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Study of the Structure of Chitosan Succinate *Bombyx mori*

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Abstract: The first time water solubility derivates of chitosan *Bombyx mori* was obtain. By FTIR method it was established the fact of formation of chitosan succinate. Influence of synthesis condition to structure of chitosan succinate was shown. It is revealed that, gel formation of chitosan succinate depend on the formation of both polyelectrolytic complex and covalent bonds.

Keywords: Chitosan *Bombyx mori*, Succinate Chitosan, Sodium-Succinate, Succinate Anhydride

1. Introduction

Chitosan (Ch) possess own biological activity, due to which it is used in various branches of the national economy. It is known that Ch, being polyelectrolyte, soluble in acidic media. However, special attention should be paid to obtaining water-soluble derivatives of Ch and their application in various field of the national economy, which showed scientific and practical interest. The presence of functional groups in structure of Ch provides the possibility of obtaining different modifications. In this case, at last years, interest to synthesis of water-soluble derivatives of chitosan, in particular, succinate chitosan (ChS) and its application in agro-industrial complex, medicine, veterinary medicine, cosmetics and other industries are increased [1-2]. Earlier, we were obtained samples of *Bombyx mori* ChS and were studied the effect of synthesis conditions on the composition of desired product [3].

In this paper we were obtained discussed results of FTIR spectroscopy of *Bombyx mori.* Chitosan is used in various pharmaceutical formulations, both as diluent in direct compression processes [4] and as vehicle in sustained release systems [5].

Further, its chemical features as a poly aminoglycoside allow its physic chemical properties to be modulated by covalent links to different residues. The conjugation of chitosan to various medicinal agents is also facilitated by its nature as amino sugar polymer [6]. Chitosan is the deacetylated product of chitin, the second most abundant natural polysaccharide next to cellulose [7]. Chitosan is non-toxic, biodegradable, biocompatible and novel natural resource material with antibacterial and antitumor activities, also easily modified owing to the -OH and NH_2 positions [8]. N-succinyl-chitosan (NSC), synthesized via introduction of succinyl groups at the N-position of the glucosamine unit of chitosan, which water-soluble derivative with the same bioactivity of chitosan. Many reports focused their attention on the application of NSC as a carrier for protein, peptide and gene in cancer therapy [9] [10].

Chitosan succinate (ChS) is an anionic chitosan derivative prepared in our laboratory through the acylation of the chitosan's amino group using succinic anhydride [11, 12, 13]. At th and the ChS has a completely different solubility profile and enteric dissolution properties [11]. It has been recently used by another research group in the preparation of microspheres for improving oral bioavailability of insulin [14].

2. Experimental Part

2.1. Synthesis

For the synthesis, of ChS was used chitosan *Bombyx mori* with a molecular weight of 60,000 a degree of deacetylation

80%, N_{total}=7.88%. Succinate anhydride (CA) was obtained by dehydration of succinic acid at temperature 300°C [15]. To neutralize the protonated amino groups of Ch, the pH of the solution was adjusted by adding 0.1N NaOH. The interaction of chitosan with powdered succinate anhydride was carried out in an aqueous-alkaline solution, and the desired product was isolated by centrifugation, followed by drying in freeze drying (Table 1).

Table 1. *Effect of synthesis conditions on solubility of chitosan succinate ChS: CA=5.28 wt. respectively; the pH of the solution is Ch=7.1; τ synthesis=30 min; t=13-15°C.*

#	Samples	[Ch], mol/l	pH of solution Ch	Output,%	Water solubility,%
1	ChS-1	2.80	8.5	96.1	gel
2	ChS-2	3.10	8.5	89.7	gel
3	ChS-3	2.10	9	96.7	55

2.2. IR-FTIR Spectra of Chitosan Samples and Chitosan Succinate

The results of experiment has shown that, under the selected synthesis conditions in the samples ChS-1 and ChS-2, unlike the ChS-3 sample, were formed, the gel which possibly related to the intermolecular cross-linking of the macromolecules through the amino and hydroxyl groups of chitosan. Perhaps the titration of the ChS solution with NaOH to pH=9.0 (Table 1, Example #3) promotes the formation of the sodium salt of succinate ChS with a water solubility about 55%. Note that the yield of the target product in all cases is ≥90%. The structure of obtained samples of chitosan succinate was studied by FTIR spectroscopy (Figure 1-3).

