Synthesis And Characterization Of Nanocellulose/Zif-8 Composite Aerogel

Tajimova Gulistan Raman qizi
PhD student, Institute of General and Inorganic Chemistry, Academy of Sciences of the Republic of Uzbekistan, Uzbekistan

Kuzieva Makhliyo Mukhammadievna
PhD, Junior researcher, Institute of Polymer Chemistry and Physics, Academy of Sciences of the Republic of Uzbekistan, Uzbekistan

Abdumutolib Abdupatto o‘g‘li Atakhanov
Doctor of technical sciences, Professor, Director of Institute of Polymer Chemistry and Physics, Academy of Sciences of the Republic of Uzbekistan, Uzbekistan

Adizov Bobirjon Zamirovich
Doctor of technical sciences, Professor, Institute of General and Inorganic Chemistry, Academy of Sciences of the Republic of Uzbekistan, Uzbekistan

Khomidov Fakhriddin Gafurovich
Doctor of Chemical Sciences, Institute of General and Inorganic Chemistry of Academy of Sciences of the Republic of Uzbekistan, Termez State University of Engineering and Agrotechnologies, Uzbekistan

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Abstract

In this study, a nanocellulose and ZIF-8 composite aerogel was successfully synthesized via an in-situ growth method. The structural and elemental properties of the obtained hybrid aerogel were investigated using FTIR and EDX/XRF analyses. FTIR results confirmed the presence of characteristic functional groups belonging to both nanocellulose and ZIF-8, indicating successful integration of the metal–organic framework into the cellulose matrix. EDX/XRF analysis revealed distinct zinc-related peaks corresponding to Zn Kα and Zn Kβ emissions, confirming the incorporation of ZIF-8 particles within the porous aerogel structure. The obtained results demonstrated the successful formation of a stable hybrid porous material.

Keywords: Nanocellulose, ZIF-8, composite aerogel, in-situ synthesis, hybrid porous material.

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1. Introduction

Aerogels are a unique class of highly porous materials characterized by extremely low density, large specific surface area, and interconnected three-dimensional porous structures. Due to their remarkable physicochemical properties, including low thermal conductivity, high porosity, and tunable surface functionality, aerogels have attracted considerable attention in recent decades for applications in adsorption, catalysis, thermal insulation, gas storage, energy conversion, and environmental remediation [1–3]. Conventional silica-based aerogels exhibit excellent thermal and structural properties; however, their brittleness, high production cost, and limited mechanical flexibility have encouraged researchers to develop alternative organic and hybrid aerogel systems [4,5].

Among emerging biopolymer-based aerogels, nanocellulose is considered one of the most promising materials due to its renewability, biodegradability, low density, high mechanical strength, and porous structure [6,7]. Its abundant surface hydroxyl groups enable easy chemical modification, while the interconnected pore network provides excellent adsorption properties for oils, dyes, and heavy metal ions [8–11].

Metal–organic frameworks (MOFs) are highly porous crystalline materials with large surface area, tunable pore structures, and versatile functional properties, making them attractive for adsorption, catalysis, gas storage, and environmental applications [12]. However, pure MOF aerogels often suffer from poor mechanical stability [13]. To overcome this limitation, MOFs are commonly combined with biopolymeric matrices such as nanocellulose, which offers renewability, biodegradability, high mechanical strength, and abundant hydroxyl groups [14]. The porous nanocellulose network provides an effective scaffold for MOF growth, while surface hydroxyl groups enhance MOF dispersion and structural stability in the composite aerogels [15].

Several studies have shown that nanocellulose/MOF hybrid aerogels exhibit enhanced adsorption performance due to the synergistic combination of hierarchical porosity and functional active sites [16]. In these composites, nanocellulose provides flexibility and mechanical stability, while MOFs contribute high surface area and tunable adsorption properties. Consequently, nanocellulose/ZIF-8 aerogels have demonstrated strong potential for water purification, gas adsorption, dye removal, and other environmental applications because of their lightweight structure, porous morphology, and improved functional performance [17–19].

Materials and Methods

Nanocellulose was used as the primary biopolymeric matrix for the preparation of composite aerogels. Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) and 2-methylimidazole (2-MIM) were employed as precursors for the synthesis of ZIF-8. Distilled water was used as the solvent throughout all experiments. All chemicals were of analytical grade and used without further purification.

