PRODUCTION AND DESCRIPTION THE CHARACTERIZATION OF GUAR GUM GALACTOMANNAN BUTYL ETHER

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ABSTRACT
Guar gum galactomannan is a common polysaccharide isolated from the legume plants Cyamopsis tetraganoloba. Guar gum derivatives are widely used in many industrial fields. In this work, a method for obtaining a new derivative of guar gum, galactomannan butyl ether, is considered. The introduction of the butyl group into the structure of galactomannan was proved by IR spectroscopy, by the appearance of absorption bands in the regions corresponding to the vibrations of the $-C - O - C-$, CH_2 and CH_3 groups. According to X-ray diffraction data, it was shown that in the process of obtaining butyl ether of galactomannan, the structure is amorphous. According to the data of scanning electron microscopy, the powder of galactomannan butyl ether has a porous morphology. The pore sizes in the particles of galactomannan butyl ether range from 70 to 300 µm. The obtained galactomannan butyl ether can find application in medicine - as a filler for tablets, in construction - as an additive for adhesives and in other fields.
Key words: galactomannan, buthyl ester, chemical modification, medical plants.

الإنتاج والوصف توصيف صمغ الغار إستر جالاكتومانان بوتيل

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المستخلص

صمغ الغار غالاكتومانان هو عديد السكاريد الشائع المعزول من نباتات البقوليات. Cyamopsis tetraganoloba تستخدم مشتقات صمغ الغوار على نطاق واسع في العديد من المجالات الصناعية. في هذا العمل ، تم النظر في طريقة للحصول على مشتق جديد من صمغ الغوار ، غالاكتومانان بوتيل استر. تم إثبات إدخال مجموعة البوتيل في بنية الجالاكتومانان عن طريق التحليل الطيفي للأشعة تحت الحمراء ، من خلال ظهور نطاقات الامتصاص في المناطق المقابلة لاهتزازات مجموعات $-C-$ $C - O$و CH_2 و CH_3. وفقًا لبيانات حيود الأشعة السينية ، فقد تبين أنه في عملية الحصول على بوتيل إيثر من غالاكتومانان ، يكون الهيكل غير متبلور. وفقًا لبيانات المسح المجهري الإلكتروني ، فإن مسحوق غالاكتومانان بوتيل استر له شكل مسامي. تتراوح أحجام المسام في جزيئات غالاكتومانان بوتيل استر من 70 إلى 300 ميكرومتر. يمكن العثور على غالاكتومانان بوتيل استر الذي تم الحصول عليه في الطب – كمواد مالئة للأقراص ، في البناء – كمادة مضافة للمواد اللاصقة وفي مجالات أخرى.

الكلمات المفتاحية: غالاكتومانان ، بوتيل استر ، تعديل كيميائي ، نباتات طبية.

INTRODUCTION

Polysaccharides from various sources are ideal natural resources for obtaining many valuable substances used in food, pharmaceutical, medical and many other fields (6,30). Chemical modification of polysaccharides makes it possible to obtain a wide range of useful products with various structural, physical, chemical and biologically active properties. There is a growing body of research on modified polysaccharides, most of which have ideal properties for function groups with pharmacetical effects (30,19). Alkyl derivatives of polysaccharides have found wide application in many areas: in medicine - obtaining capsules for drugs, various lubricants and fillers; in the food industry - obtaining thikness and emulsifiers, as food additives; in construction - obtaining tile and wallpaper adhesives; in biology - for the replication of viruses and slowing down the motility of bacterial and protozoal cells; in industry - as a sizing in the production of paper and textiles (21,28). Galactomannans - polysaccharides consisting of galactose and mannose are widely used in the food, textile, pharmaceutical industries as stabilizers, food additives, flocculants and thickeners (8,31). Pharmacological studies have shown that galactomannans and their derivatives exhibit hepatoprotective and analgesic properties (14,11). Guar gum (GG) is a galactomannan, which is a carbohydrate polymer that is used as a thickening agent and absorption reagent (11,25). GG and its derivatives are used in explosives, food, cosmetics, pharmaceuticals, petroleum, mining, paper and textile industries, mainly as a binder for water (18,,25). The aim of this study was to obtain new butyl esters of galactomannan guar gum and to study them using FTIR spectroscopy, X-ray phase analysis, scanning electron microscopy, and thermal analysis.

MATERIAIS AND METHODS

In this work, guar gum galactomannan (Sigma-Aldrich) was used.

