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# THE INFLUENCE OF THE DEGREE OF MOISTURE AND TEMPERATURE OF STARCH HEAT TREATMENT ON THE VISCOSITY OF PASTE SOLUTIONS

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The possibilities of heat treatment of starch have not yet been fully utilized. The fact is that thermal treatment of starches can produce not only «split» starches, but also «cross-linked» starches, in which polycarbohydrate molecules are additionally bound by ether bonds. The latter can be formed by condensation of polycarbohydrate hydroxyl groups during starch heating without the use of additional chemical reagents. It has been suggested that the content of hydrate water in dry starch is an important factor because it can correlate with the number of intermolecular essential links of polycarbohydrate chains. Therefore, the purpose of the research is to determine the dependence of the viscosity of the paste solutions on the degree of initial humidity and the temperature of the heat treatment of starch. Studies have shown that there is a linear correlation between the kinematic viscosity of starch solutions and the hydrated water content in starch. The dependence of the viscosity of native starch paste solutions on the heat treatment temperature has the form of a convex curve, whereas for vacuum starch this dependence has the form of a rapidly declining exponential curve. It has been shown for the first time that preliminary vacuum dehydration of starch allows for a significant reduction in the temperature and time of starch dextrinization. FTIR studies of dehydrated starch spectra show a decrease in the number of intermolecular hydrogen bonds, which may be the main reason for the acceleration of their thermal degradation compared to starches with a higher degree of moisture.

**Keywords**: heat treatment of starch, starch dehydration, dextrinization, vacuum, FTIR spectra, airwave communication, amylose paste.

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## *Introduction*

Starches are the most common polycarbohydrates in the plant world after cellulose. They are used in the food, pulp and paper, textile, pharmaceutical industries, in the construction industry, in the production of packaging materials and disposable tableware [1]. Native starch has limited application primarily due to disadvantages such as low solubility and instability of paste solutions. Modified starches are used to overcome these limitations [2,3].

The method of modifying starch by heat treatment, known as dextrinization or pyroconversion, has become widespread, the essence of which is the dry heating of native starches in the presence or absence of catalysts. This modification method allows for the production of «split» starches, starches with a

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lower molar mass of polycarbohydrates due to their depolymerization. It is known that when starches are heated, the first stage is a dehydration process, which begins immediately when the temperature only rises and ends at approximately 100–110$^0$C [4]. As a rule, it is believed that the dehydration process does not affect the subsequent thermal decomposition of starch, since all water evaporates before its decomposition begins [5,6].

The second stage of starch weight reduction begins at a temperature of 200$^0$C and ends at approximately 350$^0$C. It has been proven that polycarbohydrates not only break down into oligomers, but also chemically transform in the atmosphere to form water, carbon oxides, acetaldehyde, methane, and furans [7]. It should be noted that the efficiency of the starch dextrinization process depends not only on the temperature, but also on the duration of heat treatment. For example, during short-term heat treatment (15–20 min), dextrinization processes are carried out at temperatures above 200$^0$C, while dextrinization at temperatures of 120–130$^0$C is carried out for at least 2.5 hours.

In our opinion, the possibilities of heat treatment of starch have not yet been fully utilized. The fact is that by heat treatment of starches, it is also possible to obtain not only «split» but also «cross-linked» starches, in which polycarbohydrate molecules are additionally linked by ether bonds. The latter can be formed by the condensation of hydroxyl groups of hydrocarbons during the heating of starch without the participation of additional chemical reagents. Obviously, for this it is necessary to carry out heat treatment in such a way as not to initiate the processes of destruction of polymer chains of saccharides.

This hypothesis is based on the results of studies of starches using FTIR spectroscopy during their thermal treatment [8]. Based on the analysis of changes in the content of water, OH groups, and ether bonds during heating of starches, it was concluded that the condensation reaction between the OH groups of starch polycarbohydrates occurs in a wide temperature range. This is explained by the gradual involvement of new hydroxyl groups in the esterification reaction, which were initially unavailable for interaction, but over time acquire such properties due to conformational changes in amylose and amylopectin macromolecules during heating. According to the results of FTIR spectroscopy of starches, it can be assumed that the content of hydration water in dry starch is an important factor, since it can correlate with the number of intermolecular ester bonds between polycarbohydrate molecules.

