

STABILIZATION OF IRON NANOPARTICLES BY BIOPOLYMERS DURING CHEMICAL REDUCTION WITH SODIUM BOROHYDRIDE

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Abstract. *Introduction.* One of the effective ways to regulate the processes of formation and agglomeration of FeNPs is the use of biopolymer stabilizers. *The goal* is to investigate the influence of biopolymer stabilization on the size, morphological and crystalline characteristics of FeNPs obtained by chemical reduction of NaBH₄. *Methods.* FeNPs were synthesized by reduction of Fe³⁺ with NaBH₄, stabilized with chitosan and sodium carboxymethylcellulose. The optical properties were studied using UV-Visible spectrophotometry, FTIR to identify functional groups, SEM-EDS and TEM to examine morphology and elemental composition, XRD to determine crystalline phases, and DLS-ELS to evaluate hydrodynamic diameter and ζ-potential. *Results and discussion.* UV-Vis spectra showed a characteristic 260-300 nm absorption band of iron-containing nanostructures. FTIR spectra indicated Fe-O bonds and interactions between FeNPs and biopolymer functional groups. SEM analysis showed reduced aggregation in FeNPs/Na-CMC and FeNPs/CS compared with unstabilized FeNPs. EDS analysis confirmed the presence of Fe and O elements and the formation of an oxide/hydroxide phase. XRD analysis revealed the formation of crystalline iron oxide phases. The average-sized crystallites are approximately 18 nm for FeNPs, 14 nm for FeNPs/Na-CMC, and 10 nm for FeNPs/CS. TEM and DLS/ELS confirmed the formation of spherical FeNP/CS nanoparticles with a size range of 20.03 ± 3.62 nm and a zeta-potential of -30.6 mV. *Conclusion.* Biopolymer stabilizers were found to reduce the growth and aggregation of FeNPs. CS effectively limits crystallite growth and suppresses agglomeration, while Na-CMC forms structurally organized composite particles. The results demonstrate the potential of using biopolymers to stabilize iron-containing nanomaterials.

Key words: iron nanoparticles, biopolymers, sodium carboxymethylcellulose, chitosan, sodium borohydride reduction

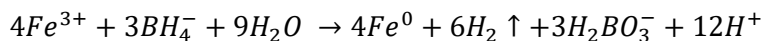
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1. Introduction

Iron nanoparticles (FeNPs) have attracted scientific interest and are widely used in medicine, biotechnology and agriculture due to their redox activity, catalytic ability, magnetic properties and environmental compatibility [1-2].

Among the chemical methods for the synthesis of FeNPs [3], the most optimal is the reduction of Fe^{3+} with sodium borohydride:



The advantages of this synthesis method include the availability of reagents and equipment, as well as the ability to control nanoscale sizes [4]. Despite the efficiency of reduction, in an aqueous environment, Fe^0 nanoparticles oxidize to form oxide phases with a core-oxide shell structure and are prone to agglomeration due to insufficient stabilization [5]. Stabilization of FeNPs during synthesis is a key factor determining particle size distribution, morphology, and colloidal stability. The use of sodium carboxymethylcellulose (Na-CMC) and chitosan (CS) biopolymers as effective FeNPs stabilizers is relevant due to their non-toxicity, biocompatibility, and complexing ability with metals. The stabilization mechanism includes the complexation of Fe^{3+} ions with nitrogen ($-NH_2$) and oxygen ($-OH$, $-COO^-$) atoms, which act as electron pair donors and form Fe-N, Fe-O donor-acceptor bonds [6,7]. In the chemical synthesis of FeNPs using the CS stabilizer, a distinctive condition is maintaining $pH \approx 5.5-6.5$ for protonation of $-NH_2$ and obtaining a polycation. Further, the interaction of Fe^{3+} with the cationic center of NH_3^+ through the “bridges” of anions and water molecules causes deprotonation of NH_3^+ due to the high affinity of Fe^{3+} for the electron pair of nitrogen. As a result, chelation of Fe^{3+} ions with the NH_2 group and the formation of the Fe-N donor-acceptor bond [8]. The aim of the study is to investigate the effect of biopolymer stabilization on the size, morphological and crystalline characteristics of FeNPs obtained by chemical reduction of $NaBH_4$.