Synthesis of galactomannan butyl ether. The synthesis of galactomannan butyl ether was carried out in a round-bottom flask equipped with a stirrer at room temperature (25 ° C). For this, 2 g of galactomannan was added to 50 ml of bromobutane with constant stirring, the pH

was adjusted to neutral with 10% sodium hydroxide solution. The reaction was carried out for 1 and 24 hours. After carrying out the synthesis reaction of galactomannan butyl ether, the product was dried into 60˚C oven to constant weight. Then the dried product was ground to fine particles in a porcelain mortar and washed with ethanol on a filter paper (to remove residues of bromobutane and sodium hydroxide). Then, galactomannan butyl ether was dried in air to constant weight (Fig. 1). Experiment scheme is shown in Fig. 2.
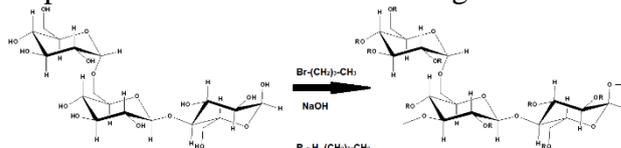

Figure 1. Scheme of obtaining butyl ether of galactomannan (on the example of the elementary chain of galactomannan)
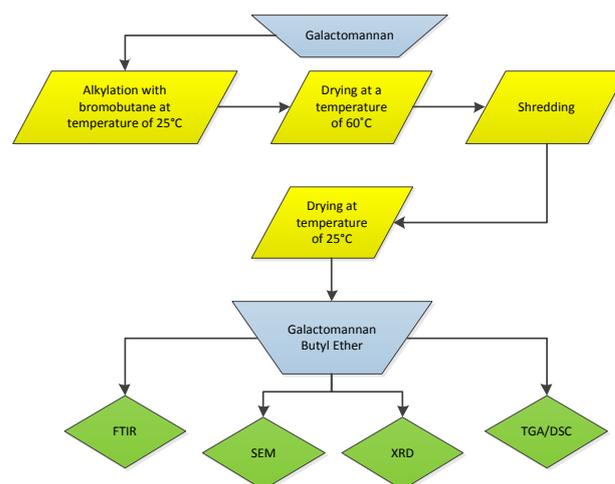

Figure 2. Schematic of the experiment for obtaining butyl ether of galactomannan

Study of galactomannan butyl ether by physical and chemical methods

Fourier transform infrared spectroscopy (FTIR). The FTIR spectra of initial galactomannan and galactomannan butyl ether were recorded using a Shimadzu IR Tracer-100 spectrometer (Japan) within the wavelength range of 400–4000cm^{-1}. The spectral information was analysed using the OPUS program (version 5.0). Solid samples for analysis were prepared in the form of pills in a KBr matrix (2 mg sample/1000 mg KBr).

X-ray diffraction (XRD). The X-ray diffraction phase analysis was carried out on a DRON-3 X-ray diffractometer using CuKα

monochromatized radiation (λ = 0.154 nm), voltage 30 kV, current 25 mA. The scanning step is 0.02 deg; intervals for 1 s per data point. The measurement was carried out in the interval of the Bragg angles 2Θ from 5.00 to 70.00 Θ.

Scanning electron microscopy (SEM). Electron micrographs were obtained on a Hitachi TM-1000 scanning electron microscope (Japan) at an accelerating voltage of 15 kV and a magnification from 100 to 10000× with a resolution of 30 nm. The electron micrographs were processed using the ImageJ software (version 1.8.0_112).

Thermogravimetric analysis (TGA). The thermogravimetric study and data analysis were performed using a NETZSCH STA 449 F1 Jupiter simultaneous thermal analysis instrument (Germany). The thermal degradation of the samples was analyzed in argon in the temperature range from 30 to 600°C; the protective and purge gas flow rates were 20 and 50 ml/min, respectively. The samples were heated in a dynamic temperature regime (10° C/min) in corundum crucibles. The measurement results were processed using the NETZSCH Proteus–Thermal Analysis.5.1.0 software supplied with the instrument.

