Chemical Journal of Kazakhstan
Volume 2, Number 94(2026), 158-167 https://doi.org/10.51580/2026-2.2710-1185.23

UDC 54.057

SYNTHESIS AND PHYSICOCHEMICAL CHARACTERIZATION OF CHITOSAN–SiO_2-BASED COMPOSITES

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Abstract. *Introduction.* Composites based on chitosan are of particular interest owing to their biodegradability, environmental safety, and strong affinity for metal ions. Immobilization of chitosan on an inorganic support such as silica can be used as a method to improve physicochemical properties of hybrid materials such as their thermal stability and mechanical strength. *Objectives.* The main purpose of this work was preparation of chitosan–SiO_2 composites with different chitosan loadings and determination of their physicochemical properties. *Methods.* Composite chitosan–SiO_2 with 5, 10, and 20 wt.% content of chitosan was synthesized via adsorption immobilization followed by alkaline precipitation. Physical characteristics of obtained materials were studied using methods such as thermo gravimetric analysis (TGA), nitrogen adsorption–desorption analysis (BET), and scanning electron microscopy (SEM). *Results and discussion.* In our study, we showed that alkaline precipitation greatly increased the efficiency of adsorption and immobilization of chitosan, giving immobilization yields of 93-100%. From thermo gravimetric analysis, it was found that content of organic substances in the materials is gradually increased by increasing chitosan loading. According to the classification of the IUPAC, the isotherm is classified as type IV isotherms with H3 hysteresis loop characteristic for mesoporous materials. Scanning electron microscopy revealed heterogeneous and relatively rough surface morphology. *Conclusion.* Obtained composite materials have developed mesoporous structure, high thermal stability, and good immobilization of chitosan, which allows to consider them as prospective sorbents for heavy metal ion elimination from aqueous solutions.

Keywords: chitosan, silicon dioxide, composite materials, adsorption immobilization, mesoporous materials

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Citation: Saryuisin S.N., Korganbaeva Zh.K., Talgatov E.T., Tusupkaliyev E.A., Kainarbayeva Zh.N. Synthesis and physicochemical characterization of chitosan– SiO_2-based composites. *Chem. J. Kaz.*, **2026**, 2*(94)*, 158-167. DOI: https://doi.org/10.51580/2026-2.2710-1185.23

Introduction

Heavy metal pollution in our water systems is still a big environmental challenge [1, 2]. That's mainly because these metal ions are toxic, stick around for a long time, and can build up in living organisms. Among the various pollutants, nickel (Ni(II)) and cobalt (Co(II)) are commonly found in industrial wastewater from electroplating, mining, metallurgy, and chemical processes [3]. High levels of these metals can lead to serious environmental and health issues, which is why there's a strong interest in developing efficient and eco-friendly materials to remove them [4].

One of the best ways to tackle this problem is through adsorption, as it's a straightforward and cost-effective method to extract heavy metal ions from water. Because of its simplicity and efficiency, there's been a growing focus on biopolymer-based adsorbents lately, thanks to their low toxicity, biodegradability, and easy availability [5, 6].

Chitosan stands out as a highly promising natural polymer for adsorption due to its amino and hydroxyl groups that can effectively bond with metal ions [7]. But there are some drawbacks; pure chitosan lacks mechanical stability and doesn't hold up well in acidic environments, which limits its use [8]. To overcome this, researchers have started to combine chitosan with inorganic materials like SiO_2 to enhance its structural integrity, surface characteristics, and overall adsorption capabilities.

Mesoporous silica is a great choice for support because it has a high surface area, a well-developed porous structure, and surface silanol groups, making it ideal for immobilizing polymers [9]. By combining chitosan with SiO_2, it’s possible to create hybrid materials that have improved physical and adsorption properties.

Therefore, the aim of this work was to prepare chitosan–SiO_2 composites with different polymer contents and investigate their physicochemical properties using thermogravimetric analysis, nitrogen adsorption–desorption analysis and scanning electron microscopy.