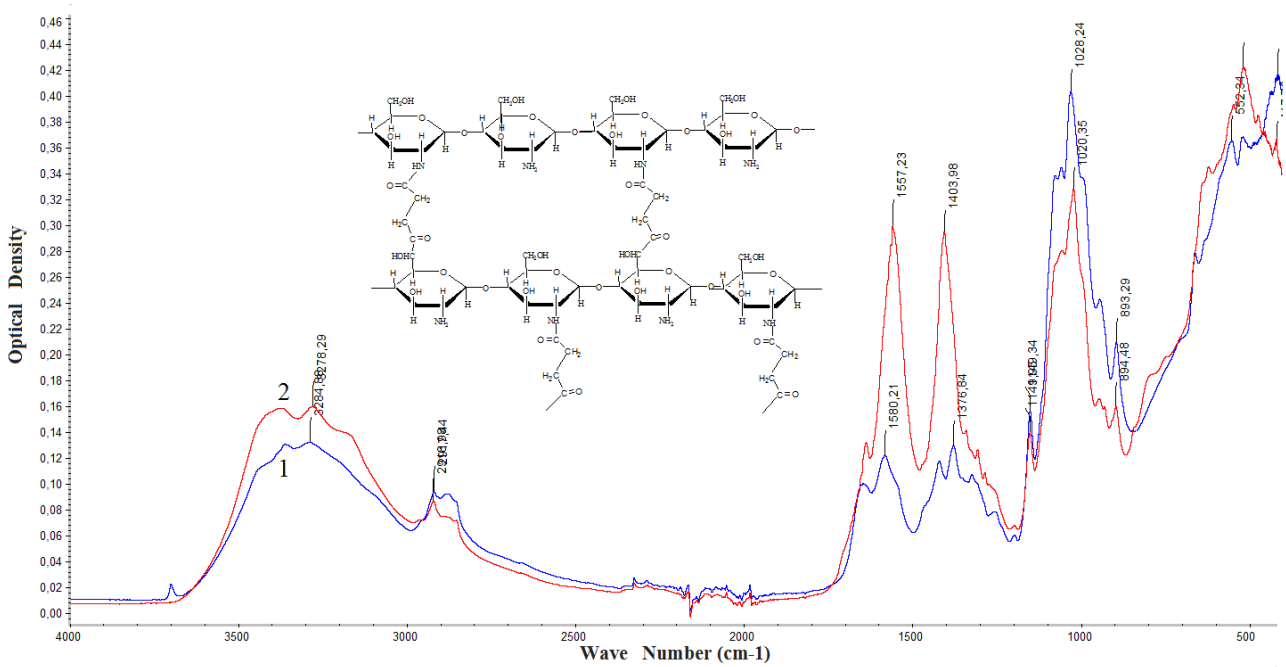

Figure 1. *IR-FTIR spectra of chitosan samples (1) and chitosan succinate-1 (2).*

You can sec from figure 1, in FTIR spectra of chitosan (Figure 1, curve -1) a broad band is observed in region 3400-3200 cm^{-1}, which refers to the stretching vibrations of -OH and -NH_2 groups, as well as at 2900-2800 cm^{-1}, vibrations of -CH groups are manifested. In the region 1650-1540 cm^{-1}, a deformation vibration of -NH_2 is observed, and in the region of 1400-1300 cm^{-1} -CH and -OH groups and at 1150-1070 cm^{-1}, the stretching vibration of the -C-O-C ether bonds is manifested according literature data its agreed [16]. In the spectrum of succinate sample of chitosan #1 (Figure 1, curve 2), an increase in the intensity of bands of 1026 cm^{-1} is observed which belong to the -CO group, which suggests the formation of a bond between macromolecules in the presence of succinate anhydride, Cross-linked samples of succinate chitosan in the form of gels [19-21].

