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SYNTHESIS AND CHARACTERISTICS OF SULFATED CHITOSAN BASED ON CHITIN/CHITOSAN FROM ARTEMIA PARTHENOGENETICA CYSTS

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Abstract: *This article presents for the first-time the sulfated chitosan (SCS), synthesized on the basis of chitin/chitosan obtained from cysts of the Karakalpak population of Artemia parthenogenetica of the Aral Sea, and some of its physicochemical characteristics. Structural studies of chitin/chitosan and SCS were carried out by infrared spectroscopy. The IR spectrum data showed amide I (C=O-NHR) bands characteristic of chitin at 1640 cm^{-1}, and for chitosan absorption bands of the amino group (-NH_2) at 1588 cm^{-1}. Signals at 870 and 614 cm^{-1} and 1125-1388 cm^{-1} in SCS comply with C-O-S and S=O bonds, which indicates the inclusion of sulfate groups in chitosan. The degree of substitution (DS) established by the gravimetric method, is in line with 1.32. The molecular weights of chitosan and SCS were calculated using size exclusion chromatography (SEC). According to the calculation of SEC for chitosan, an average molecular weight (MW) complies with 16 kDa, and for SCS - 1-3 kDa.*

Keywords: *Artemia parthenogenetica cysts, chitin, chitosan, sulfated chitosan.*
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Introduction

As natural polymer, chitosan has been actively studied in the field of biomedicine in recent decades due to its biodegradability, low toxicity, mucoadhesiveness, bacteriostatic and fungistatic activity, the presence of reactive functional groups and antiseptic properties. Because of its stable crystal structure, chitosan is usually insoluble in water and soluble in dilute aqueous acidic solutions below pKa ~ 6.3, where the amino groups (-NH_2) of glucosamine units are converted into a soluble protonated form (-NH_3^+) [1]. In recent years, interest in polyampholytes soluble in water over a wide pH range has noticeably increased. In particular, there have been reports of many water-soluble derivatives of chitosan [2-4]. Among them is sulfated chitosan, the macromolecule of which contains sulfate, sulfamide and amine groups. Substitution of the sulfo-group (SO_3H) in chitosan (CS) makes it possible to obtain a sulfated derivative with valuable biomedical properties (antibacterial, anticoagulant, antitumor, etc.) [5]. When sulfating chitosan, depending on the sulfating reagents and reaction conditions (time, ratio, and temperature), it is possible to synthesize N-sulfated chitosan (N-SCS), O-sulfated chitosan (O-SCS), and N,O-sulfated chitosan (N,O-SCS). For the sulfation of chitosan, various reagents are used, including concentrated sulfuric acid, oleum, sulfuric anhydride, sulfuric anhydride/pyridine, sulfuric anhydride/trimethylamine, and chlorosulfonic acid (CSA). The most commonly used was CSA.

In this case, the reaction runs under homogeneous or heterogeneous conditions, in

such media as dimethylformamide (DMF), formamide (FA), dichloroacetic acid (DCCA), tetrahydrofuran, and formic acid at different temperature ranges or under microwave irradiation [6]. According to the authors of [7], N,O-SCS can be synthesized by reacting DMF with chlorosulfonic acid (Table 1). Table 1 below shows, for comparison, the parameters of SCS obtained under different conditions.

Table 1. Formation of SCS depending on various sulfating reagents

№	Chitosan	Name	Sulfating agent	Conditions	Sulfur content (%) or DS	Link
1	MW=15 kDa, DD=80%	6-O-SCS	$HClSO_3/H_2SO_4$	-	DS=0,95-1,0	[8]
	MW=200-300 kDa, DD=>90%		$FA/H_2SO_4/HClSO_3$	0-4°C, 3 h.	Sulfur content 9,04%	[9]
	DD=88%		$HClSO_3/H_2SO_4$	at room temperatur e. 1h.	DS=0,86	[10]
2	50-80 kDa, DD=>90%	2-N- SCS	SO_3-pyridine	60°C, 24 h.	Sulfur content 7,95%	[9]
3	-	3,6-O- SCS	$HClSO_3$*DMFA	50°C, 3 h.	DS=1,39	[10]
4	DD=89%	2-N,3,6-O-SCS	$HClSO_3$*DMFA	at room temp. 5 h.	DS=1,45	[11]
5	MW=200-300 kDa, DD=>90%	2-N,6-O-SCS	$HClSO_3$/DMFA	45-55°C, 2 h.	DS=1,96-2,25	[9]
	MW=460 kDa, DD=82%		$HClSO_3$/DMFA	at room temp. 5h.	DS=0,87	[12]
			$HClSO_3$/HCOOH	at room temp. 3h.	DS=0,67	

