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M.V. Nikolenko [a], Yu. Sakhno [b], V.D. Myrhorodska-Terentieva [a], D.S. Hrechka [a], O.Yu. Vashkevych [a]

SEARCHING FOR THE CHEMICAL COMPONENT OF THE STARCH DEHYDRATION PROCESS

[a] Ukrainian State University of Science and Technologies, Dnipro, Ukraine
[b] University of Delaware, Newark, USA

As is well known, the first stage of starch heat treatment is the dehydration process, which proceeds in a kinetic mode with a relatively high activation energy value of 40–60 kJ/mol. The reason for this may be both the energy-consuming process of hydrogen bond destruction in starch and the chemical reaction between the OH groups of polycarbohydrates with the formation of ester bonds. The aim of the study was to find evidence of the formation of such bonds during the heat treatment of corn starch. The studies were performed by FTIR spectroscopy in the temperature range of 60–255^{0}C at a heating rate of 4^{0}C/min. It was found that the number of OH groups of polycarbohydrates gradually decreases during starch heating, while the water content and ether-bond signals, on the contrary, increase. An increase in water concentration was observed when starch was heated to a temperature of 130^{0}C. It was concluded that these trends are well explained by the course of the condensation reaction between the OH groups of amylose and amylopectin molecules. For the first time, it was found that during prolonged drying of starch in a vacuum, even at a temperature of 50^{0}C, the crosslinking of polycarbohydrate molecules occurs due to the formation of ester bonds. It is noteworthy that such crosslinking occurs without the use of chemical reagents, but only by shifting the equilibrium of the condensation reaction as a result of the continuous removal of water from starch.

Keywords: FTIR spectroscopy, corn starch, activation energy, heat treatment, condensation reaction, ether bond.

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Introduction

Despite the long history of studying native and modified starches, their research is still relevant due to their widespread use in the food industry, pharmaceuticals, pulp and paper, and textile industries [1,2]. Native starch is of limited use due to its inherent disadvantages, such as poor solubility and solution stability, as well as a high tendency to retrogradation. Various methods of starch modification are used to improve the characteristics and expand the practical application: physical, chemical, enzymatic and their combinations.

Physical modification has the greatest advantages, which, unlike chemical modification, is more environmentally friendly, as it is implemented without chemicals and solvents [3]. Physical modification includes thermal and non-thermal treatment (using grinding, non-thermal plasma, high pressure, ultrasound, pulsed electric discharge or γ-irradiation [4]). The thermal method of starch modification is called dextrinization or pyroconversion. Pyroconversion at 120–200^{0}C produces split starches that have shorter polycarbohydrate molecular chains compared to native ones. It has been shown that the

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first stages of starch heating result in its dehydration, i.e., loss of bound moisture. Dextrinization begins at a temperature of about 130^{0}C and a starch moisture content of just over 3%. It is believed that the initial period of the process is dominated by the breakdown of starch polycarbohydrates. However, the products of their degradation can enter into polymerization and condensation reactions to form highly branched structures [5].

The processes of heat treatment of starches have been repeatedly studied by thermal analysis methods [6–10]. Based on the study of corn starch by differential thermal analysis, it was found that the apparent activation energy of the dehydration process is 44 kJ/mol [9]. However, studies by thermogravimetry and differential scanning calorimetry and calculations using the Horowitz–Metzger formula showed a much higher value of the activation energy of the dehydration process of corn starch, 63 kJ/mol [10]. Such different values of the activation energy can be explained by the different degrees of initial moisture content of the studied starch samples. The dependence of the kinetic parameters of the starch dehydration process on the water content is still in the field of research [11,12].

The relatively high activation energy of the process indicates that it proceeds in a kinetic mode. It is known that the breaking of one hydrogen bond requires an energy of about 21 kJ/mol. Therefore, the high values of the activation energy of the dehydration process can be explained by the fact that water molecules in starch are simultaneously bound to polycarbohydrates by several hydrogen bonds. Obviously, when there is an excess of water in starch, its molecules do not form several hydrogen bonds, i.e., they have a lower binding energy with starch polycarbohydrates.