2. Experimental part

2.1 Synthesis of FeNPs without stabilizers

To a 150 mL mixture of isopropanol and deionized water (1:1) added 0.5 g $FeCl_3 \cdot 6H_2O$ and stirred on a JOANLAB Lab 6 magnetic stirrer (China) at $50^\circ C$ and 1000 rpm for 30 minutes. The beaker with the reaction mixture was covered with aluminum foil to limit contact with air, and 50 mL of cooled 0.5 M $NaBH_4$ solution was added dropwise until a black suspension appeared. The resulting suspension was cooled to room temperature with stirring, then left at rest for 15-20 minutes. The suspension was separated on an OPN-8 centrifuge (Kyrgyzstan) at 3000 rpm. The supernatant was decanted, and the black precipitate was frozen and dried in a Scientz-12 freeze dryer (China) at $-60^\circ C$ and reduced pressure < 5 Pa. This yielded a dry powdered FeNPs product for further study.

2.2 Preparation of FeNPs stabilized by Na-CMC and CS

To synthesize FeNPs stabilized with Na-CMC (FeNPs/Na-CMC) and CS (FeNPs/CS), a solution of 0.5 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 50 mL of deionized water was added to 120 mL of 1% biopolymer solutions. The resulting reaction mixture was stirred with a magnetic stirrer at 80°C and 1500 rpm for 2 hours to ensure uniform distribution of Fe^{3+} iron ions in the biopolymer matrix. After completion of the thermal treatment, the mixture was cooled to room temperature. Next, 10 mL of a cooled 1 M NaBH_4 solution was added dropwise to the reaction mixture. To limit contact with the environment, the reaction beakers were pre-wrapped in aluminum foil. The resulting suspension was centrifuged to separate the solid nanoparticles. The resulting precipitates were frozen and dried in a Scientz-12 freeze dryer (China) at a temperature of -60°C and reduced pressure <5 Pa. The physicochemical characteristics of the biopolymers used were studied earlier in our study [9].

2.3 UV-Vis analysis

The optical absorption spectrum was measured using a PE-5400UV spectrophotometer (Russia) at a scan rate of 40 nm/min in the wavelength range of 200-600 nm. Measurements were performed in a quartz cuvette with an optical layer thickness of 10 mm.

2.4 FTIR analysis

IR spectra were obtained using a Simex FTIR-801 IR spectrometer (Russia) with Fourier transform in transmission mode, scanning was carried out with a resolution of 4 cm^{-1} in the range from 500 to 4000 cm^{-1} .

2.5 SEM/EDS analysis

The surface morphology and microstructure of the coatings were examined using scanning electron microscopy (SEM) on an SEM3200 (China) instrument with a tungsten cathode and an XFlash Detector 730M-300 (Bruker) energy-dispersive X-ray microanalysis (EDS) system. The studies were performed at an accelerating voltage of 15 kV in low vacuum mode. Micrographs were obtained at magnifications of 35 \times , 250 \times , and 500 \times .

2.6 TEM analysis

The shape and size distribution of the nanoparticles were studied using a JEOL JEM-1400 Plus transmission electron microscope (Japan). The study was conducted at an accelerating voltage of 120 keV. Aliquots of 10 μl were taken from the samples, applied to 200-mesh copper grids coated with Formvar, and dried at room temperature.

2.7 XRD analysis

The crystal structure of the samples was studied by X-ray diffraction using an X'Pert PRO diffractometer (Netherlands) equipped with monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 0.1542 \text{ \AA}$). The diffractometer was operated at an accelerating voltage of 40 kV and an X-ray tube current of 30-45 mA . Diffraction intensity was recorded over a range of 10° to 80° at an angle of 2 θ . The average nanoparticle size was calculated using the Debye-Scherrer equation:

$$d = \frac{K\lambda}{\beta \cos\theta}$$

where d - the average crystallite diameter in nm, K - the shape factor (0.9 for spherical particles), λ - the X-ray wavelength (0.15 nm), β is the width of the diffraction peak in radians, θ is the Bragg angle, 2θ [4].

2.8 DLS-ELS analysis

The hydrodynamic diameter and particle size distribution were determined by dynamic light scattering (DLS) on a Zetasizer NanoZS 90 instrument (Malvern, UK) using Zetasizer Software v.7.01. The surface charge of the particles was characterized by zeta potential values measured by electrophoretic light scattering (ELS) on the same instrument.