The aim of this work is the synthesis of sulfated chitosan based on chitin and chitosan from the cysts of the Karakalpak population of *Artemia parthenogenetica* from the Aral Sea and the description of their physicochemical and structural characteristics.

Experimental part

Chitin was obtained from substandard cysts of the Karakalpak population of *Artemia parthenogenetica* of the Aral Sea by modifying the classical method of B.V. Gaikwad and others [13]. To obtain chitosan, the method of V.V. Shikera and E.S. Batashov using the "hard method for obtaining chitosan" [14-15]. The degree of deacetylation (DD) of the obtained chitosan was 84.5%, and the average molecular weight (MW) was 16 кDa.

Preparation of chitosan for sulfation

Chitosan was dissolved in 2% CH_3OOH, then the solution was filtered to remove impurities and precipitated with a 0.1 M NaOH solution. The resulting precipitate was washed with distilled water until neutral and dried in a freeze dryer [16].

Chitosan sulfation method

Sulfated chitosan was obtained according to the method [7, 16] with minor modifications. A sulfating complex was obtained by adding dropwise 4.0 ml of $HClSO_3$ with stirring to 16 ml of formamide (FA), pre-cooled to 4°C. The reaction mixture

was left for 15 minutes until the solution reached room temperature. Then 2.0 g of chitosan was added. The resulting solution was stirred for 4 hours at a temperature of 60^0C. The reaction mixture was cooled to room temperature and then precipitated with acetone. The precipitate was collected by centrifugation. The resulting precipitate was dissolved in distilled water and then neutralized with a sodium hydroxide solution to pH 7. The insoluble part was removed by filtration. The mixture was then precipitated with 99% ethanol. The precipitate was again collected by centrifugation. The resulting precipitate was dried overnight at room temperature. SCS was obtained as a light brown, fluffy powder.

Infrared spectroscopy

An IR spectroscopic study of chitin, chitosan, and SCS was carried out in the frequency range from 4400 to 400 cm^{-1} on a Perkin Elmer Spectrum 400 IR spectrophotometer.

Gravimetric method for estalishing the degree of substitution (DS)

To establish the degree of substitution gravimetrically, 500 mg of polysaccharide sulphate was hydrolyzed in 30 ml of 10% HCl by heating to boiling. For precipitation, 50 ml of a 25% $BaCl_2$ solution was added. To remove the remaining chlorides, the precipitate was washed several times with distilled water and then dried at 180°C for 3 hours. The sulphur content (S) and the DS were calculated using the following ratios [17–18]:

$$w(S)\% = \frac{a \cdot 0{,}1374 \cdot 100}{m} \quad (1)$$

$$DS = \frac{w(S)\% \cdot 162}{3200 - w(S) \cdot 102} \quad (2)$$

a is the weight of $BaSO_4$ precipitate, *g*; 0.1374 is the coefficient for converting the weight of $BaSO_4$ precipitate to sulphur; *m* is a sample of air-dry matter, *g. w* (S)% - mass fraction of sulphur; 162 is the molar mass of repeating units of chitosan (D-glucosamine); 3200 is the atomic mass of sulphur multiplied by 100; 102 g/mol is the molar mass of SO_3Na.