It is obvious that the difference in the properties of split and cross-linked starches is easiest to demonstrate by comparing the viscosity of their paste solutions. According to the data of the authors [9,10], who studied the rheological properties of starch pastes, the lower the degree of amylose polymerization, the lower the viscosity of the paste under other equal conditions, namely: concentration, temperature, shear rate, etc. Studies of the dependence of the dynamic viscosity of hot pastes on their shear rate [11] showed that the viscosity of pastes of heat-treated starches depends on the conditions of heat treatment and increases in the sequence: heat-treated starch in the presence of an acid catalyst (decomposed starch), native starch, heat-treated starch without a catalyst (cross-linked starch). These patterns were explained by the difference in the degree of polymerization of starch polycarbohydrates, but the reasons for such differences have not been studied in detail.

Therefore, the purpose of this study is to determine the dependence of the viscosity of pastes on the degree of initial moisture content and the temperature of heat treatment of starch. The study of the regularities of starch modification processes through its thermal and vacuum dehydration and the study of the properties of their paste solutions, namely their viscosity, is of undoubted scientific and practical importance.

## *Experimental*

The experiments used top-grade potato starch (Ukrainian state standard DSTU 4286:2004) with a mass fraction of moisture of 15.9%. The moisture content of starch samples was determined in accordance with the state standard GOST 7698-93 «Starch. Acceptance rules and methods of sampling» by drying to constant weight.

For vacuum dehydration, starch was placed in a vacuum flask and connected to a vacuum pump. Starches were kept under a vacuum of 0.06–0.08 MPa at room temperature for 6–10 hours. Samples with different heat treatment temperatures were prepared from native and vacuum-treated starches. For this purpose, the starches were spread in a thin layer in a drying oven and heated to a selected temperature in the range of 40–160$^0$C at a rate of 10$^0$C/min. The starches were heated for 2.5 hours (the time was started from the moment the set temperature was reached in the drying oven). The paste solutions were prepared from the obtained samples according to the standardized method and measured their kinematic viscosity.

To produce paste solutions up to 100 ml of distilled water heated in a water bath up to 75$^0$C, added when stirring 1.00 g of starch. The suspension was kept for one hour under stirring, and then cooled to room temperature and settled. The solution above the sediment was used to measure kinematic viscosity using an Ostwald capillary viscometer. Viscosity measurements were performed at least 5 times, and the average value, confidence interval, and relative standard deviation were calculated. Its value for all measurements performed did not exceed 2% on average, which indicates a sufficient degree of reproducibility of measurements.

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. For the purpose of maximum dehydration, a sample of starch before spectroscopic studies was kept in a vacuum with a residual pressure of no more than $10^{-5}$ mm Hg for 12 hours at a temperature of 50$^0$C. The vacuum was created using a turbomolecular pump paired with a forevacuum pump.

### *Results and discussion*

*Spectroscopic studies of starches with different degrees of moisture*

Thermal transformations of starch are a set of reactions, and it is obvious that the transformation routes and reaction rate constants may depend on such parameters as the heating rate, the degree of starch crystallinity, and the presence of catalysts, acids, alkalis, or salts. It can be assumed that such factors include hydrated water, as it can be a participant in the chemical transformations of polycarbohydrates. From this point of view, the patterns of chemical transformations of native and dehydrated starches during their heat treatment may differ significantly. Therefore, it was of interest to conduct spectroscopic studies of starches directly during the heating process in order to compare their FTIR spectra and determine the temperature range in which similarity of their spectral characteristics is observed.

Figure 1 shows the FTIR spectra of dehydrated potato starch, recorded during its heating in the temperature range of 50–250$^0$C. Similar data for native potato starch were published elsewhere [8]. Comparison of the spectra showed that the main difference is observed for the absorption band in the range of 4000–3000 cm$^{-1}$. As is known, this band in the FTIR spectra of starches characterizes the vibrations of both water molecules and hydroxyl groups of polycarbohydrates. They are characterized by three types of vibrations: symmetrical and asymmetrical vibrations of the O–H bond atoms with wave numbers at ~3700 and ~3600 cm$^{-1}$, and vibrations of hydrogen atoms with a change in the ∠HOH angle at ~1600 cm$^{-1}$. As can be seen from Fig. 1, the peak at 1600 cm$^{-1}$ is completely absent from the spectra, which indicates the absence of water in the starch sample under study. Due to the absence of water in starch, the absorption band at 4000–3000 cm$^{-1}$ is due to vibrations of only the OH groups of starch polycarbohydrates.

