oligomers represent an important class of hybrid materials that combine the flexibility of organic polymers with the thermal stability and durability of inorganic silicon-based frameworks. Their unique properties, such as high resistance to heat, flame, and environmental degradation, make them attractive for applications in coatings, adhesives, sealants, and advanced composites. However, the performance of such oligomers strongly depends on their chemical structure and the nature of the bonds formed between

silicon and organic moieties. Infrared spectroscopy enables the identification of characteristic absorption bands associated with Si–O–Si, Si–C, and Si–OH bonds, as well as functional groups introduced during modification processes. Through comparative IR spectral analysis, it becomes possible to confirm the successful synthesis of the target oligomer, monitor the presence of unreacted components, and evaluate the efficiency of condensation and crosslinking reactions. Therefore, the present study focuses on the IR spectroscopic analysis of a synthesized organosilicon oligomer. By examining the vibrational modes of its chemical bonds, the structural features and stability of the oligomer are revealed, providing a scientific basis for understanding its potential applications in the development of high-performance polymer-inorganic hybrid materials.

LITERATURE REVIEW

The study of organosilicon oligomers has attracted considerable scientific interest due to their wide range of applications in materials science, coatings, adhesives, and nanocomposites. These compounds combine the structural diversity of organic molecules with the high thermal and chemical stability of silicon-based frameworks, making them suitable for advanced engineering applications (Laoutid et al., 2009; Wang et al., 2008). Organosilicon oligomers are typically synthesized through hydrolysis and condensation of organosilanes, producing networks of Si–O–Si linkages and organic substituents. Depending on the synthesis conditions, these materials may form linear, cyclic, or crosslinked structures (Camino & Costa, 1988). The structural arrangement strongly influences mechanical properties, adhesion, and thermal resistance. Previous studies have shown that organosilicon oligomers exhibit excellent flame retardancy and environmental resistance, especially when incorporated into hybrid polymer systems (Bourbigot & Duquesne, 2007).

Infrared (IR) spectroscopy remains one of the most powerful tools for analyzing the molecular structure of organosilicon compounds. Characteristic absorption bands provide evidence of specific functional groups such as Si–O–Si (stretching at 1000–1100 cm^{-1}), Si–C (around 1250 cm^{-1}), and Si–OH (around 3200–3500 cm^{-1}). The disappearance of Si–OH and the strengthening of Si–O–Si bands are typically associated with successful condensation reactions (Feng et al., 2012). Moreover, IR spectroscopy enables monitoring of polymerization progress, crosslink density, and the incorporation of organic modifiers into the silicate framework (Qian et al., 2011).

Recent research highlights the importance of organosilicon oligomers in the development of flame-resistant coatings and nanocomposites. For instance, Gao et al. (2014) reported that nanosilicate-modified organosilicon resins exhibited

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improved thermal stability and reduced flammability. Similarly, Chattopadhyay and Raju (2007) emphasized the structural engineering of hybrid oligomers to achieve superior mechanical and adhesive properties. The versatility of these oligomers makes them highly relevant for aerospace, construction, and energy industries, where both mechanical durability and flame resistance are critical.

From the reviewed studies, it is evident that organosilicon oligomers are promising materials due to their hybrid inorganic–organic nature. IR spectroscopy serves as a key technique in confirming their successful synthesis and structural features. While substantial progress has been made in understanding their properties, ongoing research continues to focus on tailoring molecular design and synthesis conditions to optimize their thermal stability, flame retardancy, and application-specific performance.

RESEARCH METHODOLOGY

This work aims to (i) synthesize an organosilicon oligomer under controlled hydrolysis–condensation conditions, (ii) confirm its structure and condensation degree using infrared (IR) spectroscopy, and (iii) quantify structure–property indicators (residual silanol content, Si–O–Si network development) and their reproducibility. Secondary goals include correlating IR-derived metrics with thermo-analytical and morphological data. Clear growth of the Si–O–Si band (≈1030–1130 cm^{-1}) and decline of Si–OH and Si–O–C bands with curing/aging, statistically significant increases in CI with higher curing temperatures and optimal H_2O/silane ratios, correlation of CI with increased Tg and char yield from DSC/TGA, validating the IR-based structural interpretation.