SEC is one of powerful methods for investigation and determination of molar mass distribution of plant polysaccharides [19-20]. SEC of the obtained polysaccharides of chitosan was carried out on an Agilent 1260 Infinity high-speed liquid chromatograph (USA) with a refractive index detector (RI). TSK GM PW_{XL} was used as a sorbent with a linear calibration dependence in the molecular weight separation range from 100 Da to 2 10^3 kDa. The eluent flow rate was 0.8 ml/min and the volume of the injected sample was 25 µl. Chromatographic data were processed using the Windows Chemstation-7 programme.

The molecular weight of chitosan was determined using the universal calibration principle in SEC. The chromatographic column was calibrated using pullulan standards (PSS, Germany) with narrow polydispersity. The following Mark-Kuhn-Houwink constants were used for the calculation: pullulan K=1.91 10^{-4}, dl/g; a = 0.67; chitosans K = 1.38 10^{-4}, dl/g; a = 0.85 [21]. For SCS, K = 7.92 10^{-4}, dl/g; a = 1 [22].

Results and discussion

In our case, we used chitosan obtained on the basis of chitin from cysts of the Karakalpakstan population and the crustacean Artemia parthenogenetica of the Aral Sea with MW = 16 kDa and DD = 84.5% to obtain SCS. Chitosan was pre-treated by dissolving in acid followed by precipitation with alkali to remove insoluble, coarse impurities present in the original sample. As a result of purification, the physicochemical characteristics of the biopolymer were changed due to partial acid hydrolysis and corresponded MW = 10 kDa, and DD = 84.5%. Besides, the re-precipitation procedure makes it possible to obtain samples that are more homogeneous in their composition. When sulfating chitosan with CSA, depending on the reaction conditions, the sulfo-group can be introduced into positions at

C-2 (NH_2 group) and C-3 (OH group), as well as at C-6 (CH_2OH group). Fig.1 below presents the reaction for obtaining sulfate chitosan:

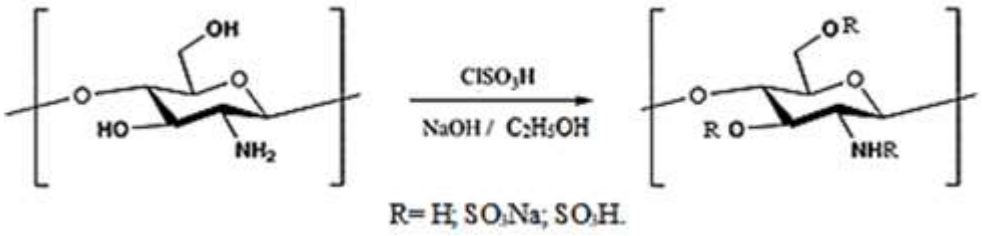

Fig. 1. Reaction to obtain sulphate chitosan

Table 2 shows the performance of SCS synthesized by the interaction of CSA with chitosan.

Table 2. Qualitative characteristics of SCS synthesized on the basis of chitosan obtained from *Artemia parthenogenetica* cysts

Characteristics	SCS
Appearance	powder
Colour	light brown
Molecular weight, kDa	1-3
Degree of substitution, %	1.32

The DS of the SCS sample was established by sulfate groups via gravimetric method.

Gravimetric analysis of sulphate chitosan

Using gravimetric analysis, the amount of sulphur in SCS was calculated according to the above method. The analyses were repeated three times and average total value obtained. It was established that the sulphur content in the obtained sulfachitosan is 14.26%, that is, the DS complies with 1.32.

IR spectra of chitin, chitosan, and SCS

To characterise the structural features of SCS, the IR spectroscopy method was used in comparison with the data on chitin and chitosan. In the IR spectrum of chitin obtained from cysts of Artemia parthenogenetica of the Aral Sea, the following absorption bands were observed (Fig. 2a): The broad absorption band at 3444 cm^{-1} complies with asymmetric and symmetric stretching vibrations ν in s (CH_2 and CH_3) of methylene and methyl groups. Absorption bands of amide I (C=O-NHR) and amino group ($-NH_2$) were observed at 1640 cm^{-1} and 1588 cm^{-1} [23], respectively. 1381 cm^{-1} corresponds to bending vibrations of C-H bonds in the CH3 group. In the spectrum, the signals at 1320–1000 cm^{-1} correspond to the stretching vibrations of the C-O, C-H, C-N, C-O-C, and C-C bonds.