3. Results and discussion

3.1 Characterization of FeNPs, FeNPs/Na-CMC, FeNPs/CS

The synthesized FeNPs samples differ in appearance depending on the biopolymer stabilizers (Figure 1). The FeNPs sample (Figure 1a), obtained without stabilizers, is a dense black powder. FeNPs/Na-CMC (Figure 1b) has a looser, light gray appearance with a fibrous texture. The FeNPs/CS sample (Figure 1c) is also characterized by a non-uniform, loose, gray-black mass.

The synthesized FeNPs were characterized by ultraviolet-visible spectroscopy (UV-Vis), Fourier transform infrared spectroscopy (FTIR), scanning and transmission electron microscopy (SEM and TEM), energy dispersive analysis (EDS), X-ray diffraction (XRD), dynamic and electrophoretic light scattering (DLS and ELS).

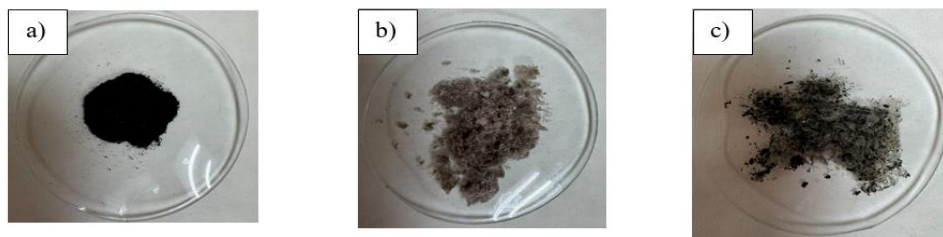


Figure 1 – Synthesized FeNPs (a), FeNPs/ Na-CMC (b), FeNPs/CS (c)

3.2 UV-Vis analysis results

UV-Vis spectra of all three samples were studied in the range from 200 to 600 nm (Figure 2a). In the region of 260-300 nm, a broad absorption band characteristic of iron-based nanoparticles is observed in all cases. The broadening of the absorption band is likely due to various electronic transitions in the iron/iron oxide system, in contrast to noble metal nanoparticles (Au, Ag), which are characterized by a clearly defined surface plasmon resonance. For the FeNPs sample, the most intense band is located at 270 nm, for FeNPs/Na-CMC it shifts

to 260 nm, and for FeNPs/CS to 280 nm. Such shifts and changes in intensity indicate that biopolymer stabilizers alter the electronic environment and optical properties of nanoparticles, affecting the surface oxide layer and the degree of aggregation [10].

3.3 FTIR analysis results

Analysis of the FTIR spectra (Figure 2b) revealed the presence of peaks at 534, 557, and 562 cm^{-1} , characteristic of the Fe-O bond. The presence of the Fe-O bond demonstrates partial oxidation of reduced iron upon exposure to air [2].