It should be noted that, the IR spectra of samples ChS-1 and ChS-2 are almost identical, i.e. in the selected synthesis conditions, partially cross-linked ChS samples are formed (Figure 2).

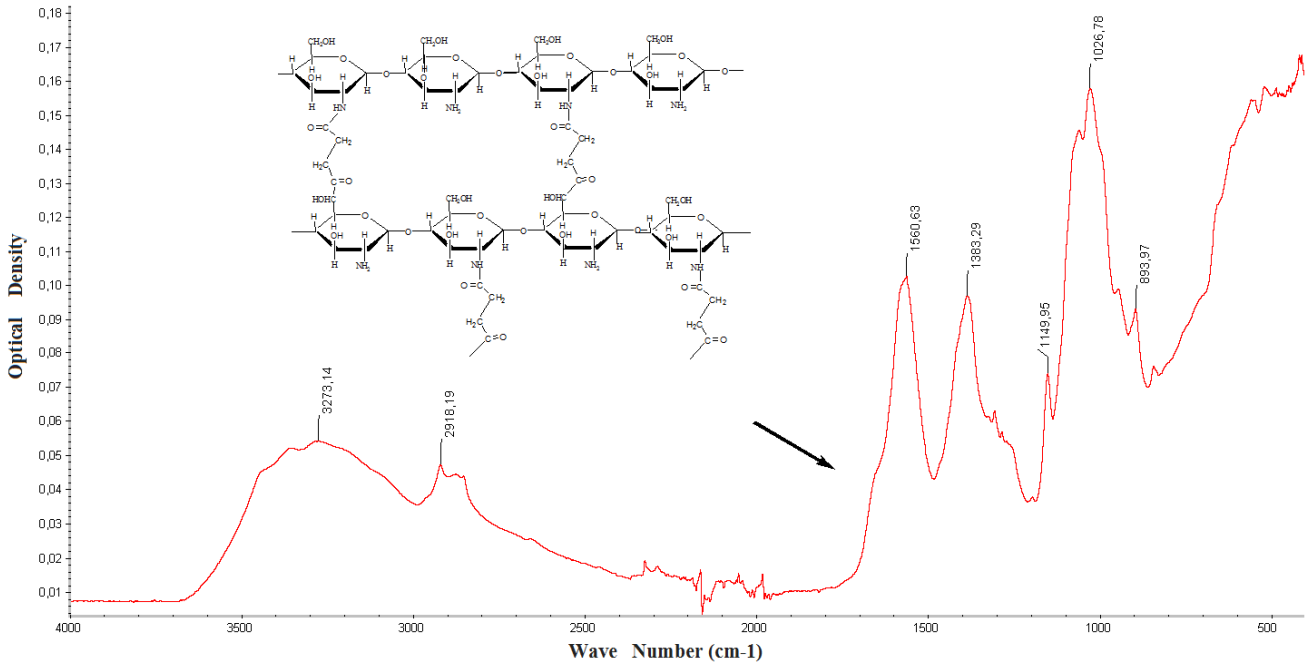

Figure 2. *IR-FTIR spectrum of chitosan succinate-2.*

However, a weak shoulder is observed in the ChS spectrum at 1030 cm^{-1}, which also indicates the presence of ChS sodium salt in the system [20].

An increase in the pH of the ChS solution to 9.0 contributes to the formation of the sodium salt of ChS in the powder form. In the IR spectrum of ChS-3, a doublet band is observed at 1720 and 1680 cm^{-1}, which refers to the -C=O group of carboxylic acids, and a band at 1636 and 1544 cm^{-1}, referring to Amide I and to Amide II (3).