In the spectra of chitosan (Fig. 2.b), a wide absorption band at 3295 cm^{-1} is due to the stretching vibrations of the hydroxyl groups (OH). Signal 2868 cm^{-1} corresponds to asymmetric and symmetric stretching vibrations, as well as s (CH_2 and CH_3) of methylene and methyl groups [24]. The absorption band of the amino group ($-NH_2$) was observed at 1588 cm^{-1} [23], and the absorption at 1380 cm^{-1} corresponds to bending vibrations of C-H bonds in the CH3 group. In the spectrum, the signals at 1320–1000 cm^{-1} corresponds the stretching vibrations of the C-O, C-H, C-N, C-O-C, and C-C bonds.The IR spectrum of sulfated chitosan (Fig. 2.c) also has characteristic absorption bands. The absorption band at 3178 cm^{-1} is due to the stretching vibrations (OH) of the hydroxyl groups. A new signal of sulfated groups is observed at 1125–1388 cm^{-1}, 870 cm^{-1}, and 614 cm^{-1} and complies with S=O and C–O–S bonds [25–26]. Compared to chitosan, sulphate chitosan has narrower vibrations of amino and hydroxyl groups. The decrease in intensity of these amino and hydroxyl groups indicates that the sulphate groups are partially attached to both the amino groups and the hydroxyl groups.

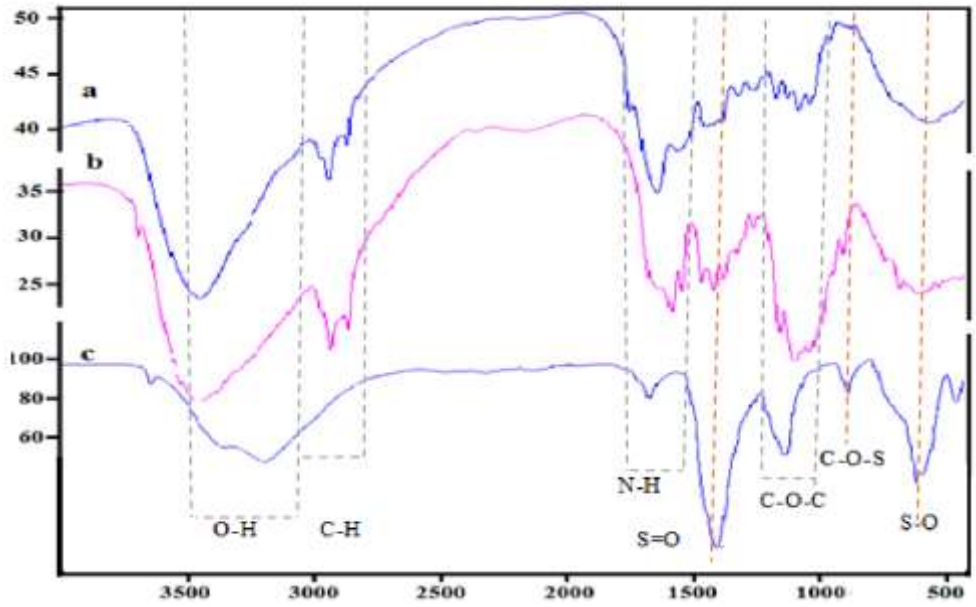

Fig. 2. IR spectra of chitin (a), chitosan (b) and SCS (c)

The results of the IR spectra are in complete agreement with the data published in the literature and confirm the formation of chitosan and SCS [27].

