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NITRO-CARBOXYMETHYLINULIN SYNTHESIS

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Abstract: This work presents data on the nitration of inulin ether with a nitrating mixture. The influence of the nitrating mixture concentration and the molar ratio of nitric acid on the composition of nitro-Na-CMI was studied. It has been established that changes in nitration parameters have a determining effect on the depth of chemical modification. The structure of the obtained inulin mixed ethers was proven by IR spectroscopy.

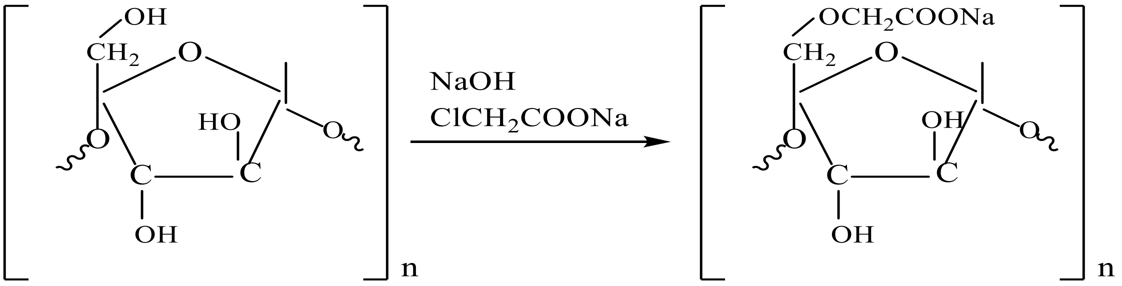
Keywords: polymers, inulin, carboxymethylinulin, nitration, nitro-Na-CMI.

Introduction. The physicochemical properties of polymer materials are a key factor determining their fields of practical application. Modern approaches to the development of functional polymers are based on establishing the relationship between the chemical structure of a macromolecule and the properties it exhibits [1–3]. At the same time, the set of polymer properties is determined not only by manifestations of their high-molecular nature (such as the type of macromolecular matrix, degree of polymerization, etc.), but also by the specific role of functional groups present within the monomer units. The number and uniformity of substituent distribution along the macromolecular chain are also of great importance. From this perspective, it can be assumed that purposeful regulation of physicochemical and technological properties may be achieved by varying the set of functional groups within the polymer composition. Such an approach makes it possible not only to modify polymer solubility and reactivity but also to impart new biologically significant functions.

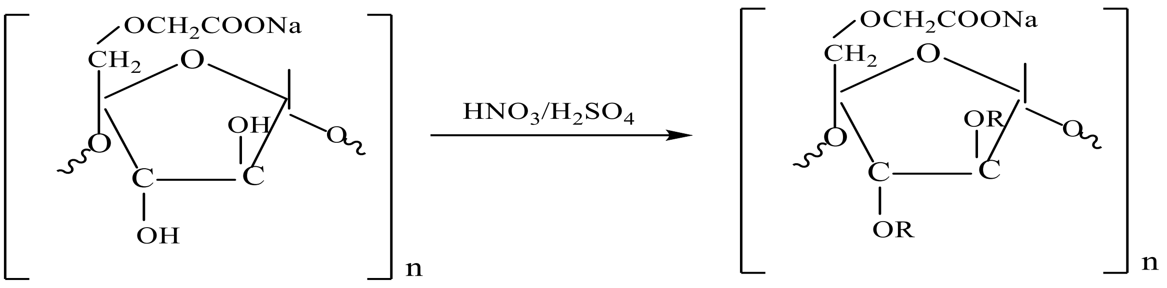
It is well known that acidic groups within polymer structures determine a wide range of biomedical activities, including wound-healing, antimicrobial, and hemostatic effects [4–7], which are characteristic, for example, of oxidized cellulose, pectin, alginic acid, and several other polysaccharides. High biocompatibility and biodegradability make such systems particularly promising for medical applications. At the same time, organic nitrites (compounds containing –O–N=O groups) and nitrates (containing –O–NO_2 groups) are widely used in medicine as agents that improve blood circulation and myocardial metabolism [8]. However, the pharmacological action of such drugs is generally short-lived and requires repeated administration. From the standpoint of prolonging the therapeutic effect, the covalent incorporation of nitrate groups into a polymer matrix appears to be a promising approach. A prolonged effect could be expected when using polysaccharide nitrates; however, their practical application is limited by their low solubility in biological fluids.