The nanocellulose and ZIF-8 composite aerogel was synthesized by first dispersing nanocellulose in distilled water until a homogeneous suspension was obtained. Subsequently, a zinc nitrate solution was gradually added, and the mixture was continuously stirred for a certain period. In the next step, a 2-methylimidazole solution was introduced to promote the in-situ formation of ZIF-8 crystals on the surface of the nanocellulose fibers. The resulting mixture was then kept at room temperature to allow the formation of a hydrogel structure. The obtained hydrogel was repeatedly washed to remove residual reagents. Finally, the sample was freeze-dried to produce a lightweight nanocellulose and ZIF-8 composite aerogel with a highly porous structure.

Fourier-transform infrared (FTIR) spectra were recorded to identify the functional groups and chemical interactions in the synthesized aerogels. FTIR spectra of the samples were recorded in the range of 4000–400 cm^{-1} using a PerkinElmer Spectrum Two spectrometer.

The elemental composition of the composite aerogel was analyzed using X-ray fluorescence (XRF) spectroscopy.

Results and Discussion

FTIR Analysis

The FTIR spectrum of the synthesized nanocellulose and ZIF-8 aerogel confirms the successful incorporation of ZIF-8 into the nanocellulose matrix and demonstrates the existence of intermolecular interactions between the organic and inorganic phases. The broad absorption band observed in the range of 3200–3500 cm^{-1} is attributed to the stretching vibrations of hydroxyl (–OH) groups originating from nanocellulose. The broad nature of this band indicates the presence of extensive intermolecular hydrogen bonding within the three-dimensional aerogel network, which is characteristic of cellulose-based porous materials.

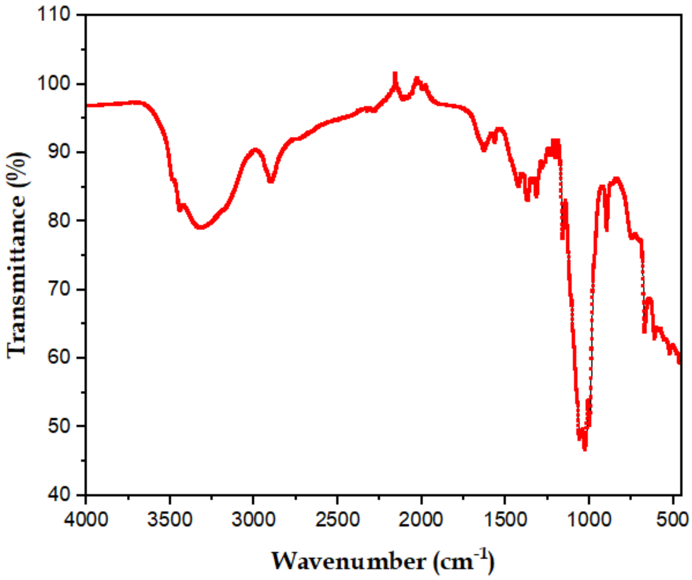

Fig.1. FTIR spectrum of aerogel samples.

The absorption peaks around 2900 cm^{-1} correspond to the symmetric and asymmetric stretching vibrations of aliphatic C–H groups in the cellulose backbone. These characteristic bands confirm that the fundamental polysaccharide structure of nanocellulose remained preserved after the incorporation of ZIF-8 particles.

A distinct absorption region observed near 1580–1650 cm^{-1} can be associated with the stretching vibration of C=N bonds and ring vibrations of the imidazolate linker present in ZIF-8. In addition, weak contributions from adsorbed water molecules may also overlap in this region due to the hydrophilic nature of nanocellulose. The presence of these characteristic imidazole-related peaks confirms the successful formation and structural stability of the ZIF-8 framework inside the aerogel matrix.

The peaks detected in the range of 1350–1500 cm^{-1} are assigned to the stretching vibrations of C–N bonds and methyl groups from 2-methylimidazole ligands of ZIF-8. These findings are in good agreement with previously reported FTIR characteristics of ZIF-8-based composite materials.

Furthermore, the strong absorption bands located at approximately 1000–1150 cm^{-1} are attributed to C–O–C and C–O stretching vibrations of the β-(1→4)-glycosidic bonds in nanocellulose. The persistence of these peaks demonstrates that the cellulose crystalline domains were retained during the synthesis process.