Determination of molecular weight (MW) of chitosan and SCS by SEC

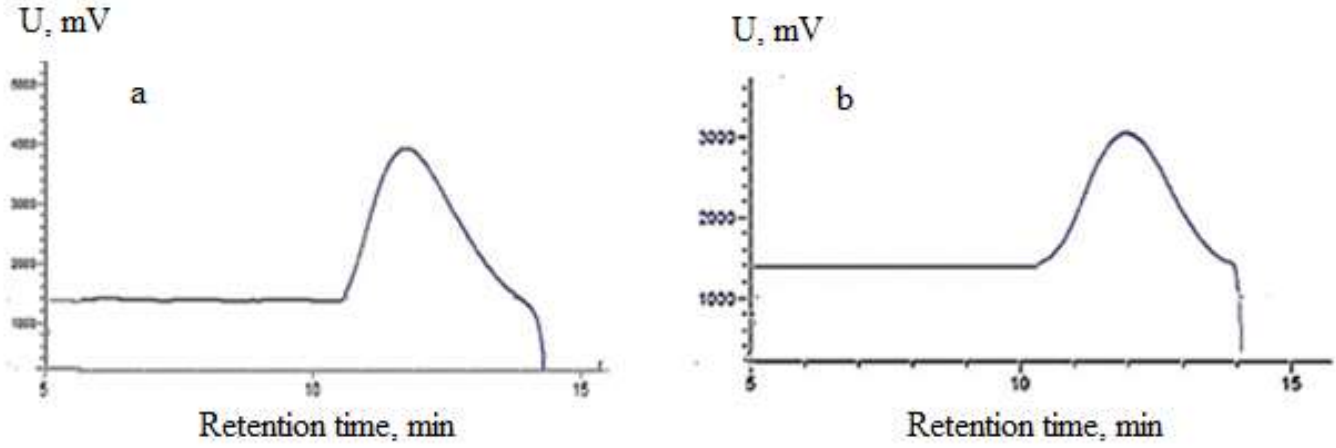

Fig. 3. Gel chromatography of non-pecipitated (a) and precipitated chitosan (b).

Fig. 3 shows the gel chromatogram of chitosan obtained in 0.2 M acetate buffer solution (pH 4.4) as eluent. The calculation of the molecular weight of chitosan was carried out by means of universal calibration in SEC. The calculated MW value for non-activated chitosan was 16 kDa (Fig.3a).

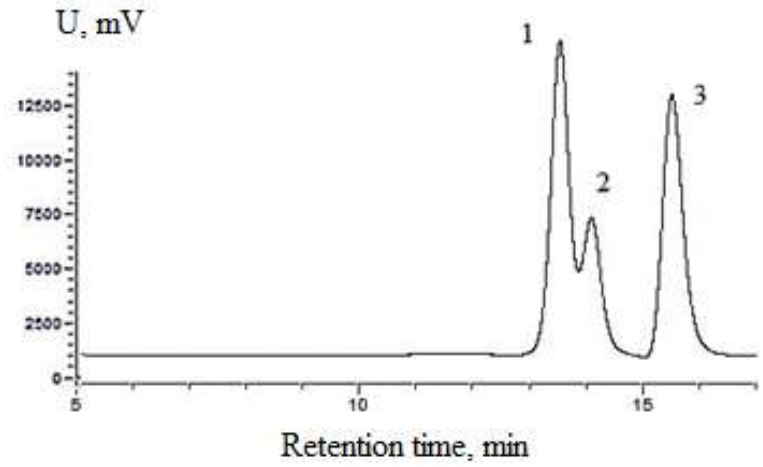

Fig. 4. Gel chromatogram of SCS with MW 3 kDa (1) and 1 kDa (2). Curve 3 is the solvent peak. Chromatography column: TSK GM PW_{XL}. Eluent: 0.1 M $NaNO_3$.

The sample has a broad MW distribution, and its polydispersity is Mw/Mn = 3. The gel chromatogram of activated chitosan is shown in Fig. 3b. The MW for activated chitosan decreased and amounted to 10 kDa. The molecular weight of SCS synthesized in a medium with CSA was determined using the SEC method (Fig.4). The molecular weight of SCS was determined according to the universal calibration principle in SEC. Two peaks (1 and 2) which comply with two fractions having molar masses of 3 kDa and 1 kDa are separated by the size-exclusion molecular separation mechanism. The chromatogram of the sample consists of two peaks (1 and 2) and a solvent peak (3), shown in Fig. 4. Two fractions with MWs of 3 kDa and 1 kDa are separated according to the size-exclusion mechanism of SEC-separation.