Experimental part

Materials and reagents

Silicon dioxide (SiO_2) and chitosan (degree of deacetylation ≥75%, derived from shrimp shells) were used as the main reagents and purchased from Sigma-Aldrich (Germany). Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used in the experiments. All chemicals and reagents were of analytical grade and used without further purification.

Preparation of chitosan–SiO_2 composites

A 1% chitosan solution was prepared by dissolving 1.0 g of chitosan in 100 ml of 1% hydrochloric acid solution (Figure 1a). The working HCl solution was prepared by diluting 2.34 ml of concentrated HCl with distilled water to a final volume of 100 ml. Dissolution was carried out at room temperature under magnetic stirring until complete polymer dissolution.

Chit/SiO_2 composites containing 5, 10, and 20 wt.% of chitosan were prepared by adsorption immobilization from solution. For this purpose, 1.0 g of SiO_2 was dispersed in 34.7, 28.9, and 15 mL of distilled water, respectively, and stirred for 10–15 min at room temperature. Then, 5.3, 11.1, and 25 ml of 1% chitosan solution were added to the suspensions (Figure 1b). In all cases, the total volume of the system was adjusted to 40 ml. The resulting suspensions were stirred for 1 h, after which the pH was adjusted to 7.5 using NaOH solution in order to decrease the protonation degree of chitosan amino groups and enhance its adsorption onto the SiO_2 surface (Figure 1c). The mixtures were then additionally stirred for 2 h. The systems were left at room temperature for 12 h to reach adsorption equilibrium (Figure 1d). The solid phase was separated by filtration, washed with distilled water until neutral pH, and dried at room temperature to constant weight (Figure 1e). The filtrates were collected and analyzed by viscometry.

Thus, SiO_2 composite samples modified with different chitosan contents were obtained and further used for physicochemical characterization and adsorption studies.

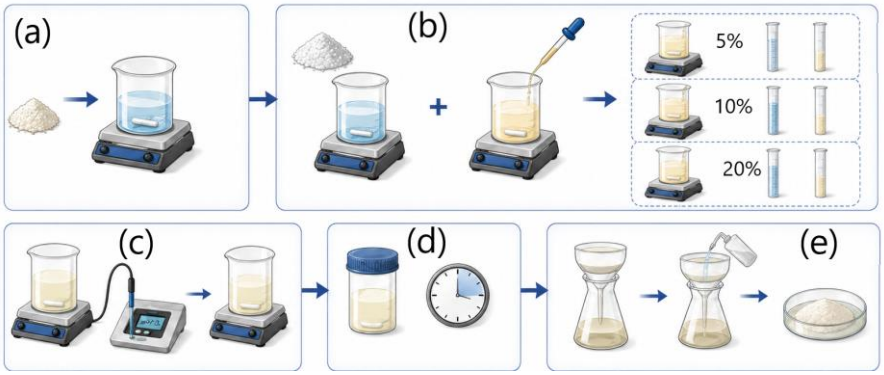


Figure 1 –Experimental procedure for the preparation of chitosan–SiO_2 composites

Results and discussion

The chitosan-SiO_2 composites containing 5, 10, and 20 wt.% of chitosan were synthesized via the adsorption immobilization method. The extent of polymer adsorption on the surface of the matrix was calculated based on the remaining amount of the polymer in the mother liquor after adsorption. The measurement of mother liquor viscosity allowed estimating the remaining chitosan amount based on the previously constructed calibration curve.

Quantitative data of chitosan adsorption in Chit/SiO_2 composites, fabricated both in the absence and in the presence of an alkaline precipitation, are presented in Table 1. It has been established that the synthesis method significantly affects the effectiveness of the composite formation. In the samples synthesized without

precipitation, the adsorption degree stayed comparatively low at 28–30%, regardless of the increasing initial chitosan content. This can be caused by low interaction strength between chitosan macromolecules and the silica surface at acidic pH due to the repulsion forces acting between molecules [10]. Consequently, only a low number of polymer molecules are adsorbed, and hence, the content of chitosan in composites becomes low. This is associated with protonation of amino groups (- NH_3^+) preventing their interaction with silica silanol groups (Si-OH) [11].

