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# Aminofunctional Silica-Polysaccharide Materials as Adsorbents of Cu (II) and Zn(II) from Contaminated Water

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**ABSTRACT:** The aminofunctional silica-polysaccharide adsorbent was synthesized with specified structural and functional characteristics by sol-gel process and their physic-chemical properties have been studied. Scanning Electron Microscopy (SEM) analysis demonstrated that the synthesized chitosan/silica hybrid material exhibited spherical particles with diameters varying between 3 and 15 µm. The IR spectroscopy analysis indicated shifts in the absorption bands of the nanohybrid adsorbent samples from 1650 $cm^{-1}$ and 1584 $cm^{-1}$, related to chitosan's carbonyl amide groups, to 1618 $cm^{-1}$ and 1424 $cm^{-1}$. These changes suggest hydrogen bond formation between silanol and carbonyl groups within the amide structure of chitosan. The obtained adsorbent exhibited mesoporous structure, confirmed by BET surface area of 292 $m^2/g$, 42 nm average pore size (via porosimetry), and Langmuir V-type adsorption-desorption isotherm. The adsorption properties of silica-polysaccharide nanohybrid adsorbent, i.e., the effect of pH and initial metal concentration on the adsorption of copper (*II*) and zinc(II) ions were studied and demonstrates notable adsorption efficiency for both metal ions within a pH range of 2 to 7, enabling the removal of up to 91% of Cu(II) ions and up to 75% of Zn(II) ions from aqueous solutions.

**KEY WORDS:** Sol-gel process, nanohybrid, chitosan, silica, adsorbent, copper (*II*) and zinc(II) ions.

## I.INTRODUCTION

The industrial revolution has significantly increased the discharge of pollutants into the environment, with heavy metals being among the most prevalent. Metals like Pb, Cu, Ni, Zn, Cd, and Cr are extremely toxic to humans and animals, making their presence in surface water and groundwater at concentrations exceeding natural background levels highly undesirable. Excess Cu(II) ion in organisms can cause severe health issues, including liver, brain, heart, skin, and pancreatic problems. Similarly, Zn(II) ion toxicity in humans triggers nausea, dizziness, and dehydration. Therefore, removing heavy metals from water is vital to protect public health, as pollution levels exceed the environment's self-purification capacity [1-3].

Toxic metal-containing wastewater can be treated using anions to precipitate metals as insoluble salts. Other methods, such as membrane filtration, activated carbon adsorption, and ion-exchange resins [4], are widely used for industrial wastewater treatment but show limited efficiency due to weak interactions with metal cations. To overcome this, researchers have developed advanced functional adsorbents like hybrid materials, organoclays, and surface-modified mesoporous substances.

Recent progress in nanotechnology and engineering has led to innovative solutions for numerous challenges in water filtration and purification. Techniques involving nanosorbents, nanocatalysts, bioactive nanoparticles, nanostructured catalytic membranes, and nanoparticles are also reported to improve the efficiency of water purification [6,7]. In the case of wastewater treatment, an ideal adsorbent for heavy metal ions should possess properties such as a large surface area, appropriate pore size, pore volume, high adsorption capacity, high mechanical stability, good compatibility, availability, cost-effectiveness, and environmental friendliness.
Biopolymers, such as chitosan, have demonstrated high efficiency in binding heavy metals due to the presence of functional groups that facilitate the formation of chemical bonds with metal ions [8]. However, the activity of

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these adsorbents can be limited by their physical properties. Modification with silica significantly enhances sorption characteristics by creating composites with improved mechanical strength and increased surface area [9]. Based on this, one of the primary objectives of this study is the development of a novel aminofunctional silica-polysaccharide adsorbent that combines the advantages of both components, enabling the effective removal of Cu(II) and Zn(II) ions from contaminated waters.

Hybrid materials incorporating chitosan and different inorganic substances have been developed, showcasing significant benefits, particularly in terms of enhanced mechanical strength. As a result, combining macromolecules with inorganic particles has emerged as a highly innovative and promising field of study. These composite materials display notable improvements in catalytic, optical, thermal, and mechanical properties, attributed to the synergistic interactions whether physical or chemical between their organic and inorganic components. Examples of various polysaccharide/inorganic combinations include cellulose/silica, chitosan/silica, chitosan/titanium dioxide, and chitosan/zeolite A [10-12].

This study aimed to develop and characterize an amino-functionalized silica-polysaccharide adsorbent with improved mechanical strength, as well as to assess its adsorption capabilities for Cu(II) and Zn(II) ions.

## II. METODOLOGY

An aminofunctionalized silica-polysaccharide adsorbent was synthesized following the methodoly detailed in [13].
The synthesized adsorbent was examined using scanning electron microscope (SEM, JEOL JSM-6510LA), which was coupled with energy-dispersive X-ray spectroscopy (EDX) and operated at an accelerating voltage of 10 kV. Infrared spectra of the samples were obtained with Fourier transform infrared (FT-IR) spectrophotometer (Bruker, FT-IR Spectrometer, USA). The samples were prepared for analysis by grinding in an agate mortar and forming tablets with the assistance of KBr.