RESUITS AND DISCUSSION

Synthesis of galactomannan butyl ether

In order to calculate the degree of substitution in the obtained products, the theoretical ratios (wt%) of C, H, O elements were calculated (Table 1). An elementary unit of galactomannan, consisting of 1 unit of galactose and 2 units of mannose, was taken as a model for the calculation. The effect of the duration of the process of modifying galactomannan with butyl groups on the elemental composition of the resulting products are shown in Table 2. Comparison of the experimental and theoretical values of the elemental composition was carried out in order to calculate the degree of substitution in the obtained samples of galactomannan butyl ether. According to the elemental analysis data (Table 2), with an increase in the duration of the reaction for producing galactomannan butyl ether, the degree of substitution increases, reaching 34.0%. This is indicated by an increase in the content of carbon and hydrogen and a regular decrease in the content of oxygen in the reaction product. It should be noted that an increase in the duration of the reaction for producing galactomannan butyl ether does not lead to an increase in the content of carbon and hydrogen, and, therefore, in the degree of substitution. This phenomenon is probably associated with steric hindrances and diffusion of reagents to reactive groups. For further analyzes, we used galactomannan butyl ether with the maximum degree of substitution.

Table 1. Theoretical values of the influence of the degree of substitution on the elemental composition of galactomannan butyl ether

Number of butyl groups	Elemental composition, wt. %			Substitution rate,%
	C	H	O	
0	43.5	4.8	51.6	0
1	47.7	6	46.3	11.1
2	51.1	6.9	42	22.2
3	54.0	7.6	38.4	33.3
4	56.4	8.3	35.4	44.4
5	58.4	8.8	32.8	55.5
6	60.1	9.3	30.5	66.7
7	61.7	9.7	28.6	77.8
8	63.0	10.1	26.9	88.9
9	64.2	10.4	25.4	100

Table 2. Experimental values of the elemental composition of galactomannan butyl ether

| Duration, h	Elemental composition, wt. %			Substitution rate,%
	C	H	O	
0	42.7	4.7	51.7	0
1	45.9	5.7	47.5	10.7
24	54.8	7.9	36.7	34.0

FTIR spectroscopy. Galactomannan and its butyl ether were analyzed by FTIR spectroscopy (Fig. 3).
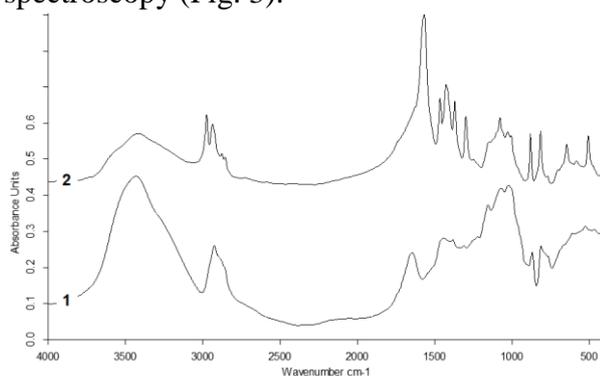
**Figure 3. FTIR spectra: 1 - galactomannan, 2 - galactomannan butyl ether**

The FTIR spectrum of the initial galactomannan contains a broad absorption band in the region of 3700-3000 cm^{-1}, which corresponds to the stretching vibrations of the OH group. The absorption band in the region of 2900-2800 cm^{-1} corresponds to the vibrations of CH_2 groups. In the FTIR spectra of butyl ether of galactomannan, a decrease in the intensity of the absorption band of vibrations of OH groups and an increase in the intensity of the absorption band of vibrations of CH_2 and CH_3 - groups are observed in comparison with the initial galactomannan. Thus, the IR spectra of galactomannan butyl ether contain absorption bands at: 2973 cm^{-1} (υas), 2875 cm^{-1} (υ_s), 1463 cm^{-1} (δ_{as}), 1368 cm^{-1} (δ_s), which correspond to the vibrations of CH_3 -groups; 2935 cm^{-1} (υ_{as}), 2854 cm^{-1} (υ_s), 1426 cm^{-1} (δ), corresponding to vibrations of CH_2-groups. In addition, the IR spectrum of galactomannan butyl ether contains absorption bands at 1077, 879, 815 and 796 cm^{-1}, corresponding to the vibrations of the ether bond of the -C-O-C- groups. Thus, the introduction of a butyl group into the structure of galactomannan has been proven by IR spectroscopy.

