

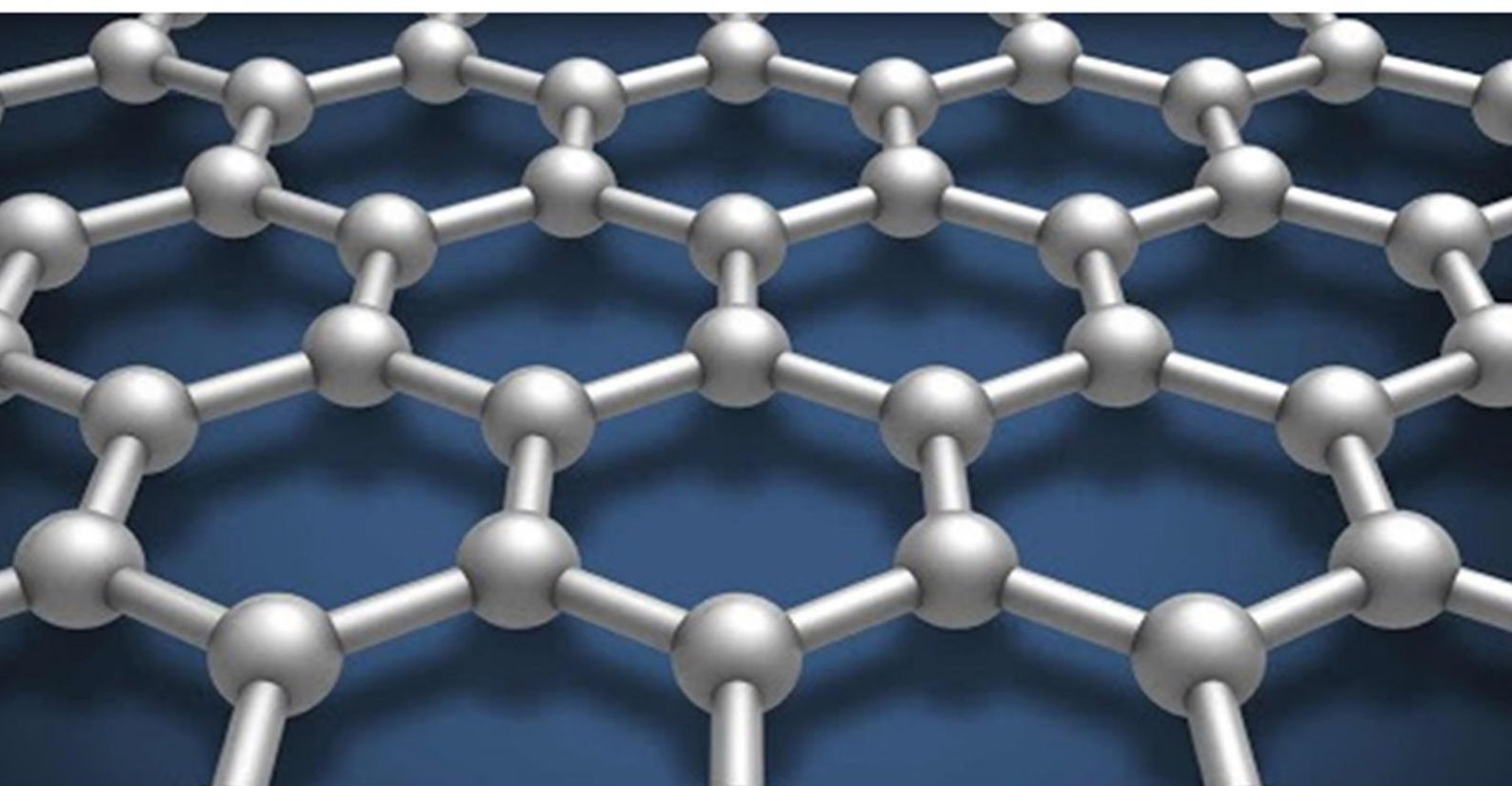
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**SYNTHESIS OF IRON-MODIFIED CHITOSAN FOR THE PURIFICATION OF HYDROGEN SULFIDE WASTE FROM THE OIL AND GAS INDUSTRY****Bakhromova Iroda Alisher qizi, Vokhidova Noira Rahimovna***Institute of Polymer Chemistry and Physics of the Academy of Sciences of the Republic of Uzbekistan*

**Abstract.** In this study, a chitosan-based composite containing up to 51.73 wt.% iron was synthesized using chemical precipitation and cross-linking methods for the effective absorption of hydrogen sulfide ( $H_2S$ )-a component of natural gas emissions released into the atmosphere. The composite was characterized using FTIR, XRD, and EDS techniques. Its  $H_2S$  absorption properties were evaluated under industrial conditions at the “Gazli oil and gas production department” in the Bukhara region, and its  $H_2S$  absorption capacity was determined to be 1.82 mg/g.