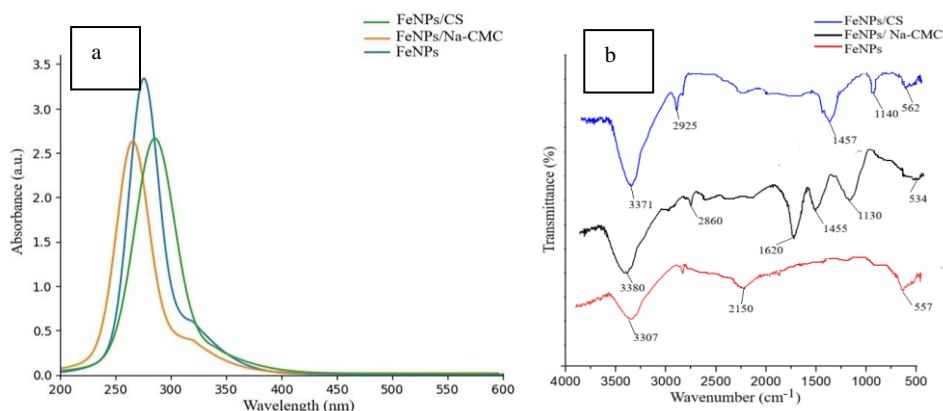


Figure 2 – Spectral characteristics (a) UV-visible spectra, (b) FTIR spectra

The FTIR spectrum of the FeNPs sample has a peak at 3307 cm^{-1} for the hydroxyl group -OH. Literature data confirm that the presence of the -OH group is associated with the formation of an oxide-hydroxide shell from the compounds $\text{Fe}(\text{OH})_2$, FeOOH , Fe_2O_3 and Fe_3O_4 [10]. The spectra of the FeNPs/Na-CMC and FeNPs/CS samples show absorption peaks characteristic of the -OH, $-\text{COO}^-$, $-\text{NH}_2$ functional groups. The peaks at 3371 and 3380 cm^{-1} correspond to the stretching vibrations of the -OH and $-\text{COO}^-$ groups. The peaks at 2860 and 2925 cm^{-1} are due to the symmetric stretching vibrations of the -C-H bond. In the FeNPs/CMC sample, the peak at 1620 cm^{-1} is due to the asymmetric vibrations of $-\text{COO}^-$ and 1455 cm^{-1} to the symmetric vibrations of COO^- . Glycosidic bonds C-O-C show characteristic peaks at wavenumbers of 1130 cm^{-1} . In the FeNPs/CS sample, the peak at 1457 cm^{-1} corresponds to the amino group $-\text{NH}_2$ of chitosan CS, and 1140 cm^{-1} is related to the stretching vibrations of the glycosidic bond C-O-C [9]. Comparison of FTIR spectra of the studied samples shows the presence of biopolymer stabilizers in the obtained nanocomposites.

3.4 SEM/EDS analysis results

The obtained SEM images show the morphology of the synthesized FeNPs (Figure 3).

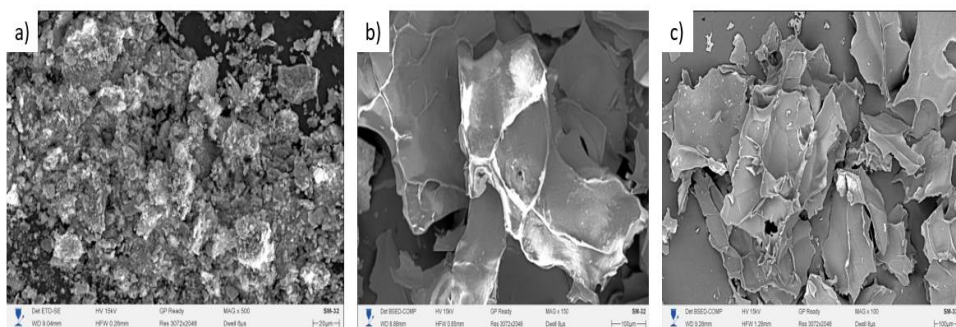


Figure 3 – SEM images of synthesized FeNPs (a), FeNPs/Na-CMC (b), FeNPs/CS (c)

The FeNPs sample without stabilizers consists of flaky aggregates 20-100 μm in size (Figure 3a). This morphology is typical of FeNPs obtained by borohydride reduction followed by partial oxidation [3]. Individual FeNPs are not clearly visible due to pronounced aggregation. The morphology of FeNPs/Na-CMC and FeNPs/CS is characterized by a large, lamellar-film structure with a smooth polymer coating relief. FeNPs/Na-CMC aggregates appear less dense compared to the FeNPs sample. FeNPs/Na-CMC have sizes up to 500 μm (Figure 3b). The ability of Na-CMC to adsorb on the surface of nanoparticles via carboxyl groups, forming steric and electrostatic repulsive interactions that lead to increased colloidal stability, has been widely discussed in the literature [6]. FeNPs/Na-CMC particle aggregates have a loose, porous surface compared to FeNPs/Na-CMC. The fine-grained structure within the aggregates is poorly visible due to complete coverage by the CS polymer matrix. Due to more effective suppression of agglomeration by the CS stabilizer, FeNPs/CS particles have sizes up to 306 μm (Figure 3c).

EDS results show the elemental composition of the resulting nanoparticles (Figure 4).