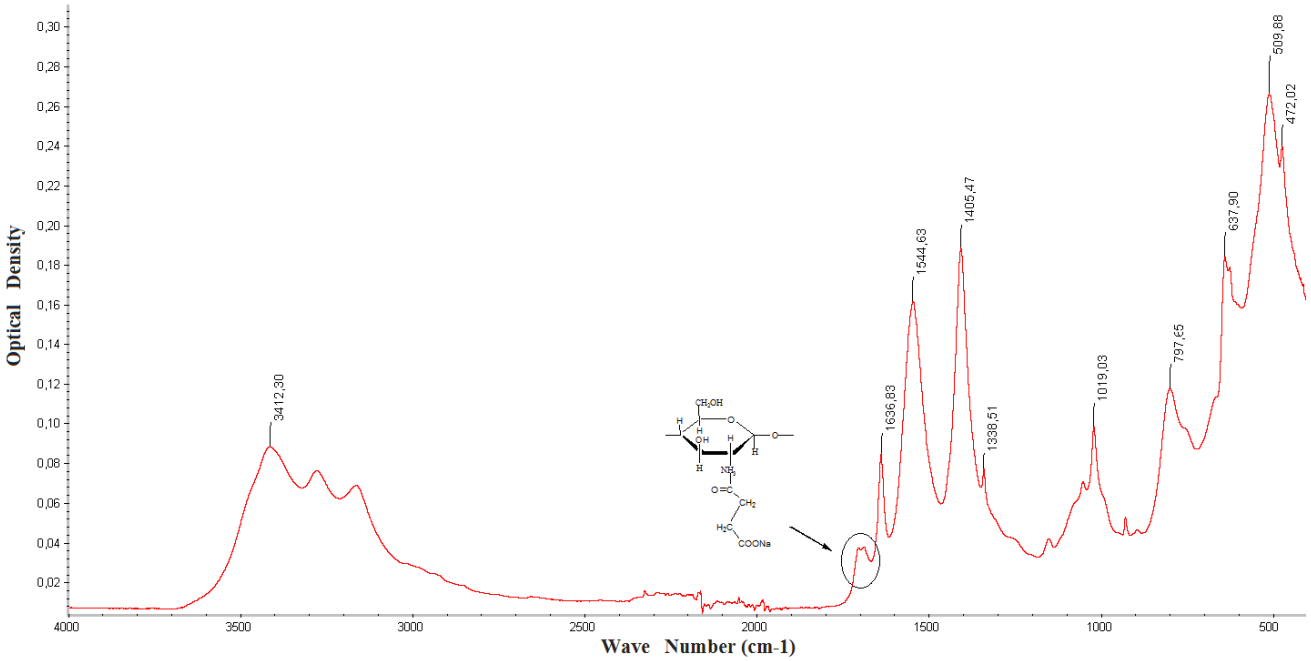

Figure 3. *IR-FTIR spectrum of chitosan succinate-3.*

At 1405 cm^{-1}, a strong narrow band is observed, related to the (scissor) deformation vibration of the $-CH_2$ group of the succinic acid $-CH_2-CO-$, i.e. In this sample, all the bands characteristic of succinate of chitosan are observed, which confirms the formation of chitosan sodium-succinate [16-17].