**Keywords:** chitosan,  $CS/Fe^{3+}$ , hydrogen sulfide, sorbent.

**Introduction.** In recent years, metal-ion-modified chitosan (CS) composites ( $CS/Me^+$ ) have been recognized as promising sorbents for ecosystem protection. Chitosan's biodegradability and the presence of electron-donating functional groups-specifically  $-NH_2$  and  $-OH$  impart effective sorption properties to the material. Modifying CS with  $Fe_xO_y$  enhances the sorbent's specific surface area and its mechanical and chemical stability; furthermore, it creates additional active sites, thereby improving its sorption characteristics. Literature analysis indicates [1] that iron-modified chitosan effectively adsorbs heavy metal ions, anions (such as arsenate, phosphate, and chromate), and toxic gases like hydrogen sulfide ( $H_2S$ ). High sorption capacity, selectivity, and the potential for regeneration further underscore the scientific and practical significance of this material [2-3].

These modified sorbents demonstrate high efficiency in  $H_2S$  capture. This efficiency is determined by surface chemical properties that can enhance the adsorption-catalytic oxidation mechanism for  $H_2S$ . These processes lead to the conversion of  $H_2S$  into various sulfur-containing products, even under conditions of very low oxygen content and high humidity. Potential products include elemental sulfur (S),  $SO_2$ , sulfates, sulfuric acid, and metal sulfides [4]. In this context, the synthesis of  $Fe^{3+}$ -modified CS-based sorbents and the study of their structural properties are of significant interest for the development of eco-friendly sorbents.

The aim of this study is to determine the conditions for preparing an iron-modified chitosan composite and to characterize it, as well as to investigate the potential of  $CS/Fe^{3+}$  for removing  $H_2S$  from natural gas and biogas.

**Materials and methods.** The following materials were used to prepare the composite:  $FeCl_3 \cdot 6H_2O$  (99%, Macklin, China), chitosan (CS; degree of deacetylation  $\geq 95\%$ , Mw 50 kDa, Macklin, China), 25% glutaraldehyde (Hungary),

NaOH (analytical grade), acetic acid, and distilled water.

To investigate the structure of the composite, Fourier-transform infrared (FTIR) spectroscopy was employed in the  $400-4000\text{ cm}^{-1}$  range using a Bruker INVENIO-S spectrophotometer (Bruker Optik GmbH, Ettlingen, Germany). Samples were prepared as potassium bromide (150-250 mg KBr) pellets; the solid sample (1.5-2 mg) was ground into a fine powder in a porcelain mortar. The resulting mixture was placed in a mold, held under vacuum, and pressed at a pressure of  $7 \times 10^8\text{ Pa}$  for 2 minutes.

XRD analysis was performed using an XRD-6100 X-ray diffractometer (Shimadzu, Japan). Diffraction patterns were recorded and monitored via computer. The XRD-6100 instrument utilized Ni-filtered  $CuK\alpha$  radiation, with the X-ray tube operating at a current of 30 mA and a voltage of 30 kV. The detector scanning speed was set to  $4^\circ/\text{min}$ , the scanning angle range was  $4^\circ$  to  $80^\circ$ , and the step size was  $0.02^\circ$  ( $2\theta$ ).

Energy-dispersive spectroscopy (EDS) was used to determine the elemental composition of the obtained samples.

**Results and discussion.** Chitosan and  $FeCl_3$  were subjected to a co-precipitation process at  $22^\circ\text{C}$ , involving varying mass ratios and intensive stirring at 400 rpm for 10 minutes. The mixture was then titrated with a 0.5 M NaOH solution. The resulting chitosan composite ( $CS/Fe^{3+}$ ) was decanted and washed repeatedly with distilled water. In the second stage of the process, 12.5% glutaraldehyde was used to cross-link the obtained  $CS/Fe^{3+}$  powder. To ensure complete cross-linking, the  $CS/Fe^{3+}$  was stirred continuously with glutaraldehyde at  $22^\circ\text{C}$  for 24 hours. The  $CS/Fe^{3+}$  composite was washed multiple times with distilled water and subsequently dried in an oven at  $60^\circ\text{C}$  for 24 hours until a constant mass was achieved. The resulting composite was found to contain 51.73% Fe. FTIR spectroscopic analysis was performed on the sample to identify the functional groups of the composite (Figure 1).