In addition to this hypothesis, it can also be assumed that the high value of the activation energy of the starch dehydration process is due not so much to the breaking of hydrogen bonds between water molecules and polycarbohydrates as to the chemical reaction of the formation of an ether C–O–C bond between starch polycarbohydrate molecules. This conclusion was made by the authors of [13] in the study of dextrinization of potato starch. So far, the possibility of the formation of intermolecular ester bonds during the heat treatment of corn starch has not been experimentally proven.

Thus, the aim of this work was to find evidence of the formation of ether bonds between the macromolecules of corn starch polycarbohydrates during its heat treatment. In our opinion, these processes are conveniently studied by IR spectroscopy, since this method, based on the analysis of the vibration characteristics of chemical bonds of molecules, allows us to directly observe changes in the chemical composition of a substance.

Experimental

High-grade corn starch (according to the Ukrainian state standard DSTU 3976-2000) with a mass fraction of hydrated water of 13.5% and total ash of 0.20% was used in the experiments.

FTIR spectra were collected in a Nicolet 380 FTIR spectrometer employed with single reflection Attenuated total reflectance (ATR) cell–Golden Gate with solid diamond enabling spectra collection in the temperature range of 50–250^{0}C. To refer intensity to the same optical density, the dynamometric limiter of the ATR cell was adopted to ensure that the pressure applied to the diamond crystal through the sample was the same in each measurement. FTIR spectra of starches were recorded during heating of samples from 50 to 250^{0}C with a heating rate of 4^{0}C/min. Starch samples were previously lyophilized over 12 using a freeze dryer.

Results and discussion

Figure 1 shows the FTIR spectra of corn starch recorded during its heating to 255^{0}C. The heating rate was 4^{0}C/min. A Fourier transform infrared spectrometer was used, which allowed almost instantaneous recording of spectra at a constant temperature during the heating of the samples. The starch samples were pre-incubated for 12 hours at room temperature in a vacuum (a lyophilizer with a residual vacuum of 10^{-2} mm Hg was used).

As can be seen from Fig. 1, the intensities of the spectra naturally increase with increasing

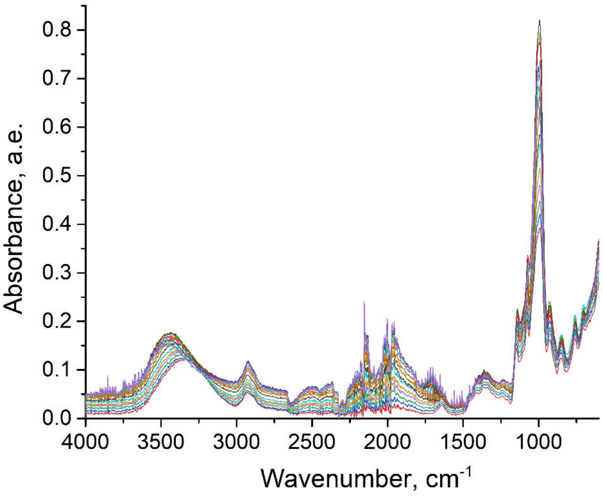

Fig. 1. FTIR spectra of corn starch recorded during its heating. The spectrum was recorded every 12 min, the lower curve is for 60^{0}C, the upper curve is for 255^{0}C

temperature. At the same time, the maxima of the bands in the range of 3800–3000 cm^{-1}, caused by the vibrations of atoms in O–H bonds, monotonically shift with increasing temperature towards higher wavenumbers. Obviously, the shift of the maxima of the absorption bands in the range of 3800–3000 cm^{-1} indicates a change in the vibration characteristics of O–H bonds not in water molecules, but in the hydroxyl groups of starch polymers. It is likely that with increasing temperature, the number of OH groups that were connected by hydrogen bonds decreases. Such changes are caused by changes in both the structure and intermolecular interaction of starch polymers.