However, in the samples prepared through an additional alkaline precipitation, a considerable increase in adsorption degree up to 93–100% was observed. It means that nearly all initial chitosan is adsorbed and immobilized on the silica surface. It is connected with the shift in the pH value towards neutral and the corresponding deprotonation of amino groups ($-NH_3^+ \rightarrow -NH_2$). As a result, interaction between polymer and silica becomes more intensive.

Therefore, the polymer content in the obtained composites was significantly higher than in precipitate-free samples and reached the level of 4.7, 10, and 20 wt.% according to the initial loading.

Table 1 – The results of the assessment of chitosan content in Chit /SiO_2 composites

m(Chit) in the Initial Solution, mg	m(Chit) in Solution after Sorption, mg	m(Chit) Adsorbed, mg	Adsorption Degree, %	Chit Content, %
Chit/SiO_2 without precipitation				
53	38.0	15.0	28	1.4
111	79.2	31.8	29	2.9
250	175.0	75.0	30	6.0
Chit/SiO_2 with precipitation (by NaOH)				
53	4.0	49.0	93	4.7
111	0.0	111.0	100	10.0
250	0.0	250.0	100	20.0

The thermal behavior of pure chitosan and Chit/SiO_2 composites with different polymer contents was investigated using TG–DTA analysis (Figure 2). Pure chitosan exhibited significant thermal degradation accompanied by a total mass loss of approximately 66.9%. The initial weight loss below 150–180℃ was associated with the removal of physically adsorbed and bound water molecules. The main decomposition stage started at approximately 292.5℃ and corresponded to degradation of the chitosan polymer backbone, including decomposition of saccharide and amino-containing groups. In comparison, the Chit/SiO_2 composites demonstrated considerably higher thermal stability due to the presence of the inorganic silica matrix. All composites showed gradual mass loss with increasing temperature while preserving the characteristic thermal decomposition behavior of immobilized chitosan. The first weight-loss stage below 200℃ was mainly related to desorption of adsorbed water and partial

dehydroxylation of silanol groups. The corresponding mass losses were approximately 5.01%, 5.07%, and 5.18% for 5%, 10%, and 20% Chit/SiO_2 composites, respectively. The second decomposition stage observed in the range of approximately 250–400℃ was attributed to thermal degradation of the immobilized chitosan phase. The mass loss in this region increased with increasing polymer content and reached 3.38%, 3.89%, and 7.85% for 5%, 10%, and 20% Chit/SiO_2 composites, respectively. The total mass losses for the composites were 11.86%, 14.54%, and 18.56%, respectively. The progressive increase in mass loss with increasing chitosan loading confirms the successful incorporation of chitosan into the SiO_2 matrix and the formation of hybrid organic–inorganic composites. As one can see, the experimental data agree well with literature sources [12].

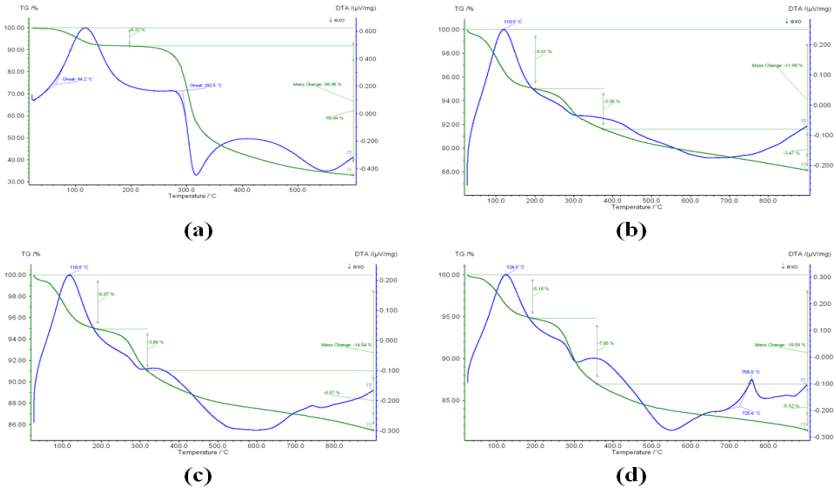


Figure 2 – TGA and DTA of (a) chitosan (b) 5% Chit/SiO_2 (c) 10% Chit/SiO_2 (d) 20% Chit/SiO_2