In the low-wavenumber region below 800 cm^{-1}, several characteristic bands corresponding to Zn–N stretching vibrations can be observed, which are considered fingerprint features of the ZIF-8 framework. The appearance of these peaks confirms the coordination interaction between Zn^{2+} ions and imidazolate ligands, indicating the successful growth of ZIF-8 crystals within the nanocellulose network.

Additionally, slight shifts and intensity variations in several absorption bands compared with pristine nanocellulose and pure ZIF-8 suggest strong interfacial interactions between hydroxyl groups of nanocellulose and the ZIF-8 surface. Such interactions may improve the structural integrity, porosity, and adsorption performance of the resulting hybrid aerogel.

Overall, the FTIR analysis demonstrates that the synthesized nanocellulose/ZIF-8 aerogel possesses the characteristic functional groups of both nanocellulose and ZIF-8, confirming the successful fabrication of a hybrid porous structure. The coexistence of hydroxyl-rich cellulose domains and metal–organic framework functionalities indicates the potential applicability of the aerogel in adsorption, catalysis, gas capture, and environmental remediation processes.

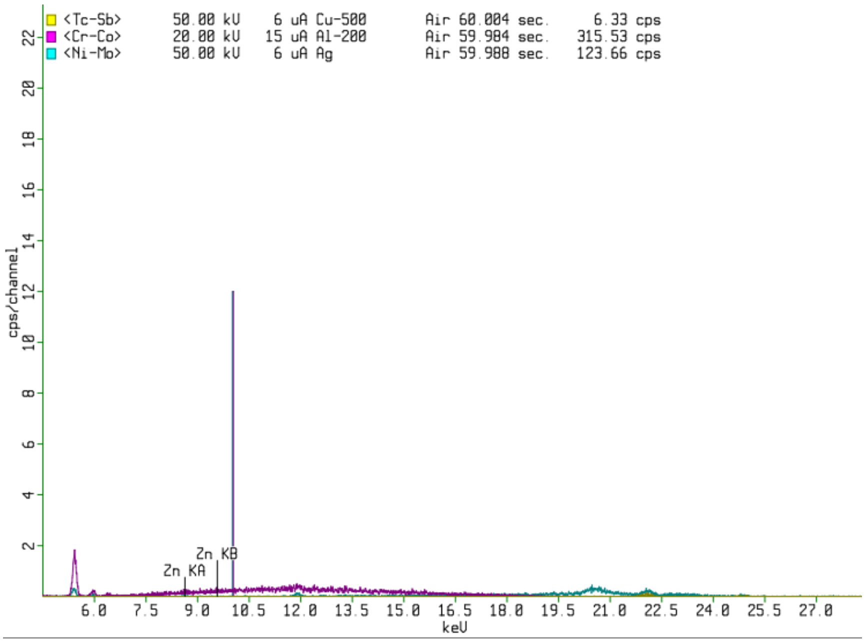

Figure 2. XRF spectra of ONC@ZIF-8 aerogel.

The elemental composition of the synthesized nanocellulose/ZIF-8 aerogel was investigated using energy-dispersive X-ray spectroscopy (EDX), and the obtained spectrum is presented in Fig. 2. The analysis confirmed the successful incorporation of ZIF-8 particles into the nanocellulose-based porous framework through the identification of characteristic zinc-related emission peaks.

As observed in the EDX spectrum, the dominant peaks located at approximately 8.6 keV and 9.6 keV correspond to Zn Kα and Zn Kβ emissions, respectively. These characteristic signals clearly verify the presence of zinc species originating from the ZIF-8 structure. The relatively high intensity of the Zn peaks indicates that Zn-containing metal–organic framework particles were successfully immobilized within the aerogel matrix and remained structurally stable after the synthesis and drying processes.

Conclusions

The FTIR analysis confirmed the successful incorporation of ZIF-8 into the nanocellulose matrix and demonstrated the presence of intermolecular interactions between the organic and inorganic phases of the composite aerogel. The characteristic absorption bands corresponding to hydroxyl, C–H, C=N, C–N, C–O–C, and Zn–N groups verified the coexistence of nanocellulose and ZIF-8 structural components within the hybrid porous network. In addition, the EDX/XRF spectrum revealed distinct Zn Kα and Zn Kβ emission peaks, confirming the successful incorporation and structural stability of zinc-containing ZIF-8 particles within the aerogel framework. Overall, the obtained results demonstrate the successful formation of a nanocellulose/ZIF-8 composite aerogel with a stable hybrid porous structure.

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