It is interesting to note that in the process of heating starch, the peak at 1645 cm^{-1}, which is due to the presence of hydrated water, does not change its position along the frequency axis. Undoubtedly, with a decrease in the content of hydrated water during starch heating, a decrease in the intensity of this peak should be expected. However, the data obtained indicate an increase in the height of the peak at 1645 cm^{-1} with increasing temperature, as for all other absorption bands. As is well known, to compare the intensities of the absorption bands of the IR spectrum at different temperatures, it is necessary to correct the baseline. For such a correction, we chose the absorption band at 2929 cm^{-1}. From the reference data, it is known that the band at 2929 cm^{-1} characterizes the vibrations of C–H bonding atoms, which is the basis of the carbon backbone of amylose and amylopectin. As can be seen from Fig. 1, the position of the maxima of this absorption band does not depend on the increase in the starch heating temperature. This allows it to be used to assess the effect of temperature on the intensity of spectral bands. It was found that the intensity of the absorption band at 2929 cm^{-1} monotonically increases with increasing temperature. Only at temperatures above 200^{0}C does the intensity increase stop, which is due to the beginning of the starch destruction (dextrinization) process [10].

Figure 2 shows the results of comparing the intensity of the maximum absorption bands in the IR spectra of starch at 1656 and in the range of 3800–3000 cm^{-1} with increasing temperature. The figure also shows the differential curves constructed using the baseline correction procedure, which was chosen as the data for the absorption band at 2929 cm^{-1}.

As mentioned above, the fluctuation at 1645 cm^{-1} in the IR spectrum of starch is due to the presence of water. Therefore, in accordance with curve 4 in Fig. 2, it should be concluded that the water content in the starch sample under study increases continuously when heated to a temperature of 133^{0}C. At the same time, it is known from thermogravimetric studies [10] that starch loses water during heating: mass loss begins immediately with heating and ends at about 100^{0}C.

In our opinion, the increase in the water content of starch when it is heated above 100^{0}C should be explained by the condensation reaction between the OH groups of amylose and amylopectin molecules. For such a reaction in the starch solid phase, it is necessary that the OH groups of polycarbohydrates are not only located opposite each other, but also connected by a hydrogen bond, i.e., have a suitable configuration for the formation of an ether bond with the cleavage of a water molecule:

$$R_1{-}OH\cdots OH{-}R_2 \rightarrow R_1{-}O{-}R_2 + H_2O\uparrow, \qquad (1)$$

where R_1 and R_2 are fragments of starch polycarbohydrates.

Obviously, when the starch runs out of such «reactive» OH groups, the reaction will stop. Due to the high temperature, the water will continuously evaporate, which is observed on curve 4 in Fig. 2.

The differential curve 2 in Fig. 2 for the absorption band maxima in the range of 3800–3000 cm^{-1} shows an almost linear decrease in its intensity, which indicates a continuous decrease in the content of OH groups. However, attention should be paid to the deviation from the monotonic decrease in the intensity of this absorption band in the temperature range of 100–150^{0}C. The positions of the maxima along the temperature axis in differential curves 2 and 4 are

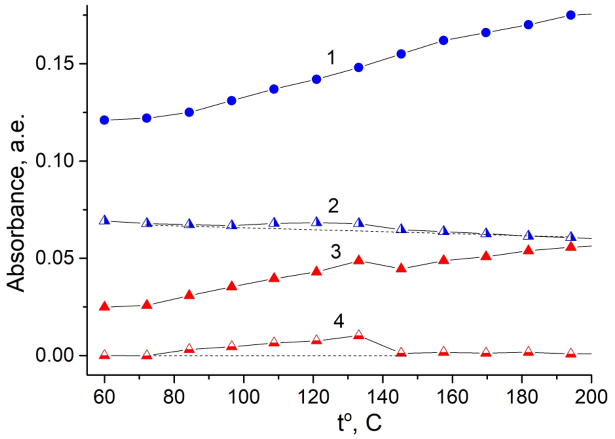

Fig. 2. Changes in the intensity of the maximum absorption bands at 3800–3000 cm^{-1} (1) and 1656 cm^{-1} (3) for starch during its heat treatment. Curves 2 and 4 are differential curves constructed in accordance with the baseline correction procedure for the 3800–3000 and 1645 cm^{-1} bands, respectively

well correlated, which allows us to explain them by the same reasons: the release of water by reaction (1) with a parallel decrease in the content of OH groups of starch polycarbohydrates.