The probable process of the interaction of chitosan with the SiO_2 surface is depicted in Figure 3. At an acidic pH value, chitosan dissolves owing to the protonation of amino groups with the generation of $–NH_3^+$ groups. During the neutralization reaction with NaOH and the further increase in pH value up to 7.5, the deprotonation of amino groups occurs partially, enhancing the capability of chitosan macromolecules to bind with the SiO_2 surface. The binding of chitosan molecules with silica is governed mainly by hydrogen bonds and physical adsorption.

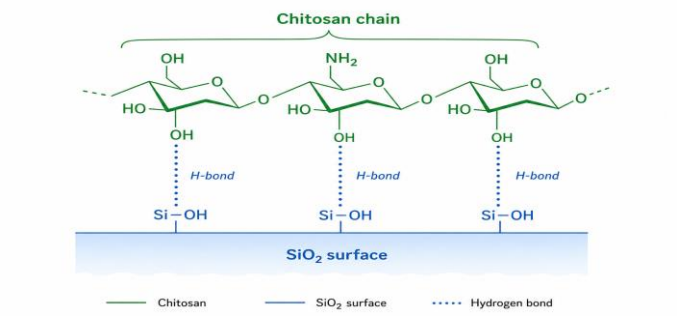
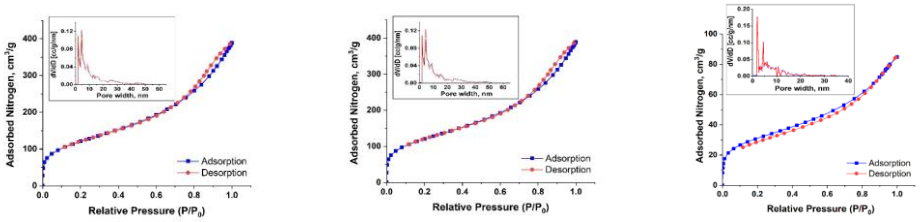


Figure 3 – Schematic illustration of the interaction between chitosan chains and the SiO_2 surface via hydrogen bonding

Figure 4 shows the adsorption-desorption isotherms and the pore size distribution for SiO_2, chitosan, and 10% Chit/SiO_2. Based on the classification of adsorption isotherms by IUPAC, the isotherms are classified as type IV adsorption isotherms with the existence of hysteresis loops of H3 type [13]. Silica is characterized by the mesoporous structure with a high value of nitrogen adsorption in the whole range of relative pressures. A hysteresis loop at medium and high relative pressure indicates capillary condensation inside mesopores. Additionally, the pore-size distribution proves the prevalence of mesopores with uniform pore structure [14].

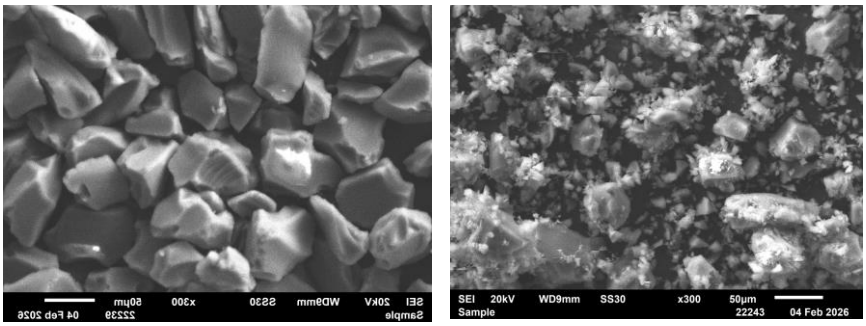


(a) (b) (c)

Figure 4 - N_2 -adsorption−desorption isotherm plots of (a) SiO_2 (b) Chitosan (c) 10%Chitosan-SiO_2 composite. The insets show the corresponding pore-size distribution

A much lower amount of nitrogen absorption by the chitosan sample indicates poor development of the porous structure and low specific surface area. The 10% Chit/SiO_2 composite still preserves mesoporous character of silica; however, the volume of nitrogen adsorption decreased after the immobilization process. It occurs due to the blockage of silica pores with chitosan macromolecules. Nevertheless, preservation of isotherms of type IV indicates that silica mesoporous structure has not been destroyed completely. The analysis of experimental results confirms the successful immobilization of chitosan molecules to the surface of silica without a complete breakdown of its porous structure.