The present article reports the results of studying the synthesis conditions of mixed inulin esters, as well as an evaluation of the influence of their functional composition on certain physicochemical properties. Particular attention is given to the relationship between the chemical nature of substituents and the solubility of the obtained derivatives [9-10]. The selection of inulin as an object of chemical modification is обусловed by its availability, oligomeric nature, and a wide range of biomedical properties. This biopolymer does not represent an individual substance but rather a mixture of two structurally different monosaccharides—fructose and glucose. These structural features of inulin provide a scientific basis for targeted chemical modification and the development of functional materials.

Experimental Section. Synthesis of the Sodium Salt of Carboxymethyl Inulin (Na-CMI). Carboxymethylation of inulin was carried out in an isopropyl alcohol (IPA) medium. For this purpose, finely powdered inulin was placed in a three-necked flask equipped with a reflux condenser, mechanical stirrer, and thermometer, and dispersed in the IPA medium for 2 hours. Next, a 10% alkali solution was added to the suspension, and the mixture was stirred for 1 hour. Carboxymethylation of mercerized inulin was performed using sodium monochloroacetate (Na-MCA) by adding the alkylating reagent at an inulin:Na-MCA ratio of 1:3, at a temperature of 70 °C for 1.5 hours. The sample was purified by extraction with 96% ethanol and finally dried at 50–60 °C. The degree of substitution of the synthesized Na-CMI was 30 mol% [11-14]. The general scheme of Na-CMI synthesis:



Nitration of Na-CMI. A 1-liter three-necked flask equipped with a mechanical stirrer and a reflux condenser was charged with 300 mL of fuming nitric acid, after which the flask was placed in a thermostat maintained at 0–3 °C. The samples were treated with a mixture of concentrated H_2SO_4 and HNO_3. The nitration duration ranged from 30 to 180 minutes. After nitration, the nitro-CMI was separated from the nitrating mixture, washed with distilled water, stabilized with a 2% $NaHCO_3$ solution, and rinsed with hot water. The nitro-Na-CMI samples were then dried to constant weight, and the yield and degree of substitution were determined. The general scheme of nitration and preparation of nitro-Na-CMI:



R = -NO_2

FTIR Spectra. The FTIR spectra of the samples were recorded on a Vector-22 spectrometer in the wavelength range of 400–4000 cm^{-1} using KBr pellets (3 mg of sample per 300 mg of KBr). The pellets were pressed under a pressure of 8–10 t for 1–2 minutes to obtain transparent disks.

Determination of Nitrogen Content. The nitrogen content in the synthesized inulin derivative was determined by the Kjeldahl method [15].

Results and Discussion. The data in Table 1 show that the composition of the nitrating mixture and the duration of nitration significantly affect the degree of substitution (DS) of the reaction products. When using a mixture with an increased water content (10%) and a relatively short reaction time (30 min), minimal values of nitrogen content and DS are observed, indicating limited nitration efficiency in a diluted medium. Increasing the HNO_3 content while simultaneously reducing the water content to 5% leads to a noticeable increase in nitrogen content and DS, which is associated with enhanced nitrating ability of the system. However, extending the nitration time to 180 min results in a sharp rise in nitrogen content and DS, indicating the involvement of additional reactive centers. It should be noted that the concentration of sulfuric acid plays a key role not only as a dehydrating agent but also as a stabilizing factor for the nitrating system. Overall, the results indicate that the combination of a highly concentrated nitrating mixture and prolonged reaction time ensures the maximum degree of substitution.

The results presented in Table 2 indicate a pronounced dependence of the nitration degree of Na-CMI on the molar ratio. At low acid ratios (1:1–1:3), a gradual increase in nitrogen content and degree of substitution (DS) is observed, indicating a limited amount of nitrating agent in the reaction system.

Increasing the proportion of HNO_3 to ratios of 1:4–1:5 leads to a more pronounced increase in the degree of substitution (DS), which is associated with a higher probability of interaction between the nitrating species and the functional groups of inulin ether. Under these conditions, the reaction predominantly occurs at the most accessible and reactive centers of the macromolecule. It is likely that the excess HNO_3 facilitates deeper penetration of the reagent into the supramolecular structure, involving previously inaccessible regions of the polymer chain. Further increasing the ratio up to 1:30 continues to enhance these parameters.