We considered the possibility of confirming the condensation reaction (1) based on the analysis of changes in the absorption bands of water and hydroxyl groups of amylose and amylopectin. However, reaction (1) has one more product, the C–O–C ether bond. It is obvious that the condensation reaction of hydroxyl groups should increase the content of C–O–C bonds, and therefore, the intensity of the absorption bands caused by the vibrations of atoms in ether bonds should increase.

It is known that asymmetric valence vibrations of C–O–C in the IR spectra are usually located in the range of 1310–1000 cm^{-1} and their frequencies can vary depending on the surrounding atoms that give or take electron density from carbon atoms. The symmetric valence vibrations of C–O–C are usually located in the range of 1055–870 cm^{-1}. In the infrared spectrum of the starch we studied, we observe two peaks in the wavenumber range for asymmetric C–O–C valence vibrations: at 1146 and 1076 cm^{-1}, and in the range of 1055–870 cm^{-1} there are peaks: 1012, 990, 927, and 850 cm^{-1}. The analysis of the change in the intensity of the largest absorption band at 990 cm^{-1} as a function of the sample heating temperature showed that its intensity increases monotonically even after the baseline correction (Fig. 3, curve 3). Thus, the IR spectral data for the C–O–C valence vibrations also confirm the course of the condensation reaction of OH groups with the formation of additional ether bonds.

The conclusions about the possibility of the condensation reaction of hydroxyl groups of amylose and amylopectin at temperatures below 100^0C are quite unusual. Therefore, experiments were conducted to study the IR spectra during the heating of maximally dehydrated starch. The fact is that against the background of the process of removing water from starch at 50–100^0C, the formation of new water by reaction (1) is not noticeable. In our opinion, it is possible to detect water formed by the condensation reaction in starches by studying the infrared spectra of starches in which water has been completely removed. In order to maximize dehydration, the starch samples were kept in a vacuum with a residual pressure not exceeding 10^{-5} mm Hg for 12 hours at a temperature of 50^0C (the temperature was not raised higher to avoid initiating the process of starch gelatinization). The vacuum was created by a turbomolecular pump paired with a forevacuum pump. Figure 4 shows the FTIR spectra of dehydrated corn starch in the temperature range of 50–250^0C. As can be seen from the figures, the spectra of starch completely lack peaks at 1645 cm^{-1}, which indicates its complete dehydration.

Due to the complete absence of water in starch, the absorption band at 3800–3000 cm^{-1} characterizes the content of only OH groups of amylose and amylopectin. Figure 5 shows the change in the intensity of the absorption bands at 3800–3000 cm^{-1} for fully dehydrated starch during its heat treatment. The differential curve (as the difference between the intensity at the maximum of the band at 3800–3000 cm^{-1} and the intensity of the background line of C–H bonds) is almost linear. This means that the number of OH groups in the interval 50–200^0C does not change, i.e., their condensation reaction does not occur. This fact can be explained by the fact that already at the stage of dehydration at a temperature of

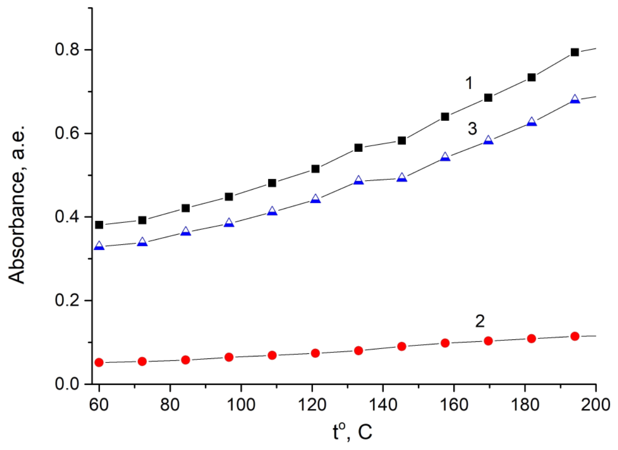

Fig. 3. Changes in the intensity of the absorption band at 990 cm^{-1} (1), CH bonds at 2929 cm^{-1} (2) and differential curve (3) for starch during its heat treatment