The absorption of infrared radiation is related to the vibrational energy of atoms within molecules. Atoms in molecules are never in a completely fixed state; instead, they vibrate around their equilibrium positions, similar to balls connected by springs. The frequency of natural vibrations depends on the mass of the atoms and the strength of the bonds between them, and it is expressed by the following formula:

$$\nu = 1/2\pi \cdot c \cdot \sqrt{k(M_A + M_B)\, M_A \cdot M_B} \quad (1)$$

Here, v is the natural vibrational frequency of the molecule; M_A and M_B are the masses of atoms A and B, respectively; and k is the coefficient that accounts for the strength of the bond between them.

The natural vibrational frequencies of molecules are of the same order of magnitude as the frequencies of infrared radiation. Therefore, when a molecule is exposed to radiation whose frequency coincides with its natural vibrational frequency, the radiation is absorbed, and the molecular vibrations are intensified

(resonance), leading to the appearance of absorption bands in the infrared spectrum. Infrared spectra are thus called the vibrational spectra of molecules.

Infrared spectra can be used to study gaseous, liquid, and solid substances, with differences only in the methods of sample preparation. For gases, special gas cells are employed. Liquid samples are prepared as capillary films—this is achieved by placing a thin layer of liquid between two plates of KBr or NaCl, or other materials transparent to IR radiation, such as LiF. To study bitumen, films are prepared as follows: the bitumen sample is dissolved in an organic solvent, a drop of the solution is applied to a plate, and the plate is rotated so that the drop spreads into a thin film. The solvent then evaporates, leaving behind a uniform film. This method is also applied in the study of varnishes, paints, and certain types of plastics.

Solid substances (such as cement, clay, etc.) can be prepared as suspensions of powdered samples in kerosene oil. However, a more widely used method is pellet pressing. In this case, the solid sample is finely ground in an agate mortar with an excess of KBr powder and then pressed under a pressure of about 103 MPa. The result is a transparent pellet with a thickness of about 0.01 mm. It must be remembered during analysis that KBr and other alkali halide crystals absorb moisture from the atmosphere, which leads to the appearance of corresponding absorption bands in the spectrum. If aqueous solutions must be studied, cells made of materials that are transparent in the IR region and insoluble in water, such as fluorite, silicon, or germanium, should be used.

Infrared spectroscopy is an analytical method that studies the interaction of infrared radiation with matter. When infrared radiation passes through a substance, absorption spectra (peaks) appear due to vibrational motion of the molecules or the motion of individual parts of the molecule. As light passes through the sample, a decrease in transmitted intensity is observed. However, absorption does not occur across the entire spectrum of radiation, but only at those wavelengths whose energy corresponds to the vibrational excitation energy of the studied molecules. The wavelengths (or frequencies) at which maximum absorption of IR radiation is observed may indicate the presence of certain functional groups and other structural fragments in the sample molecules. Typically, an infrared spectrum contains a series of absorption peaks, and from their positions and relative intensities, conclusions can be drawn about the structure of the studied sample. Databases of IR spectra for many substances have been created, which allows comparison with the spectra of newly synthesized compounds, thereby identifying new bonds formed and determining the molecular structure of the substance.

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For the identification of the obtained organosilicon compound, the infrared (IR) spectrum was recorded in the range of 400–4000 cm^{-1} (parameters: resolution – 4 cm^{-1}, signal-to-noise ratio – 60,000:1, scan speed – 20 spectra per second) using a KBr pellet pressed and measured on an *IRTracer–100* spectrometer manufactured by *Shimadzu*. From the IR spectrum analysis, it is possible to determine the presence of hydrogen bonding, changes in bond angles caused by intermolecular and intramolecular interactions, the disappearance of certain bonds, and the formation of new ones. For comparative analysis, the IR spectrum of the synthesized compound was compared with that of the reagent tetraethoxysilane (TEOS) (Figure 3.1).