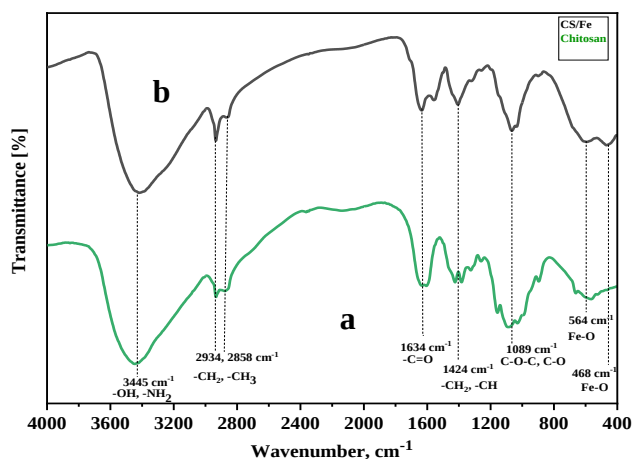


**Figure 1. FTIR spectra of chitosan (a) and CS/Fe<sup>3+</sup> (b)**

Accordingly, a broad absorption band corresponding to the vibrations of hydrogen bonds between the -OH and -NH<sub>2</sub> groups in chitosan was observed in the 3450-3300 cm<sup>-1</sup> region of the FTIR spectrum. The wavenumbers 2920 cm<sup>-1</sup> and 2858 cm<sup>-1</sup> are characteristic of -CH<sub>2</sub> and -CH<sub>3</sub> group vibrations; the fact that the intensity of these vibrations remained virtually unchanged after iron modification indicates that the glucosidic ring structure of the chitosan was preserved. Absorption associated with Amide I (C=O) and N-H groups was observed in the 1650–1590 cm<sup>-1</sup> range (specifically at 1634 cm<sup>-1</sup>). The absorption at 1424 cm<sup>-1</sup> is characteristic of the -CH<sub>2</sub> and -CH groups in chitosan. The absorption band at 1089 cm<sup>-1</sup> corresponds to the C-O-C and C-O bonds in chitosan and exhibits a slight shift in the CS/Fe<sup>3+</sup> sample. Finally, the absorption at 468 cm<sup>-1</sup> likely corresponds to the Fe-O bond, indicating that the composite has been modified with iron. These results are consistent with data reported in the [5].

Based on FTIR results, the presence of the absorption region associated with Fe-O bonds was also confirmed by the composite's EDS spectra and XRD data, indicating that the composite possesses a predominantly amorphous structure (Figure 2).

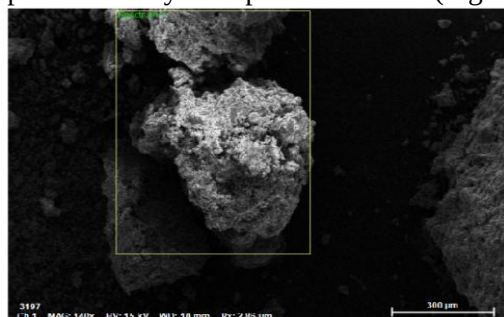


**Figure 3. EDS spectrum of CS/Fe<sup>3+</sup>**

The EDS spectrum revealed the presence of C 31.55 wt.%, O 16.14 wt.%, Fe 51.73 wt.% elements. The C and O signals may be related to the oxygen-binding functional groups and oxygen compounds of iron in chitosan. The Fe signal indicates the modification of iron to chitosan.