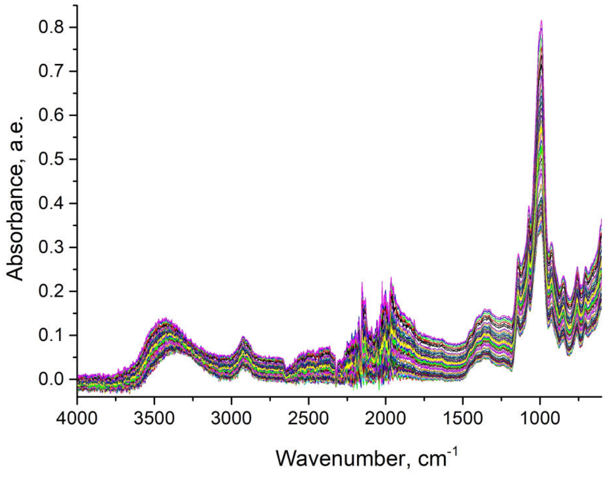

Fig. 4. FTIR spectra of dehydrated corn starch recorded during its heating. The spectrum was recorded every 5 min, the lower curve is for 50^0C, the upper one is for 250^0C

50^{0}C, all the OH groups of carbohydrate macromolecules that were located nearby and connected by hydrogen bonds entered into a condensation reaction. Obviously, the continuous removal of water from starch under vacuum conditions contributed to the shift in the equilibrium of reaction (1) toward its products according to the Le Chatelier-Brown principle.

Conclusions

The study of the FTIR spectra of starch at different heating temperatures allowed us to analyze changes in the content of hydrated water, OH groups, C–H and C–O–C bonds of starch during its heat treatment. It was found that the water content in starch does not decrease but rather increases when heated at temperatures above 100^{0}C. It was also found that during heating the number of OH groups of polycarbohydrates continuously decreases, and the content of C–O–C bonds increases. Such regularities are well explained by the course of the condensation reaction between the OH groups of amylose or amylopectin molecules with the formation of an ester bond and the release of water. Obviously, only those OH groups of polycarbohydrates that have the necessary orientation for molecular transformation and are most likely connected by hydrogen bonds can undergo such reactions.

For the first time, it was found that during prolonged drying of starch in a vacuum, even at a temperature of 50^{0}C, polycarbohydrate molecules are cross-linked due to the formation of ester bonds. It is noteworthy that such cross-linking of starch macromolecules occurs without the use of additional chemical reagents, but only due to a shift in the equilibrium of the condensation reaction from left to right as a result of the continuous removal of water from starch.

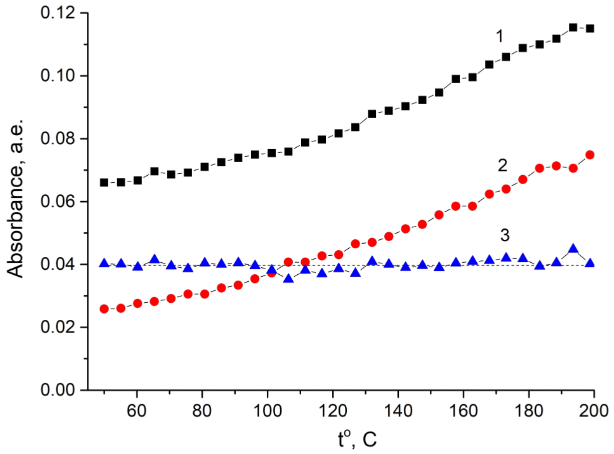

Fig. 5. Changes in the intensity of the maximum absorption bands of OH groups at 3800–3000 cm^{-1} (1), CH bonds at 2929 cm^{-1} (2) and the differential curve (3) for fully dehydrated starch during its heat treatment