Scanning electron microscopy (SEM). The powders of the original and modified galactomannan were studied by the SEM method (Fig.4,5).
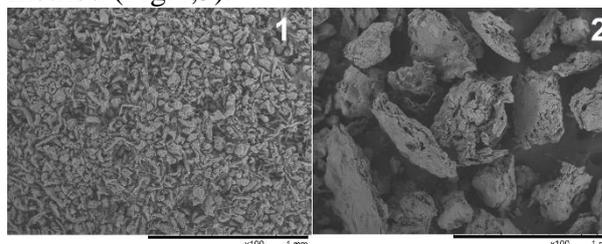
Figure 4. SEM images (x100): 1 - galactomannan, 2 - galactomannan butyl ether

According to the data of scanning electron microscopy, the initial sample of galactomannan consists of rough asymmetric particles with an average size of 200 to 800 μm (Fig. 4 (15) (12). After the introduction of a butyl group into the galactomannan molecule, its amorphous changes (Fig. 4 (2)). Galactomannan butyl ether consists of particles of various shapes, 1000–10000 microns in size.
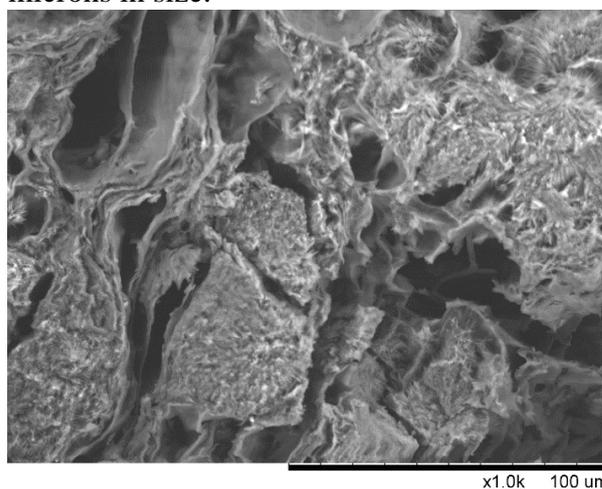
Figure 5. SEM image of galactomannan butyl ether (x1000)

According to scanning electron microscopy, galactomannan butyl ether powder is composed of porous particles (Fig. 5). The pore sizes in the particles of galactomannan butyl ether range from 70 to 300 μm.
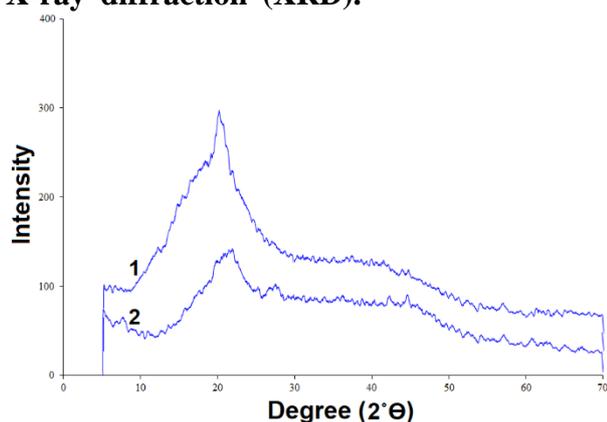
X-ray diffraction (XRD).

Figure 6. XRD diffractogramm: 1 - galactomannan, 2 - galactomannan butyl ether

Galactomannan samples have an amorphous structure (8, 15). Comparison of X-ray diffraction patterns of samples of galactomannan and galactomannan butyl ether showed (Fig. 6) that further amorphization of the material structure occurs during the addition of the butyl group to galactomannan. On the X-ray diffraction pattern of a sample of butyl ether of galactomannan, the smoothing of the peak was observed in the angle range 2 ° from 12 to 30 °. The initial galactomannan has a crystallinity index of 0.34 (12). Galactomannan butyl ether has a crystallinity index of 0.19. Thus, the introduction of a butyl group into the galactomannan structure increases amorphization by 1.8 times. The results obtained are in agreement with the literature. Earlier it was shown (6, 33, 23) that chemical modification of galactomannan leads to its amorphization.