Morphology of the surface of 10% Chit/SiO_2 composite was studied using scanning electron microscopy and the corresponding micrograph is provided in Figure 5. Composite material demonstrates heterogeneity and rather rough surface morphology featuring clusters of agglomerated particles. As compared to pure silica described in the literature sources, the surface morphology became less uniform as a result of chitosan immobilization, which indicates that the polymer layer was formed on the surface of silica nanoparticles [15]. Formation of clusters can be attributed to possible interaction of chitosan molecules and silica nanoparticles during the immobilization step. Moreover, no big crystallites or phase separation was noticed, which means that chitosan molecules are homogeneously distributed in the composite material. Surface morphology and porosity of the developed composite material are beneficial for adsorption, because they provide a lot of active sites for ion adsorption.



(a) (b)

Figure 5 - SEM micrograph of the (a) SiO_2 (b) 10%Chitosan-SiO_2

Conclusion

Successful synthesis of the Chitosan-SiO_2 composites at different polymer loadings was realized through the method of adsorption immobilization from solution and alkaline precipitation. Obtained results proved the important role of pH regulation with sodium hydroxide during immobilization process, providing maximum chitosan adsorption efficiency up to 93-100%. It was found via thermogravimetric analysis that the introduction of chitosan into silica resulted in gradual rise of material's organic content. The nitrogen adsorption-desorption isotherms proved the formation of mesoporous structure in the resulting composites with the typical type IV adsorption behavior and hysteresis loop H3 according to the International Union of Pure and Applied Chemistry classification. Moreover, results of BET analysis confirmed the presence of partially filled pores, proving that specific surface area and total pore volume decreased at an increase in the polymer content in composite composition. In conclusion, obtained results proved that synthesized Chitosan-SiO_2 composites are prospective hybrid materials for their further use as adsorbents for selective removal of heavy metals ions from aqueous solutions.

Conflict of interests: The authors declare that there are no conflicts of interests between the authors to disclose in this article.