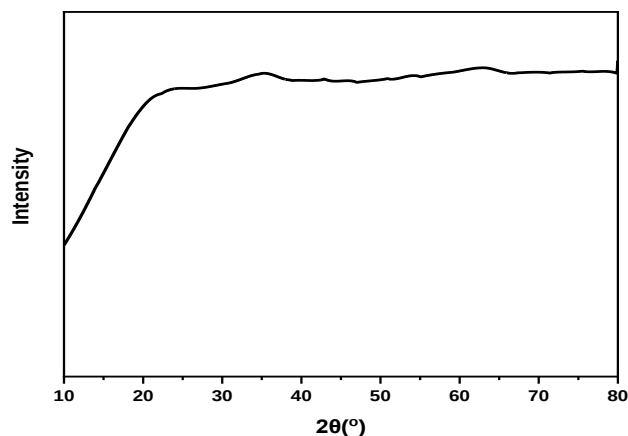


**Figure 2. X-ray phase analysis of the CS/Fe<sup>3+</sup> composite**

X-ray analysis was performed to determine the structural properties of the obtained CS/Fe<sup>3+</sup> composite; this enabled an assessment of whether the composite was crystalline or amorphous and allowed for the identification of structural changes resulting from the modification of chitosan with Fe<sup>3+</sup> ions. No distinct diffraction peaks were observed in the CS/Fe<sup>3+</sup> diffractogram (Figure 2); instead, a broad, diffuse maximum appeared. This indicates that the material has a predominantly amorphous structure. The disappearance of diffraction peaks characteristic of chitosan suggests that the degree of polymer crystallinity decreased due to the coordination bonding of Fe<sup>3+</sup> ions with the -NH<sub>2</sub> and -OH functional groups of the chitosan chains. Furthermore, characteristic peaks for iron oxide crystals were not observed, as crystalline iron oxides had not yet formed at this stage. The observation of a very weak and broad shoulder in the 33°-36° range on the diffractogram may indirectly indicate the successful incorporation of iron ions into the composite structure.

Based on the micrograph obtained using a scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) was carried out, which revealed the content of Fe<sup>3+</sup> (Figure 3).

The H<sub>2</sub>S sorption properties of the composite were investigated under industrial conditions at the “Gazli oil and gas production department” in the Bukhara region, and its sorption capacity was determined to be 1.82 mg/g.

**Conclusion.** A CS/Fe<sup>3+</sup> composite containing 51.73 wt.% Fe was synthesized, and its structure, composition, and morphology were characterized. XRD results revealed that the sample possesses an amorphous structure. The presence of absorption bands associated with Fe-O in the FTIR spectrum indicates the conversion of the FeCl<sub>3</sub> salt into an

oxygen-containing iron(III) compound. Preliminary laboratory studies on the application of this composite for the adsorption of industrial gases such as H<sub>2</sub>S and CO<sub>2</sub> demonstrate the future potential of the CS/Fe<sup>3+</sup> composite. Research is ongoing.

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## РАЗРАБОТКА И ИССЛЕДОВАНИЕ СВОЙСТВ КОМПОЗИЦИОННЫХ КОРДИЕРИТОВЫХ ЭЛЕКТРОКЕРАМИЧЕСКИХ МАТЕРИАЛОВ НА ОСНОВЕ МЕСТНОГО И ВТОРИЧНОГО СЫРЬЯ

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**Аннотация.** В статье представлены результаты разработки композиционных электрокерамических материалов на основе местного и вторичного сырья, а также исследования их керамико-технологических, физико-механических и диэлектрических свойств. Установлено, что опытные образцы композиции КМ-1, КМ-2 обладают высокими показателями электрической прочности, удельного объемного сопротивления и механической прочности.

**Ключевые слова:** тальк, каолин, бентонит, кордиеритовая керамика, электрокерамика, диэлектрические свойства, температура, обжиг, композиционные массы, опытные образцы.

**Введение.** За последние годы разнообразие керамических материалы получили широкое применение во многих отраслях промышленности. Существуют различные керамические материалы, обладающие специфическими свойствами, благодаря, которые нашли широкое применение таких отраслях промышленности, как ракетостроение, ядерной техники, самолетостроение, в металлургической, нефтехимической, электротехнической, химической и другие. Керамические материалы обладают высокой механической, термической, электрической прочностью, огнеупорностью, диэлектрическими свойствами, пьезоэлектрическими, сегнетоэлектрическими и другими свойствами.

В связи с этим предстоит большие задачи по дальнейшему развитию новых керамических материалов со специфическими свойствами,

изыскания новых сырьевых ресурсов для керамической промышленности, усовершенствованию технологии производства, разработку безотходной технологии получения высококачественных керамических материалов.

Ускоренные темпы развития электротехники, энергетики и машиностроения в республике связаны с применением в больших масштабах самых разнообразных электроизоляционных материалов. В связи с этим создается острая необходимость проведения глубоких научно-технических изысканий по определению возможности организации производства электротехнических изделий с использованием местного сырья и отходов промышленности. Необходимо создавать новые электрокерамические материалы, характеризующиеся специфическими свойствами, разработать новые технологические процессы,

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