For better data detailing, Figures 2 and 3 show fragments of FTIR spectra for the studied starches in the wave number range of 3800–3200 cm$^{-1}$. In the case of native starch with an increase in temperature, the intensity of this absorption strip continuously

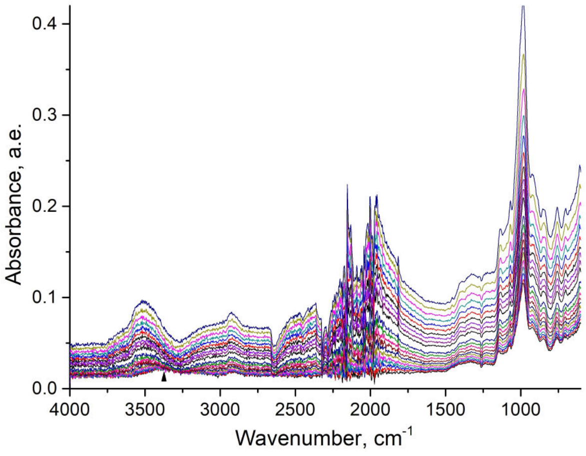

Fig. 1. FTIR spectra of dehydrated potato starch heated from 50$^0$C (bottom line) up to 250$^0$C (upper line) in 49.25 min

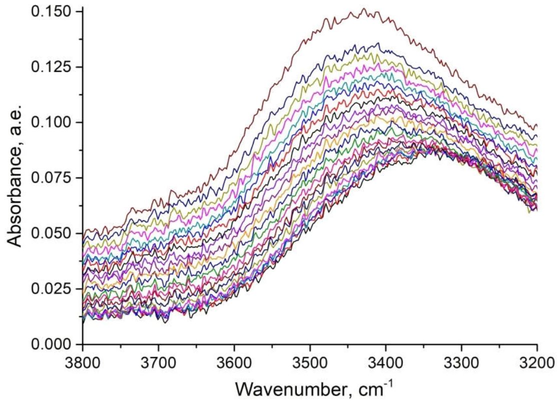

Fig. 2. Fragment of FTIR spectra of native starch heated from 50$^0$C (bottom line) up to 250$^0$C (upper line) in 49.25 min

increases and gradually shifts to the left along the axis of wave numbers. The same pattern is observed for dehydrated starch, but only up to a temperature of 160$^0$C (Fig. 3). With further heating of the starch, the intensity of the absorption band of OH groups continues to increase, but its maximum ceases to shift along the axis of wave numbers.

The shift of the band maxima in the range of 3800–3000 cm$^{-1}$ is generally accepted to be explained by a change in the polarization effect on the OH groups of their molecular environment during the heating of starch. It is known, for example, that the vibration frequencies of atoms in OH groups depend on their participation in hydrogen bonds. The stronger such bonds, the more the absorption band shifts to the low-wave region of the spectrum. Therefore, the continuous shift of the absorption band in Fig. 2 to the high-wave side of the spectrum during the heating of starch indicates a decrease in the number of OH groups linked by hydrogen bonds. Obviously, the absence of such a shift upon reaching a temperature of 160$^0$C indicates the complete destruction of hydrogen bonds between the OH groups of the polycarbohydrates of dehydrated starch.

Thus, up to a temperature of 160$^0$C, both studied starches show identical spectral characteristics, which allows us to conclude that the patterns of their chemical transformations during heat treatment are similar.

*Viscosity of starch pastes*

As is known, the viscosity of starch pastes is due to the ability of amylose macromolecules dissolved in water to form a three-dimensional grid with the participation of hydrogen bonds. The degree of viscosity is also affected by the swollen shells (so-called sacs) of starch granules, which consist of amylopectin. It is obvious that comparing the viscosity of paste solutions for native and modified starches requires standardization of their pasteification process, that is, conducting the process under identical conditions. To compare the properties of pastes, we chose a method for obtaining «amylose» pastes, that is, pastes without inclusions of starch granule shells.

Figure 4 presents the results of kinematic viscosity measurements for paste solutions of native starch (with a moisture content of 15.9%) and vacuum-dehydrated starch (with a moisture content of 7.6%). Starches were first subjected to heat treatment in the range of 40–160$^0$C for 2.5 hours, and then paste solutions were prepared from the obtained samples.

The dependence of the kinematic viscosity of paste solutions of native starch on the heat treatment temperature has the form of a convex curve: up to the isothermal holding temperature of starch of 60$^0$C, the viscosity of the paste monotonically increases, and with a further increase in temperature it monotonically decreases. It can be concluded that heat treatment of native starch above 60$^0$C reduces the degree of polymerization of starch polymers, which makes them more soluble in water and, accordingly, reduces the viscosity of pastes.