ХИТОЗАН– SiO_2 НЕГІЗІНДЕГІ КОМПОЗИТТЕРДІ СИНТЕЗДЕУ ЖӘНЕ ОЛАРДЫҢ ФИЗИКА-ХИМИЯЛЫҚ СИПАТТАМАЛАРЫ

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Түйіндеме: *Кіріспе.* Хитозан негізіндегі композиттік материалдар соңғы жылдары биологиялық ыдырағыштығы, экологиялық қауіпсіздігі және металл иондарын тиімді байланыстыру қабілетінің арқасында зерттеушілердің үлкен қызығушылығын тудырып отыр. Осындай материалдардың қасиеттерін жақсартудың тиімді тәсілдерінің бірі – хитозанды бейорганикалық тасымалдағыштардың, әсіресе диоксид кремнийдің бетіне иммобилизациялау болып табылады. SiO_2 қолдану алынған гибридті жүйелердің механикалық беріктігі мен термиялық тұрақтылығын арттыруға, сондай-ақ олардың пайдалану сипаттамаларын жақсартуға мүмкіндік береді. *Зерттеудің мақсаты.* Құрамындағы хитозан мөлшері әртүрлі хитозан–SiO_2 композиттерін синтездеу және олардың физика-химиялық қасиеттерін зерттеу болды. *Әдістемесі.* Құрамында 5, 10 және 20 масс.% хитозан бар композиттер ерітіндіден адсорбциялық иммобилизациялау және кейінгі сілтілік тұндыру әдісі арқылы алынды. Алынған материалдардың қасиеттерін зерттеу үшін термогравиметриялық талдау (ТГА), азоттың төмен температурадағы адсорбциясы (БЭТ) және сканерлеуші электрондық микроскопия (СЭМ) әдістері қолданылды. *Нәтижелер және талқылау.* Зерттеу нәтижелері сілтілік тұндыру процесі хитозанның SiO_2 бетіне бекітілу тиімділігін едәуір арттыратынын көрсетті, бұл жағдайда иммобилизация дәрежесі 93–100% аралығында болды. Термогравиметриялық талдау нәтижелері композит құрамындағы хитозан мөлшері артқан сайын органикалық бөліктің де біртіндеп көбейетінін дәлелдеді. IUPAC классификациясына сәйкес алынған изотермалар H3 гистерезис ілмегі бар IV типке жатады, бұл материалдардың мезокеуекті құрылымға ие екенін көрсетеді. СЭМ арқылы жүргізілген талдау композиттердің беті біркелкі емес және салыстырмалы түрде кедір-бұдырлы морфологиямен сипатталатынын көрсетті. *Қорытынды.* Осылайша, синтезделген хитозан–SiO_2 композиттері дамыған мезокеуекті құрылымымен, жоғары термиялық тұрақтылығымен және полимердің тиімді иммобилизациялануымен ерекшеленеді. Мұндай қасиеттер олардың ауыр металл иондарын сулы ерітінділерден бөліп алуға арналған перспективті сорбенттер ретінде қолданылуына мүмкіндік береді.

Түйінді сөздер: хитозан, кремний диоксиді, композиттік материалдар, адсорбциялық иммобилизация, мезокеуекті материалдар

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СИНТЕЗ И ФИЗИКО-ХИМИЧЕСКАЯ ХАРАКТЕРИСТИКА КОМПОЗИТОВ НА ОСНОВЕ ХИТОЗАНА И SiO_2

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Резюме. *Введение.* Композиты на основе хитозана в последние годы привлекают значительное внимание благодаря сочетанию биоразлагаемости, экологической безопасности и способности эффективно связывать ионы металлов. Одним из подходов к улучшению свойств таких материалов является иммобилизация хитозана на поверхности неорганических носителей, в частности диоксида кремния. Использование SiO_2 позволяет повысить механическую прочность и термическую устойчивость получаемых гибридных систем, а также улучшить их эксплуатационные характеристики. *Целью данной работы* являлось получение композитов хитозан–SiO_2 с различным содержанием полимера и исследование их физико-химических свойств. *Методика.* Синтез композитов с содержанием хитозана 5, 10 и 20 масс.% проводили методом адсорбционной иммобилизации из раствора с последующим щелочным осаждением. Для характеристики полученных материалов использовали методы термогравиметрического анализа (ТГА), низкотемпературной адсорбции азота (БЭТ) и сканирующей электронной микроскопии (СЭМ). *Результаты и обсуждения.* Результаты исследования показали, что проведение щелочного осаждения существенно повышает эффективность закрепления хитозана на поверхности SiO_2, обеспечивая степень иммобилизации в пределах 93–100%. Данные термогравиметрического анализа свидетельствуют о постепенном увеличении содержания органической составляющей при повышении доли хитозана в составе композитов. Согласно классификации IUPAC, полученные изотермы относятся к IV типу и характеризуются наличием петли гистерезиса H3, что указывает на мезопористую структуру материалов. Анализ поверхности методом СЭМ показал, что композиты обладают неоднородной и сравнительно шероховатой морфологией поверхности. *Заключение.* Таким образом, синтезированные композиты хитозан–SiO_2 характеризуются развитой мезопористой структурой, высокой термической стабильностью и эффективной иммобилизацией полимера. Совокупность полученных свойств позволяет рассматривать данные материалы как перспективные сорбенты для извлечения ионов тяжелых металлов из водных растворов.

Ключевые слова: хитозан, диоксид кремния, композиционные материалы, адсорбционная иммобилизация, мезопористые материалы

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