Surface area and pore volume measurements were performed using nitrogen porosimetry at -77 °C on an automated adsorption instrument (ASAP 2020, Micromeritics, USA). Prior to these measurements, the samples underwent degassing at 50 °C.

The adsorption behaviour of Cu(II) and Zn(II) ions was investigated under static conditions with periodic stirring according to a standard procedure. In particular, 100 mg of the chitosan/silica adsorbent was introduced into 25 ml aqueous solutions of copper and zinc sulfates at varying concentrations (ranging from 5 mg/L to 900 mg/L) and different pH levels (1.0 to 8.0). These pH levels were initially adjusted using $HNO_3$ or NaOH but were not monitored during the experiment. The process was carried out at room temperature, with the mixture stirred at 600 rpm. After the required time, the aqueous phase and adsorbent were separated via filtration, and the metal ion concentrations in the filtrate were determined using an atomic absorption spectrophotometer.

The amount of metal ions adsorbed per unit mass of chitosan/silica adsorbent (mg metal ions/g adsorbent) estimated using the following expression:
Adsorbed metal ions = $[(C_\circ - C) \times V] = [m \times 1000]$ where $C_\circ$ and C are the concentrations of metal ions in the aqueous phase before and after the incubation period, respectively (mg/L); V – volume of the aqueous phase (mL); m – amount of adsorbent used (g).

## III. EXPERIMENTAL RESULTS

The morphological analysis of the synthesized chitosan/silica hybrid material, conducted using SEM, revealed the formation of spherical particles with diameters ranging between 3 -15 µm (Fig. 1.a).

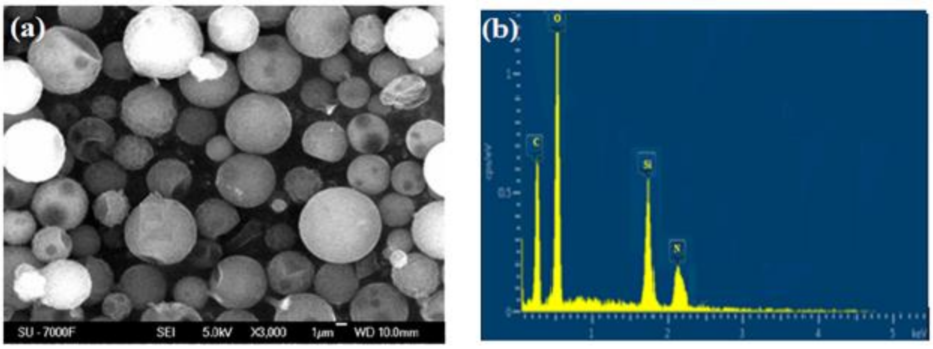


**Fig. 1.** *SEM (a) and EDS (b) analyses of chitosan/silica nanohybrid adsorbent sample.*

Element analysis conducted using the SEM-EDS method (Fig. 1(b)) identified the presence of O (52.1%), C (31.5%), Si (19.4%), and N (7.51%), confirming the composition of silica and chitosan macromolecules within the structure of the synthesized material.

This finding is further supported by IR spectroscopy analysis results (Fig. 2). The observed shift of absorption bands corresponding to the functional groups of pure chitosan (-$NH_2$, -$CONH_2$) in the IR spectrum of the final adsorbent suggests interactions between the functional groups of chitosan and the silanol groups of silica. A comparison of the IR spectra of the nanohybrid adsorbent samples with that of chitosan reveals a shift in the absorption bands situated at 1650 $cm^{-1}$ and 1584 $cm^{-1}$, characteristic of the carbonyl amide groups of chitosan, to lower frequencies of 1618 $cm^{-1}$ and 1424 $cm^{-1}$, respectively. This indicates the formation of hydrogen bonds between the silanol groups and the carbonyl groups in the amide functionality of chitosan.