The FTIR spectrum of inulin (1) is characterized by typical polysaccharide bands (Fig. 1). A broad, intense band in the region of 3218–3580 cm^{-1} corresponds to the stretching vibrations of hydroxyl groups. The bands at 2910–2850 cm^{-1} are attributed to the stretching vibrations of C–H bonds in the carbohydrate backbone, while the intense set of bands in the region of 1200–1030 cm^{-1} is due to the vibrations of C–O and C–O–C bonds in the fructose units.

The FTIR spectrum of Na-CMI (2) shows significant changes, confirming the chemical modification of inulin. The appearance of a new intense band in the region of 1610–1645 cm^{-1} is associated with the asymmetric stretching vibrations of $-COO^-$ groups, while the band around 1415–1450 cm^{-1} corresponds to their symmetric stretching vibrations. At the same time, a decrease in the intensity of the O–H band is observed, indicating partial substitution of hydroxyl groups.

The FTIR spectrum of nitro-carboxymethyl inulin (3) retains the characteristic features of carboxyl groups but additionally exhibits new bands due to the introduction of nitro groups. Intense bands in the regions of 1520–1550 cm^{-1} and 1340–1380 cm^{-1} correspond to the asymmetric and symmetric stretching vibrations of $-NO_2$ groups. The enhancement and complexity of bands in the region of 1210–1160 cm^{-1} indicate changes in the electronic structure of the C–O bonds as a result of nitration.

Conclusions. In the present study, the features of Na-CMI nitration were investigated. The influence of the composition of the nitrating mixture and the molar ratio of nitric acid on the composition of the synthesized reaction products was established. The structure of the synthesized nitro-Na-CMI was confirmed by FTIR spectroscopy. The results obtained demonstrate the possibility of purposeful regulation of the degree of substitution and the properties of the final product by modifying the nitration conditions.

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Table 1. Influence of nitrating mixture concentration and reaction time on the composition of reaction products

Nitration time, min	Composition of the nitrating mixture, %			Nitrogen content, %	DS, mol%
	H_2SO_4	HNO_3	H_2O		
30	65	25	10	5,7	1,00
90	60	35	5	6,8	1,25
90	70	30	-	7,6	1,48
120	70	30	-	7,8	1,56
180	70	30	-	9,7	1,90

Table 2. Influence of the molar ratio of nitric acid on the composition of reaction products

Molar ratio of Na-KMI:HNO_3	Nitrogen content, %	DS, mol%
1:1	1,3	0,25
1:2	1,5	0,33
1:3	1,8	0,47
1:4	2,0	0,52
1:5	2,5	0,60
1:10	8,1	1,64
1:30	9,4	1,95

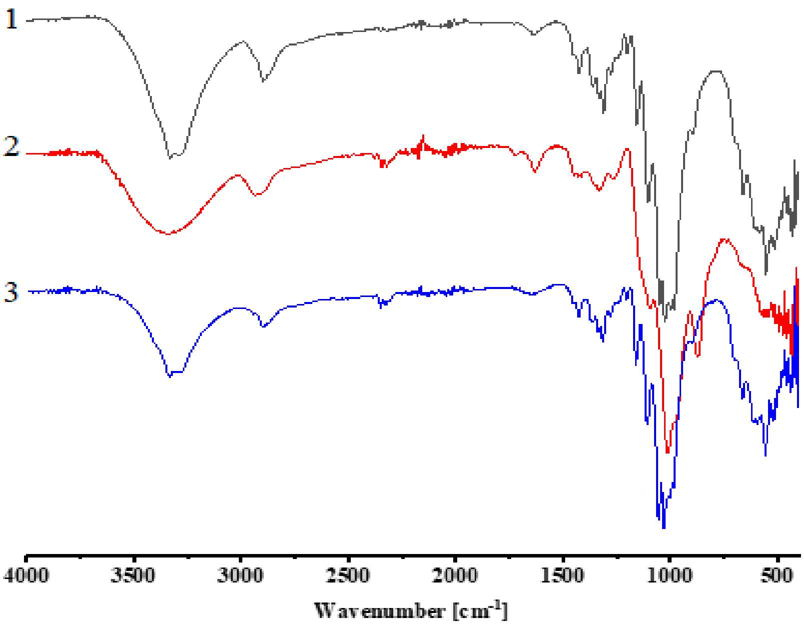

Fig.1. IR spectra of inulin (1), Na-KMI (2) and nitro-Na-KMI (3)

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