Conclusions

The synthesis of SCS based on chitin/chitosan obtained from cysts of the crustacean Artemia parthenogenetica of the Aral Sea was carried out. Using IR spectroscopic analysis, it was found that the sulfo groups are mainly attached to the hydroxyl groups of chitosan molecules. The gravimetric method established that the degree of SCS substitution complies with 1.32 (a sulphur content of 14.26%). Using the SEC method, it was established that the molecular weight of SCS was 1-3 kDa. The results obtained indicate that chitosan obtained from cysts of Artemia parthenogenetica from the Aral Sea can be an effective source for the SCS synthesis.

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ARTEMIA PARTHENOGENETICA KİSTLƏRİNDƏN ALINAN XİTİN/XİTOZAN ƏSASINDA SULFATLAŞDIRILMIŞ XİTOZANIN SİNTEZİ VƏ XÜSUSİYYƏTLƏRİ

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Xülasə: Bu məqalədə Aral dənizinin *Artemia parthenogenetica* kistlərindən əldə edilən xitin/xitozan əsasında sintez edilmiş sulfatlaşdırılmış xitozan (SCS) və onun bəzi fiziki-kimyəvi xüsusiyyətləri təqdim olunur. Xitin/xitozan və SCS-nin struktur tədqiqatları infraqırmızı spektroskopiya ilə aparılmışdır. İQ spektrinin məlumatları 1640 sm^{-1}-də xitin üçün xarakterik olan amid I (C=O-NHR) zolaqlarını, amin qrupunun (-NH_2) xitozan udma zolaqları üçün isə 1588 sm^{-1}-də göstərdi. SCS-də 870 və 614 sm^{-1} və 1125-1388 sm^{-1} siqnalları C-O-S və S=O rabitələrə uyğun gəlir ki, bu da xitosanın tərkibinə sulfat qruplarının daxil olduğunu göstərir. Qravimetrik üsulla təyin olunan əvəzetmə dərəcəsi 1.32-yə uyğundur. Xitozanın və SCS-nin molekulyar çəkiləri ölçüsü-istisna xromatoqrafiyasından (SEC) istifadə etməklə hesablanmışdır. Xitosan üçün SEC-in hesablanmasına görə, orta molekulyar çəki (MW) 16 kDa, SCS üçün isə 1-3 kDa uyğun gəlir.
Açar sözlər: *Artemia parthenogenetica kistləri,* xitin, xitozan, sulfatlaşdırılmış xitozan.

СИНТЕЗ И ХАРАКТЕРИСТИКА СУЛЬФАТИРОВАННОГО ХИТОЗАНА НА ОСНОВЕ ХИТИНА/ХИТОЗАНА ИЗ ЦИСТ *ARTEMIA PARTHENOGENETICA*

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Аннотация: В статье впервые представлены сульфатированный хитозан (СХС), синтезированный на основе хитина/хитозана, полученного из цист каракалпакской популяции Artemia parthenogenetica Аральского моря, и некоторые его физико-химические характеристики. Структурные исследования хитина/хитозана и СХС проводили методом инфракрасной спектроскопии. Данные ИК-спектрального анализа показали характерные для хитина полосы амида I (C=O-NHR) при 1640 $см^{-1}$, а для хитозана - полосы поглощения аминогруппы (-NH_2) при 1588 $см^{-1}$. Сигналы при 870 и 614 $см^{-1}$ и 1125-1388 $см^{-1}$ в СХС соответствуют связям C-O-S и S=O, что свидетельствует о включении в хитозан сульфатных групп. Степень замещения (СЗ), определенная гравиметрическим методом, соответствует 1,32. Молекулярные массы хитозана и СХС рассчитывали с использованием эксклюзионной хроматографии (ЭХ). Согласно расчету ЭХ для хитозана средняя молекулярная масса (ММ) соответствует 16 кДа, а для СХС - 1-3 кДа.

Ключевые слова: цисты Artemia parthenogenetica, хитин, хитозан, сульфатированный хитозан.