Unlike native starch pastes, there is no maximum in the dependence of the viscosity of vacuum-modified starch pastes on the temperature of its heat treatment (Fig. 4, curve 2). Reducing the moisture content of starch before its heat treatment not only changes the nature of the dependence of the viscosity of their pastes on the heat treatment temperature, but also leads, under the same conditions, to much faster

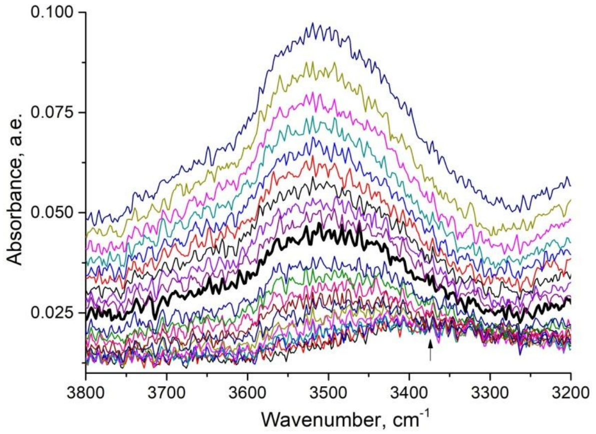

Fig. 3. Fragment of FTIR spectra of dehydrated potato starch heated from 50$^0$C (bottom line) up to 250$^0$C (upper line) in 49.25 min. The fat line shows the spectrum at a temperature of 159$^0$C. The arrow shows the maximum of the absorption strip at a temperature of 50$^0$C

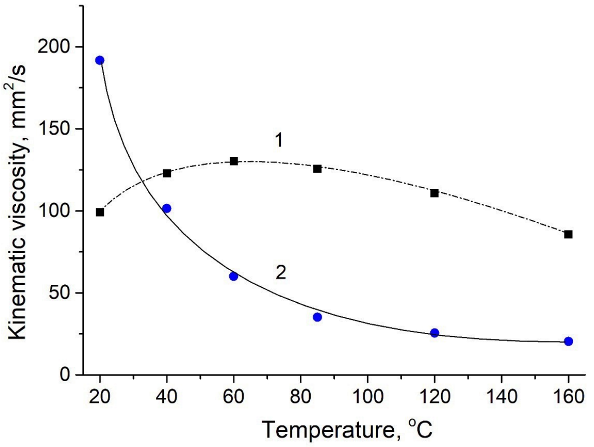

Fig. 4. Comparison of the kinematic viscosity of paste solutions of native (1) and vacuum-modified starch (2) with the temperature of heat treatment of starches for 2.5 hours

dextrinization of starch. Already at a heat treatment temperature of 40⁰C, vacuum-modified starch pastes are characterized by lower viscosity than native starch pastes. This pattern indicates the significant role of hydration water in the processes of starch dextrinization: the lower the initial moisture content of starch, the faster its thermal destruction occurs.

From a practical point of view, it is interesting to compare the viscosity of vacuum-modified starch pastes with their content of hydrated water for a wider range of samples. To conduct such research, we prepared starch samples with varying vacuuming time. Measurements of the degree of humidity showed that the content of hydration water in the obtained samples is 3.9, 4.6 and 10.1%. The results of comparing the viscosity of starch pastes with the degree of their moisture content are shown in Fig. 5. This dependence can be described by a linear correlation with a fairly large correlation coefficient $R^2$=0.9677.

Extrapolation of the established dependence shows that reducing the starch moisture content, for example, to 1%, will allow obtaining pastes with a viscosity of 245 mm²/s, which is 2.5 times higher than the viscosity of native starch paste. It should be noted that the indicated viscosity values characterize pastes obtained by adding 1.00 g of starches per 100 ml of water at a temperature of 75⁰C with subsequent separation of the amylopectin shells of starch granules from the precipitate. It is obvious that with the increase in the concentration of starch and temperature and without the separation of the starch shells it is possible to obtain pastes with much larger viscosity values.

## *Conclusions*

The dependence of the viscosity of starch solutions on the content of hydration water in starch and the temperature of its heat treatment has been experimentally proven. The dependence of the kinematic viscosity of paste solutions of native starch on the heat treatment temperature has the form of a convex curve, which can be explained by the competition of two processes: intermolecular polymerization of amylose and its destruction into oligomers with a lower molar mass. The dependence of the kinematic viscosity of the paste solution of vacuum starch has the appearance of a rapidly falling exponential curve. It has been shown for the first time that preliminary vacuum dehydration of starch allows for a significant reduction in the temperature and time of starch dextrinization, which can lead to a significant economic effect when implementing the proposed modification method in the production of dextrins. This pattern indicates the significant role of hydration water in the processes of starch dextrinization: the lower the initial moisture content of starch, the faster its thermal destruction occurs.