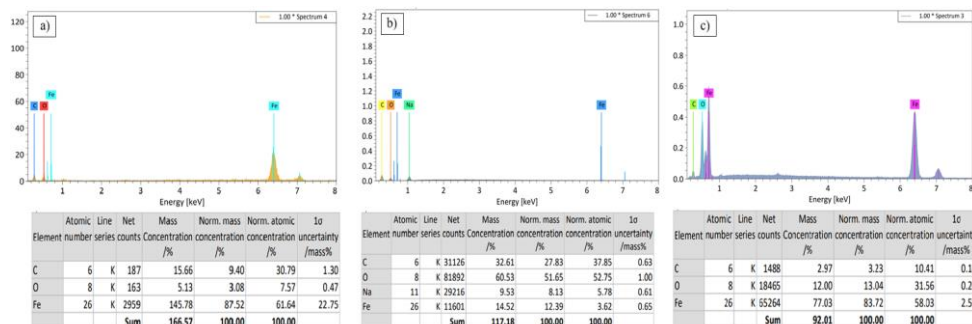


Figure 4 – EDS data of FeNPs (a), FeNPs/Na-CMC (b), FeNPs/CS (c)

Quantitative analysis of FeNPs shows that the mass fractions are 87.52% for Fe and 3.08% for O, thereby confirming the presence of Fe and O elements and the formation of an oxide/hydroxide phase. The absence of foreign elements in the EDS spectrum (Figure 4a) indicates the purity of the obtained FeNPs and confirms the effectiveness of the synthesis method using NaBH_4 [3, 5]. In the FeNPs/Na-CMC sample, in addition to Fe, O, and C, a Na peak is observed, characteristic of the Na-CMC structure. However, the mass fraction of Fe is lower (12.39%), and O is higher (51.65%) compared to other samples (Figure 4b). The lower Fe content and higher O concentration are possibly associated with an increase in the proportion of the organic phase due to stabilization by the dense polymer film of Na-CMC [10]. In the FeNPs/CS sample (Figure 4c), the Fe content is 83.72% and O content is 13.04%, indicating partial surface oxidation and the formation of an oxide shell. CS adsorbed on the nanoparticle surface also more effectively stabilizes and prevents agglomeration. The obtained SEM/EDS results confirm that biopolymers alter the surface morphology and limit the aggregation of FeNPs.

3.5 XRD analysis results

The X-ray diffraction patterns of FeNPs, FeNPs/Na-CMC and FeNPs/CS are shown in Figure 5.

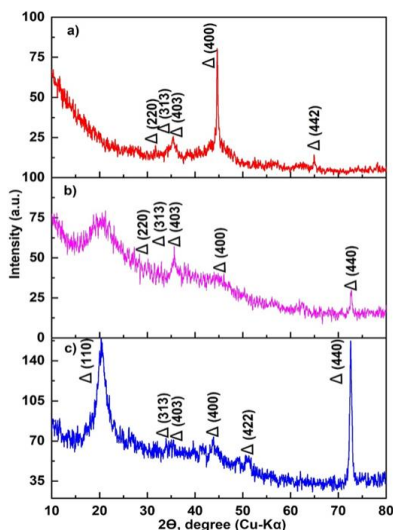


Figure 5 – Diffraction patterns of synthesized FeNPs (a), FeNPs/Na-CMC (b), FeNPs/CS (c)

The diffraction pattern of the FeNPs sample (Figure 5a) shows an intense and relatively narrow peak at 44° (400), which, along with peaks at 30.1° (220), 33.1° (313) and 35.2° (403), can be attributed to spinel-type iron oxides, thereby confirming the oxidation of zero-valent iron in the isopropanol-water system [4]. In the FeNPs/Na-CMC and FeNPs/CS samples (Figures 5b and 5c), the diffraction peaks exhibit lower intensity and a broader shape. This can be

explained by the presence of biopolymers, which reduce the degree of crystallinity and lead to a decrease in crystallite size by stabilizing the nanoparticles and preventing their aggregation [7, 8]. In the case of the FeNPs/CS sample (Figure 5c), a pronounced diffraction feature is observed near $20^\circ(110)$, which is associated with the amorphous-semicrystalline structure of CS, while an intense peak near $72^\circ(440)$ corresponds to the spinel phase of FeNPs on the CS matrix. The average crystallite size calculated using the Debye–Scherrer equation is 18 nm for FeNPs, 14 nm for FeNPs/NaCMC, and 10 nm for FeNPs/CS. Overall, X-ray diffraction analysis shows that biopolymers significantly influence the size and structure of FeNPs crystallites, facilitating the formation of stable nanocomposites. CS, in particular, has the strongest effect on the formation of the smallest crystallites. This is explained by the stronger electrostatic interaction of the polycation's $-NH_2$ groups with Fe^{3+} and the rigid and ordered conformation of the biopolymer chain.