2.3. XRD Spectra of Samples of Chitosan and Chitosan Succinate

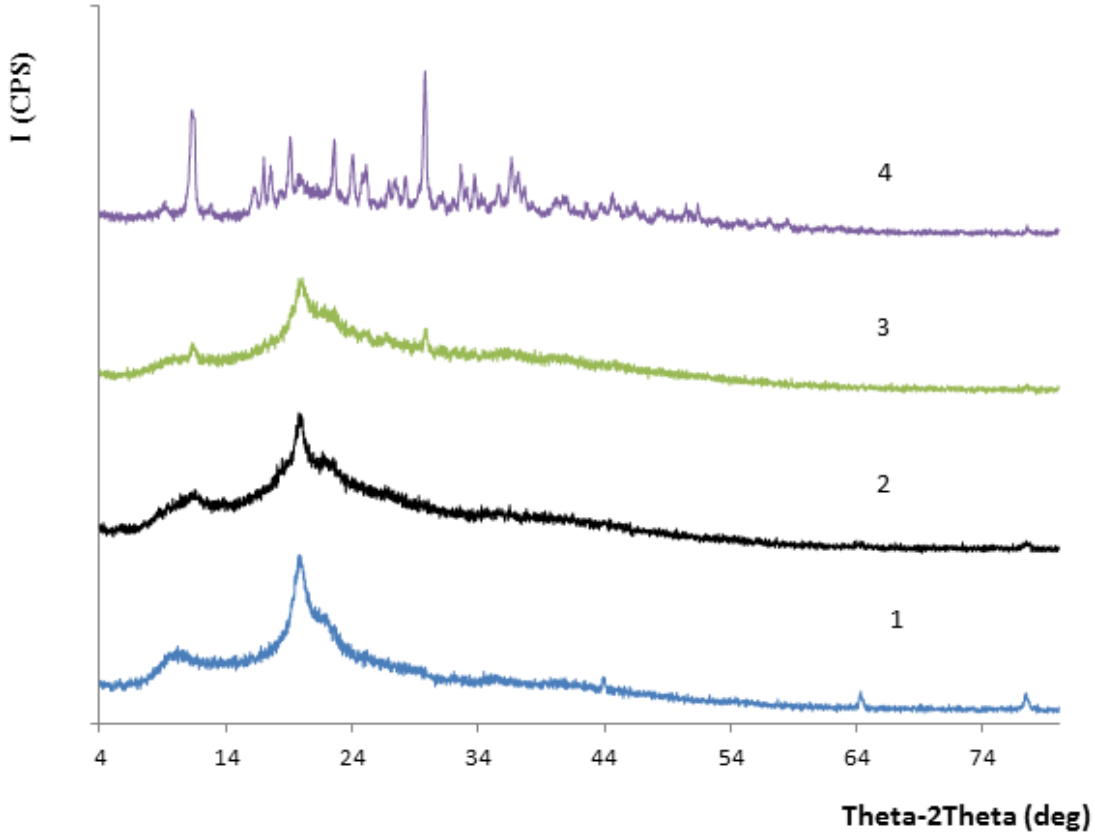

Figure 4. *XRD spectra of samples of chitosan (1) and chitosan succinate -1 (2) and chitosan succinate -2 (3) and chitosan succinate -3 (4).*

Comparative x-ray structural analyzes of chitosan and succinate chitosan samples. In the X-ray diffractogram Ch characteristic peaks appear at 2Ɵ 10 and 20°. The degree of crystallinity of Ch was calculated, which was 36%.

It should be noted that in the X-ray diffraction patterns of ChS-1 and ChS-2, characteristic Ch peaks are practically conserved, probably due to the production of the sodium salt of succinate Ch, where the degree of crystallinity increases to 37 and 45%, respectively.

Also, an increase in the pH of the medium to 9 in the reaction system during the production of ChS leads to the appearance of peaks at 2Ɵ 9-35°, which at 9 and 29° increases the intensity of the corresponding peaks. The degree of crystallinity of ChS-3 was 50%. Note that as the pH of the reaction system increases, the solubility of ChS samples increases.

3. Conclusion

Thus, the fact of formation of chitosan succinate was established by the IR FTIR spectroscopy method. Influence of the synthesis conditions on the composition of chitosan succinate has shown. It was found that, the formation of a gel of chitosan succinate occurs due to the formation of both a polyelectrolyte complex and covalent bonds. It was found that, when chitosan is modified with succinate anhydride, water-soluble samples of chitosan succinate can be obtained only at pH≥9.

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