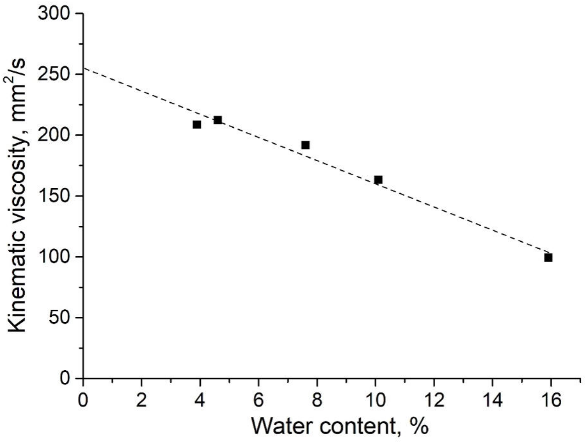

Fig. 5. Comparison of the kinematic viscosity of starch pastes with the content of hydrated water in starch. (Starch samples were not subjected to temperature treatment after vacuuming)

Studies of FTIR spectra of native and vacuum dehydrated starches showed their similarity only up to a heating temperature of 160⁰C. If for native starch with an initial moisture content of 15.9% the absorption band of OH bonds in the range of 3800–3200 $cm^{-1}$ continuously shifts along the wave number axis with an increase in temperature from 50 to 250⁰C, then for dehydrated starch (with an initial moisture content of 7.6%) such a shift occurs only up to a temperature of 160⁰C. With further heating of this starch, the intensity of the absorption band of OH groups continues to increase, but its maximum ceases to shift along the wave number axis. Such patterns can be explained by changes in the number of OH groups of starch polymers involved in the formation of hydrogen bonds. Apparently, the decrease in the number of hydrogen bonds between polycarbohydrate molecules is the main reason for the acceleration of their thermal destruction compared to starches with a higher degree of moisture.

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## ВПЛИВ СТУПЕНЮ ВОЛОГОСТІ ТА ТЕМПЕРАТУРИ ТЕРМІЧНОГО ОБРОБЛЕННЯ КРОХМАЛЮ НА В’ЯЗКІСТЬ КЛЕЙСТЕРНИХ РОЗЧИНІВ

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Можливості термічного оброблення крохмалю дотепер не використовуються повною мірою. Річ в тому, що термічним обробленням крохмалів можливо одержати не тільки «розщеплені», але і «зшиті» крохмалі, в яких молекули полісахаридів додатково зв’язані ефірними зв’язками. Останні можуть утворюватись за рахунок конденсації гідроксильних груп полісахаридів під час нагрівання крохмалю без участі додаткових хімічних реагентів. Було висказано припущення, що вміст гідратної води у сухому крохмалі є важливим фактором, оскільки він може корелювати з кількістю міжмолекулярних ефірних зв’язків полісахаридних ланцюгів. Тому метою досліджень обрано визначення залежності в’язкості клейстерних розчинів від ступеню початкової вологості та температури термічної обробки крохмалю. Дослідження показали, що між кінематичною в’язкістю крохмальних розчинів і вмістом в крохмалі гідратної води спостерігається лінійна кореляція. Якщо залежність в’язкості клейстерних розчинів нативного крохмалю від температури термооброблення має вид випуклої кривої, то для вакуумованого крохмалю така залежність має вид швидко спадаючої експоненціальної кривої. Дослідження FTIR спектрів зневодненого крохмалю показали зменшення кількості міжмолекулярних водневих зв’язків, що може бути основною причиною прискорення їх термічного руйнування порівняно з крохмалями з вищим ступенем вологості.

**Ключові слова**: термічне оброблення крохмалю, дегідратація крохмалю, декстринізація, вакуум, FTIR спектри, ефірний зв’язок, амілозний клейстер.

# THE INFLUENCE OF THE DEGREE OF MOISTURE AND TEMPERATURE OF STARCH HEAT TREATMENT ON THE VISCOSITY OF PASTE SOLUTIONS

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**Keywords**: heat treatment of starch; starch dehydration; dextrinization; vacuum; FTIR spectra; airwave communication; amylose paste.

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