3.6 TEM, DLS-ELS analysis results of FeNPs/CS

To confirm the X-ray diffraction data and refine the FeNPs/CS morphology, the sample was also examined using TEM and DLS-ELS. TEM analysis revealed that the FeNPs were predominantly spherical in shape and had a fairly uniform size distribution. The average nanoparticle core size was 20.03 ± 3.62 nm. The average hydrodynamic diameter of the FeNPs/CS in the dispersed medium was 127 ± 3 nm. The ζ -potential was -30.6 mV. The negative ζ -potential is due to the deprotonation of the $-NH_3^+$ groups of CS and the formation of a surface Fe-O⁻ oxide-hydrate shell. These parameters ensure the stability of the colloidal suspension.

4. Conclusion

FeNPs, FeNPs/Na-CMC and FeNPs/CS were synthesized using chemical reduction of Fe^{3+} ions with $NaBH_4$ and stabilization with biopolymers. UV-Vis, FTIR, SEM/EDS, XRD, TEM, and DLS-ELS analyses showed that large nanoparticle aggregates form in the absence of stabilizers, while the introduction of biopolymers leads to the formation of less agglomerated composite structures. When stabilized with CS, the nanocomposites have an average size of 10 nm and an ordered crystal structure (XRD), 20.03 nm (TEM), and 127 nm (DLS), indicating its effectiveness compared to Na-CMC. The resulting iron-based nanocomposites with biopolymers hold promise for practical applications in environmental monitoring and cleanup, biomedical technologies (diagnostics, delivery of biologically active substances), and agriculture (targeted delivery of microelements and increasing the bioavailability of iron for plants and microorganisms).

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Conflict of Interest: All authors declare that they have no conflict of interest.

НАТРИЙ БОРОГИДРИДИМЕН ХИМИЯЛЫҚ ТОТЫҚСЫЗДАНУ ПРОЦЕСІНДЕ
БИОПОЛИМЕРЛЕРДІҢ КӨМЕГІМЕН ТЕМІР НАНОБӨЛШЕКТЕРІН ТҰРАҚТАНДЫРУЖ.Ж. Нуртазина¹, Ж.С. Касымова^{1*}, Л.К. Оразжанова¹, Б. Леска²¹Шәкәрім университеті, Семей, Қазақстан²Адам Мицкевич атындағы университет, Познань, Польша

Түйіндемe. Кіріспе. FeNPs түзілуі және агрегациясы процестерін реттеудің тиімді тәсілдерінің бірі - биополимер тұрақтандырғыштарын қолдану. *Зерттеудің мақсаты мен міндеттері.* NaBH₄ химиялық тотықсыздануымен алынған FeNPs морфологиялық, құрылымдық және кристалдық сипаттамаларына биополимерлермен тұрақтандырудың әсерін анықтау болды. *Зерттеу әдістері.* Fe³⁺ иондарын натрий борогидридмен химиялық тотықсыздандыру және кейіннен хитозан және натрийкарбоксиметилцеллюлоза биополимерлерімен тұрақтандыру арқылы FeNPs синтезделді. Оптикалық қасиеттер УК-көрінетін спектрофотометрия арқылы зерттелді, функционалды топтарды анықтау үшін FTIR спектроскопиясы, морфология мен элементтік құрамын зерттеу үшін SEM/EDS және TEM, кристалдық фазаларды анықтау үшін рентгендік талдау және гидродинамикалық диаметр мен ζ-потенциалды бағалау үшін DLS/ELS қолданылды. *Нәтижелер мен талқылау.* UV-Vis спектрлері құрамында темір бар нанокұрылымдарға сәйкес келетін 260-300 нм аймақта тән сіңіру жолағын анықтады. FTIR талдауы нанобөлшектердің бетімен әрекеттесуге қатысатын Fe-O байланыстары мен биополимерлердің функционалдық топтарының болуын растады. SEM талдауы тұрақтандырғышсыз FeNPs ірі агрегаттар түзетінін көрсетті, ал биополимерлерді енгізу аз айқын агрегациясы бар нанокөпозиттік құрылымдардың пайда болуына әкеледі. EDS талдау Fe және O элементтерінің болуын, сондай-ақ оксид/гидроксид фазасының түзілуін растады. XRD мәліметтері бойынша темір оксидтерінің кристалдық фазаларының түзілуі анықталды. Кристаллиттердің орташа мөлшері тұрақтандырғышсыз үлгі үшін шамамен 18 нм, FeNPs/Na-CMC үшін 14 нм және FeNPs/CS үшін 10 нм болды. TEM және DLS/ELS әдістері өлшемі 20,03±3,62 нм және ζ-потенциалы -30,6 мВ болатын сфералық FeNPs /CS нанобөлшектерінің түзілуін растады. *Қорытынды.* Биополимер тұрақтандырғыштары борогидридті тотықсыздандыру кезінде FeNPs өсуін және агрегациясын төмендететіні анықталды. CS кристаллиттің өсуін шектеудің және агрегацияны басудың ең тиімді әдісін қамтамасыз етеді, ал Na-CMC құрылымдық тұрғыдан ұйымдастырылған композиттік бөлшектердің түзілуіне ықпал етеді. Алынған нәтижелер темір бар наноматериалдарды тұрақтандыру үшін биополимерлерді қолданудың перспективтілігін көрсетеді.