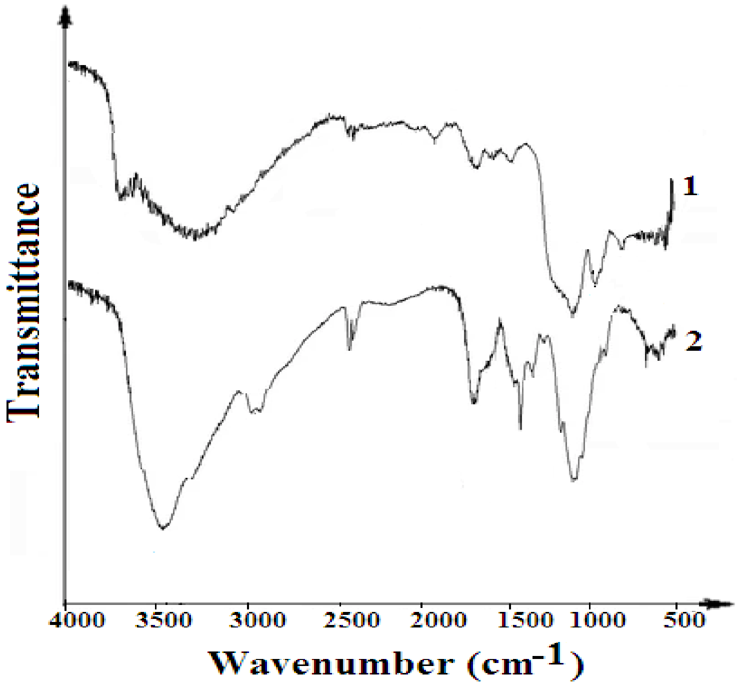


**Fig. 2**. *IR spectra of (1) pure chitosan and (2) chitosan/silica composite adsorbent.*

The structural parameters determined by porosimetry, including BET specific surface area of 292 $m^2/g$ and an average pore diameter of 42 nm, along with the adsorption-desorption isotherm shape corresponding to Langmuir type V isotherm (according to IUPAC classification) (Fig. 3), confirm that the synthesized chitosan/silica adsorbent features a mesoporous structure. The adsorption behaviour of the chitosan/silica nanohybrid adsorbent towards Cu(II) and Zn(II) ions was evaluated using solutions within a pH range of 1 to 8.

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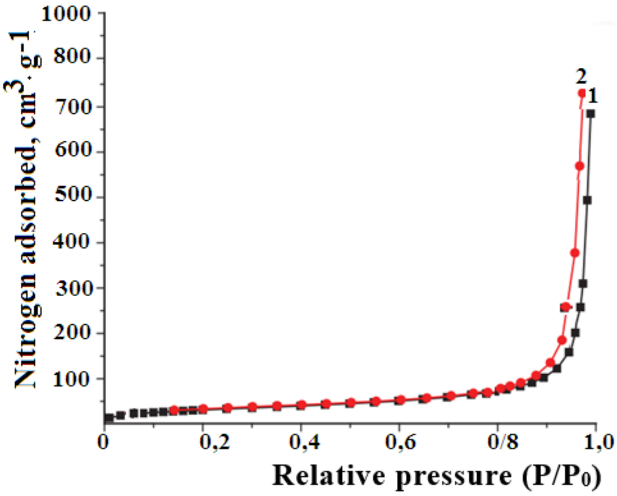


**Fig.3.** *Nitrogen (1) adsorption / (2) desorption isotherm on the chitosan/silica nanohybrid adsorbent.*

As is widely recognized, the pH level of the solution is a key factor in adsorption processes, profoundly affecting the surface charge variations of the adsorbent [14]. Figure 4 demonstrates how the adsorption efficiency of Cu(II) and Zn(II) ions varies with the pH values of the tested solutions.

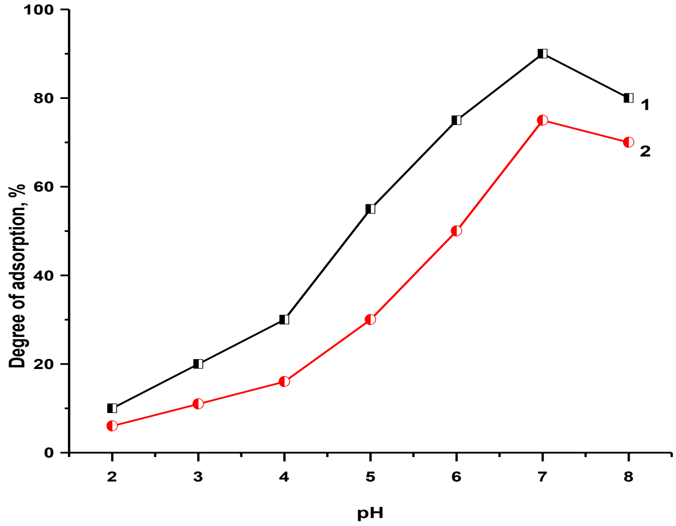


**Fig. 4.** *Dependency between the adsorption efficiency of Cu(II) and Zn(II) ions on the pH values of the analysed solutions.*

The analysis of the curves indicates that as acidity decreases (i.e., the pH of the solutions rises to 4), the adsorption levels of both metal ions increase similarly up to pH=7. However, beyond this point, when the pH is raised to 8, a decline in adsorption levels is observed. This shows that highly acidic or strongly alkaline conditions deactivate the adsorption sites on the surface of the chitosan/silica nanohybrid adsorbent. The optimal pH range for efficiently adsorbing Cu(II) and Zn(II) ions lies between 2 and 7. Within this range, the adsorption of Cu(II) ions ranges from 57% to 91%, while for Zn(II) ions, it reaches up to 75%.

## IV. CONCLUSION AND FUTURE WORK

The nanohybrid chitosan/silica adsorbent we developed and analysed demonstrates notable adsorption efficiency for Cu(II) and Zn(II) ions within the pH range of 2 to 7. It can extract up to 91% of Cu(II) ions and 75% of Zn(II) ions from the tested solutions. As such, this pH range is deemed optimal, making the adsorbent highly suitable for removing heavy metal ions from contaminated water.

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