Thermal analysis. Galactomannan and its butyl ester were analyzed by thermal analysis (Fig.7).
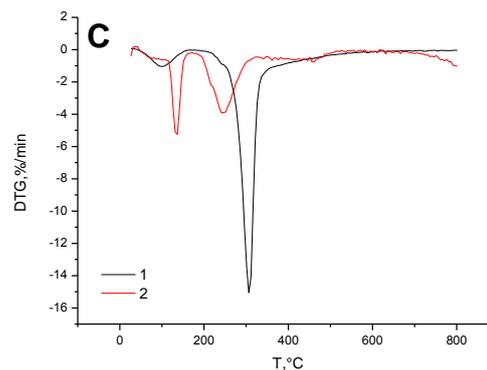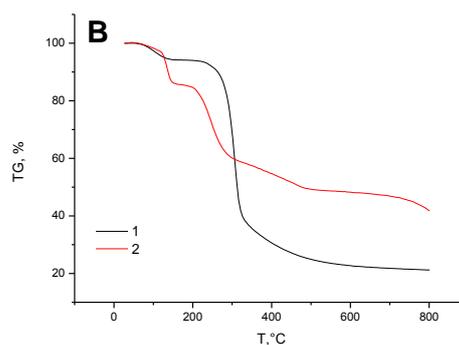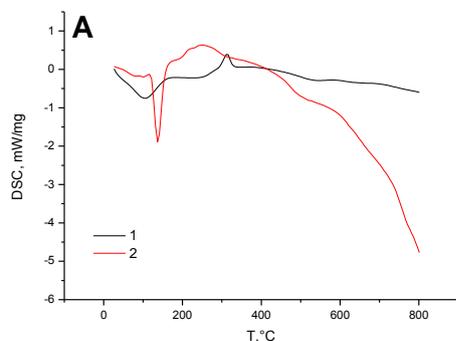

Figure 7. Thermal analysis data: A - Differential scanning calorimetry; B - Thermogravimetric analysis; C - Differential thermogravimetric analysis; (1 - original galactomannan, 2 - galactomannan butyl ether).

It is known that the shape of the TG and DTG curves depends on the heterogeneous nature of thermal decomposition, the occurrence of various chemical reactions, destruction of hydrogen bonds, and conformational and phase transitions (26). Data of thermogravimetric analysis (TGA) (Fig. 7B) shows the weight loss of the samples up to a temperature of 600 ° C occurs in several stages. The decrease in the mass of the initial galactomannan in the range from 30 to 180 ° C is associated with the desorption of moisture from the sample surface and from the bulk, as a result of the rupture of hydrogen bonds between water molecules and polar functional groups. On the DSC curve (Fig. 7A), this process corresponds to a broad endothermic peak. DSC analysis of a sample of galactomannan butyl ether shows moisture loss up to 116.5 ° C (slight sloping endothermic effect). Upon further heating, an intense narrow endopic appears with a maximum at 137 ° C, which is probably

associated with the elimination of the butyl group from the galactomannan molecule. This assumption is confirmed by the results of TG / DTG analysis: in the temperature range 120 - 152 ° C, a distinct "step" is observed on the TG curve, with a weight loss of about 10%, and DTG analysis indicates the maximum rate of weight loss (-5% / min) at 137 ° C. At a temperature of about 250 ° C (Fig. 7C), the stage of the main decomposition of the galactomannan structure begins, with an extremum at 306 ° C, which is characterized by the maximum decomposition rate (-15% / min). On the DTG curve of the butyl ether sample, the peak of the main decomposition is less intense (-4% / min) and shifts towards lower temperatures (242 ° C). From these data, it follows that the sample of galactomannan butyl ether is less heat resistant than the sample of the original galactomannan. The decomposition stage ends at a temperature of 500-550 ° C, with the formation of a coke residue.

Kinetic analysis of thermal decomposition of samples. The data of thermogravimetric analysis were used to calculate the activation energy (E_a) of the main period of thermal destruction of samples (the range of the degree of conversion of the substance α from 0.1 to 0.9) using the Coats - Redfern kinetic model.

The Coats - Redfern non-isoconversion integral method is widely used to characterize the kinetics of nonisothermal processes of polymer thermolysis. It is based on the following equation:

$$\ln\left[-\frac{\ln(1-\alpha)}{T^2}\right] = -\left(\frac{E}{RT}\right) + \left(\frac{AR}{\beta E}\right)\left(1 - \frac{2RT}{E}\right)$$

(at $n = 1$),   (1)

were α – conversion, β – heating rate, T – temperature, n – reaction order, R – universal gas constant, A – pre-exponential factor, E - energy. On the tangent of the slope of a straight line $\ln\left[-\frac{\ln(1-\alpha)}{T^2}\right]$ from 1/T find the activation energy of the process.

The activation energy calculated by this method is called effective. The value of the effective activation energy of thermal decomposition is one of the most important indicators that characterizes the resistance of natural and synthetic polymers to high temperatures. The activation energy is also used to study the mechanisms of thermal destruction, aging and stabilization of polymer materials.