Түйін сөздер: темір нанобөлшектері, биополимерлер, натрий карбоксиметилцеллюлоза, хитозан, натрий борогидридтің тотықсыздануы

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СТАБИЛИЗАЦИЯ НАНОЧАСТИЦ ЖЕЛЕЗА БИОПОЛИМЕРАМИ В ПРОЦЕССЕ
ХИМИЧЕСКОГО ВОССТАНОВЛЕНИЯ БОРОГИДРИДОМ НАТРИЯЖ.Ж. Нуртазина¹, Ж.С. Касымова^{1*}, Л.К. Оразжанова¹, Б. Леска²¹Университет Шакарима, Семей, Казахстан²Университет имени Адама Мицкевича, Познань, Польша

Резюме. Введение. Одним из эффективных способов регулирования процессами формирования и агломерации FeNPs является использование биополимерных стабилизаторов. **Цель работы.** Исследование влияния биополимерной стабилизации на размер, морфологические и кристаллические характеристики FeNPs, полученных методом химического восстановления NaBH_4 . **Методы исследования.** FeNPs синтезировали методом химического восстановления Fe^{3+} ионов NaBH_4 с последующей стабилизацией хитозаном и натрийкарбоксиметилцеллюлозой. Оптические свойства изучались с помощью УФ-видимой спектрофотометрии, использовались FTIR-спектроскопия для идентификации функциональных групп, SEM/EDS и TEM для исследования морфологии и элементного состава, рентгенодифракционного анализа для определения кристаллических фаз и DLS/ELS для оценки гидродинамического диаметра и ζ -потенциала. **Результаты и обсуждение.** UV-Vis спектры выявили характерную полосу поглощения в области 260-300 нм, соответствующую железосодержащим наноструктурам. FTIR-анализ подтвердил наличие связей Fe-O и функциональных групп биополимеров, участвующих во взаимодействии с поверхностью наночастиц. SEM-анализ показал, что FeNPs без стабилизаторов образуют крупные агломераты, тогда как введение биополимеров приводит к формированию нанокomпозитных структур с менее выраженной агрегацией. EDS анализ подтвердил наличие элементов Fe и O, а также образование оксидной/гидроксидной фазы. По данным XRD установлено образование кристаллических фаз оксидов железа. Средний размер кристаллитов составил около 18 нм для образца без стабилизаторов, 14 нм для FeNPs/Na-CMC и 10 нм для FeNPs/CS. Методы TEM и DLS/ELS подтвердили образование сферических наночастиц FeNP/CS размером $20,03 \pm 3,62$ нм и дзета-потенциалом -30,6 мВ. **Заключение.** Установлено, что биополимерные стабилизаторы уменьшают рост и агрегацию FeNPs при боргидридном восстановлении. CS обеспечивает наиболее эффективное ограничение роста кристаллитов и подавление агломерации, тогда как Na-CMC способствует формированию более структурно организованных композитных частиц. Полученные результаты демонстрируют перспективность применения биополимеров для стабилизации железосодержащих наноматериалов.

Ключевые слова: наночастицы железа, биополимеры, натрийкарбоксиметилцеллюлоза, хитозан, восстановление борогидридом натрия

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