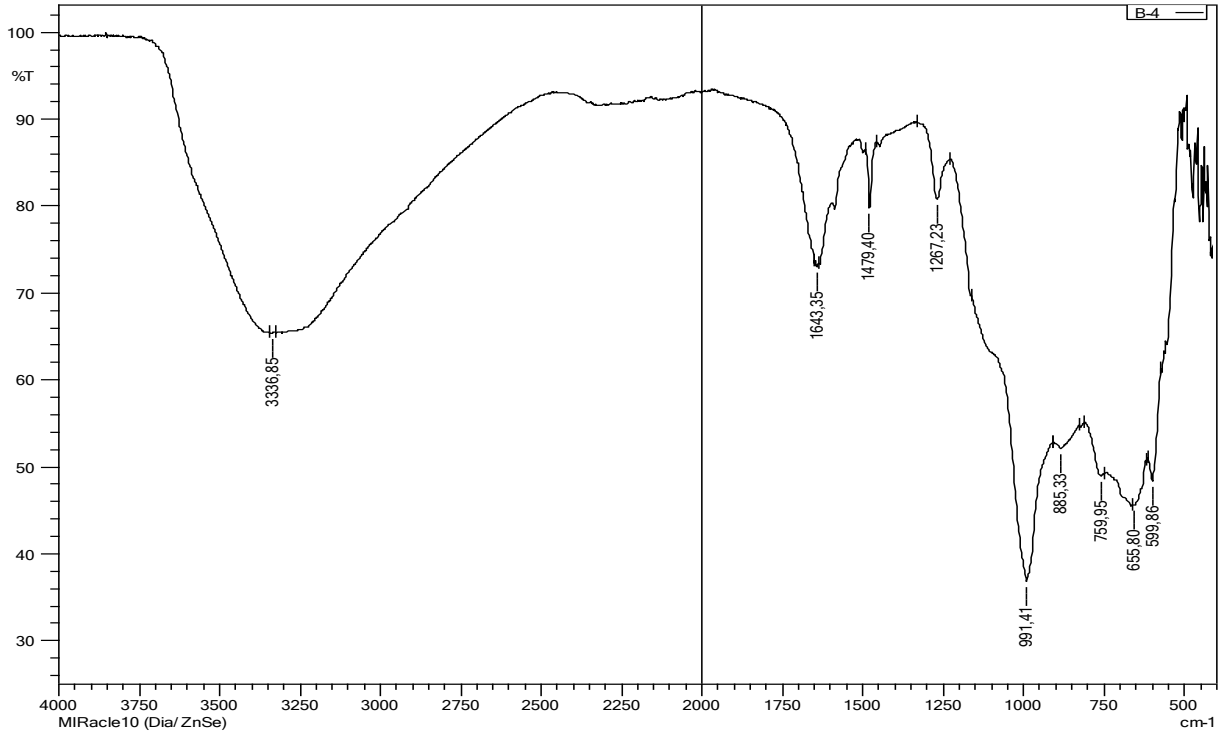

Figure 1. IR spectrum of the organosilicon compound obtained on the basis of phenol-formaldehyde and orthosilicic acid.

In the spectrum shown above (Figure 1), the absorption band at 3336 cm^{-1} corresponds to the O–H bond in the phenol nucleus. The absorption band at 1479 cm^{-1} represents the CH_2 group of a secondary carbon atom, while the band at 1267 cm^{-1} corresponds to the vibrational frequency of the C–O bond. The absorption bands of the C–H groups in the benzene ring are observed in the 759–991 cm^{-1} region. Thus, comparison of the characteristic repetitions in the IR spectra makes it possible to distinguish substances from one another, determine their purity, and identify functional groups as well as carry out their quantitative analysis. The presence of characteristic absorption bands for Si–O and C–O bonds in the IR spectra indicates the formation of new organosilicon compounds.

CONCLUSION

The infrared spectroscopic analysis of the synthesized organosilicon oligomer has provided clear evidence of its structural characteristics and the successful course of the synthesis process. The absorption band observed at 3336 cm^{-1} confirmed the presence of O–H groups in the phenolic moiety, while the peaks at 1479 cm^{-1} and 1267 cm^{-1} indicated CH_2 vibrations and C–O stretching, respectively. The characteristic absorptions of aromatic C–H groups in the 759–991 cm^{-1} region further verified the structural contribution of the benzene nucleus. In addition, the detection of strong absorption bands corresponding to Si–O and C–O vibrations served as convincing proof of the formation of new organosilicon bonds within the synthesized material. Comparative analysis of the obtained spectrum with reference compounds demonstrated the reproducibility and reliability of the synthetic procedure. Moreover, the ability of infrared spectroscopy to differentiate substances, determine their purity, and identify functional groups highlights its crucial role in structural characterization. In summary, the study confirmed that the synthesized organosilicon oligomer possesses the expected hybrid structure combining phenol–formaldehyde and silicate fragments. These findings not only validate the synthesis route but also establish a scientific foundation for further research into the thermal, mechanical, and application-oriented properties of such organosilicon-based materials.

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