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ПОШУК ХІМІЧНОЇ СКЛАДОВОЇ ПРОЦЕСУ ДЕГІДРАТАЦІЇ КРОХМАЛЮ

М.В. Ніколенко, Ю.Е. Сахно, В.Д. Миргородська-Терентьєва, Д.С. Гречка, О.Ю. Вашкевич

Як відомо, першим етапом термічної обробки крохмалів є процес дегідратації, який перебігає у кінетичному режимі з відносно великим значенням енергії активації: від 40 до 60 кДж/моль. Причиною цьому може бути як енерговитратний процес руйнування водневих зв'язків у крохмалі, так і протікання хімічної реакції між ОН-групами полівуглеводів з утворенням ефірних зв'язків. Мета даної роботи полягала в пошуку доказів утворення таких зв'язків під час термообробки кукурудзяного крохмалю. Дослідження були виконані методом FTIR спектроскопії в інтервалі температур 60–255^{0}С при швидкості нагріву 4^{0}С/хв. Встановлено, що під час нагрівання крохмалю кількість ОН-груп полівуглеводів поступово зменшується, а вміст води і ефірних зв'язків, навпаки, збільшується. Зростання концентрації води спостерігається при нагріванні крохмалю аж до температури 130^{0}С. Зроблено висновок, що такі закономірності добре пояснюється перебігом реакції конденсації між ОН-групами молекул амілози та амілопектину. Вперше встановлено, що під час тривалої сушки крохмалю у вакуумі навіть за температури 50^{0}С відбувається «зшивання» молекул полівуглеводів внаслідок утворення ефірних зв'язків. Примітним є той факт, що така зшивка відбувається без застосування хімічних реагентів, а лише за рахунок зміщення рівноваги реакції конденсації в результаті безперервного видалення води з крохмалю.

Ключові слова: Фур'є ІЧ-спектроскопія, кукурудзяний крохмаль, енергія активації, термічна обробка, реакція конденсації, ефірний зв'язок.

SEARCHING FOR THE CHEMICAL COMPONENT OF THE STARCH DEHYDRATION PROCESS

M.V. Nikolenko [a, *], Yu. Sakhno [b], V.D. Myrhorodska-Terentieva [a], D.S. Hrechka [a], O.Yu. Vashkevych [a]

[a] **Ukrainian State University of Science and Technologies, Dnipro, Ukraine**

[b] **University of Delaware, Newark, USA**

[*] **e-mail: n_nikolenko@ukr.net**

As is well known, the first stage of starch heat treatment is the dehydration process, which proceeds in a kinetic mode with a relatively high activation energy value of 40–60 kJ/mol. The reason for this may be both the energy-consuming process of hydrogen bond destruction in starch and the chemical reaction between the OH groups of polycarbohydrates with the formation of ester bonds. The aim of the study was to find evidence of the formation of such bonds during the heat treatment of corn starch. The studies were performed by FTIR spectroscopy in the temperature range of 60–255^{0}C at a heating rate of 4^{0}C/min. It was found that the number of OH groups of polycarbohydrates gradually decreases during starch heating, while the water content and ether-bond signals, on the contrary, increase. An increase in water concentration was observed when starch was heated to a temperature of 130^{0}C. It was concluded that these trends are well explained by the course of the condensation reaction between the OH groups of amylose and amylopectin molecules. For the first time, it was found that during prolonged drying of starch in a vacuum, even at a temperature of 50^{0}C, the crosslinking of polycarbohydrate molecules occurs due to the formation of ester bonds. It is noteworthy that such crosslinking occurs without the use of chemical reagents, but only by shifting the equilibrium of the condensation reaction as a result of the continuous removal of water from starch.

Keywords: FTIR spectroscopy; corn starch; activation energy; heat treatment; condensation reaction; ether bond.

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