When linearizing the experimental TG data (Fig. 8) using the Coats-Redfern method, the kinetic curves of thermal decomposition of the samples were plotted in the coordinates $\ln\left[-\frac{\ln(1-\alpha)}{T^2}\right] = f(1/T)$. In fig. 8 shows examples of graphical determination of the effective activation energy for the studied samples.

Plotting kinetic curves in coordinates $\ln\left[-\frac{\ln(1-\alpha)}{T^2}\right] = f(1/T)$ made it possible to determine the activation energy of the process of thermal decomposition of samples at these stages, since the tangent of the slope of these dependences corresponds to the value ($-E_a/R$).
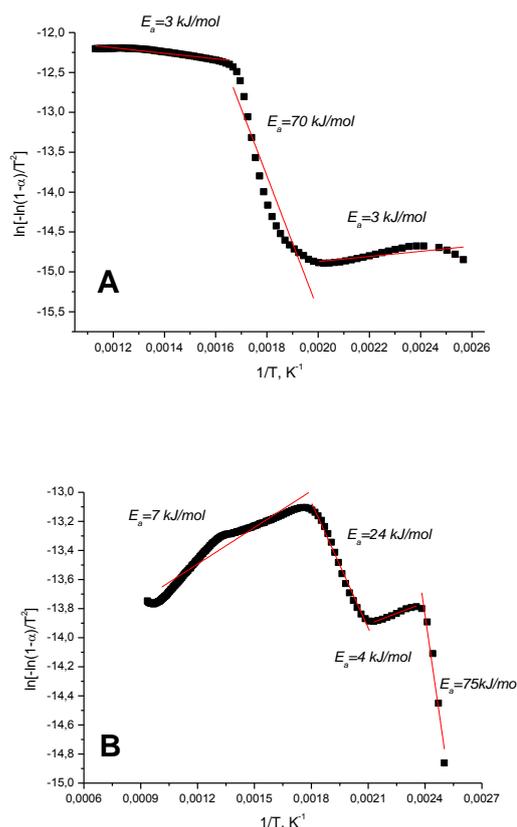

Figure 8. Linearization of experimental thermogravimetry data by the Coats-Redfern method (A - initial galactomannan, B - galactomannan butyl ether).

The process of thermal destruction of a galactomannan sample (Fig.8A) can be divided into three linear temperature ranges, these temperature ranges correspond to the

stages of the loss of adsorption water by the sample and rupture of hydrogen bonds, the maximum rate of thermal destruction, and the stage of formation of a coked residue. The activation energies of these stages Ea are 3, 70, and 3 kJ / mol, respectively.

The kinetic curve of thermal decomposition of galactomannan butyl ether (Figure 8B) has a more complex shape. With the onset of thermolysis and up to 146 ° C, the formation of products with maximum activation energy (75 kJ / mol) occurs. Then, through a plateau (151-191 ° C), the process of the main decomposition of the ether structure begins to a temperature of 281 ° C (24 kJ / mol). The end of thermolysis (formation of coke residue) is characterized by a low activation energy (7 kJ / mol).

CONCLUSION

For the first time, galactomannan butyl ether was synthesized by treatment with galactomannan bromobutane using sodium hydroxide as a catalyst. The obtained butyl galactomannan ester with the maximum degree of substitution was investigated by IR spectroscopy, X-ray phase analysis, scanning electron microscopy, and thermal analysis. The introduction of a butyl group into the structure of galactomannan was proved by FTIR spectroscopy. In the FTIR spectra of galactomannan butyl ether, the intensity of absorption bands increases at 2973 cm^{-1}, 2875 cm^{-1}, 1463 cm^{-1}, 1368 cm^{-1}, which correspond to the vibrations of CH_3-groups; 2935 cm^{-1}, 2854 cm^{-1}, 1426 cm^{-1}, corresponding to the vibrations of CH_2 groups. In addition, absorption bands appear at 1077, 879, 815 and 796 cm^{-1}, corresponding to the vibrations of the ether bond of the -C-O-C- groups, with a decrease in the intensity of the absorption band in the region of 3100-3700 cm^{-1}, corresponding to the vibrations of OH- groups. According to XRD data, the introduction of a butyl group into a galactomannan molecule leads to a greater amorphization of its structure and a decrease in the degree of crystallinity from 0.34 to 0.19.

CONFLICT OF INTERESTS :The authors declare that they have no conflicts of interest.

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