

STUDY OF THE HEAT OF DEHYDRATION OF D-FRUCTOSE SOLUTION

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Abstract. The heat of evaporation of water is the main component of the consumption during the drying of bio raw materials. Its share in the consumption depends not only on the initial moisture content of the drying object. As our previous studies have shown, the state of water in the material affects the total heat consumption. The state of water is determined by its chemical composition and structure. Drying objects rich in sugars deserve special attention. Sugars have a high tendency to hydration, are concentrated in the juice and determine both the kinetics and heat of dehydration. The most reliable data on glucose and fructose hydration are analyzed and presented. Measurement of the specific heat of evaporation of water r from fructose solutions with an initial concentration of 12.5 wt. % was performed in the evaporation calorimeter. Continuous registration of changes in heat flows and mass of the sample was ensured during the measurement process. The dependence of the reduced specific heat of evaporation of water ($R = r/r_{tab}$) on the concentration of fructose was obtained in the isothermal mode at 40, 60 and 80 °C. The presented curves reflect the thermal processes occurring in the solution during water removal. The processes are characterized by an endothermic period of dehydration and an exothermic period of simultaneous dehydration and spontaneous homogeneous crystallization of fructose. It was found that the change in the degree of hydration of fructose in solution from 12.5 to 5 mol/mol occurs with increasing, almost linear, specific heat consumption. Removal of water from the first coordination sphere, when the degree of hydration decreases to values <5 , the increase in specific heat of evaporation is steeper and reaches the excess over the tabular values of heat of evaporation of pure water r_{tab} by 6 – 7 %. The heat of spontaneous crystallization of fructose has a power greater than the power of endothermic heat consumption for dehydration, therefore, a general exothermic effect is recorded. It is shown that the measured specific heat of water evaporation increases with increasing concentration of solutions despite a decrease in the degree of hydration of fructose. It was established that water has a strong hydrogen bond with sugar in solutions in which the degree of hydration of fructose is less than ~ 5 . Assumptions about strongly and weakly bound hydrated water in solutions of monosaccharides are substantiated.

Key words: heat of evaporation, solution, glucose, fructose, hydration, free and hydrated water.

Introduction. Drying of sugar-containing raw materials has a number of features related to the chemical composition. More than half of the dry matter of many fruits and berries [1] and vegetables [2] are sugars. Thus, their content in apples can reach 66 % (fructose ~ 40.2 %, glucose ~ 14.6 %, sucrose ~ 11.0 %). Due to the good solubility in water, they are concentrated in juice, the amount of which is not infrequently 97 %. The juice concentration increases reaching 88 – 90 % of dry matter in the process of drying to low moisture. That is, the main part of the water removing in the drying process is removed from the juice under conditions of increasing its concentration.

One of the most important factors determining the energy efficiency of drying is the specific heat consumption, the core of which is the heat of water evaporation. Today, the values of the specific heat of evaporation of water from the surface of pure volumetric water are used in the calculations of the drying process. These values are given in numerous reference books.

Evaporation is an endothermic phase change of the first kind as a result of which water from the liquid condensed phase is converted into steam. In this case, the heat is spent on overcoming the forces of molecular adhesion in the liquid phase and on the work of expansion during the transformation of liquid into vapor [3]. The specific heat of evaporation of water decreases with increasing temperature due to the small proportion of heat expended on the transition of the liquid into steam in the total heat of evaporation.

β -D-fructopyranose is the main form of existence of D-fructose. In water solution in thermodynamic equilibrium with β -D-fructopyranose, there is 23.6 % fructofuranose. And the keto form of D-fructose and α -D-fructopyranose are completely absent. The content of fructofuranose in the solution decreases to 15.2% (at 0 °C) with decreasing temperature, and increases to 44.2 % (at 50 °C) with heating. D-glucose forms several forms like D-fructose. The crystalline forms of α -D-glucose and β -D-glucose are quite stable. However, in the solution, each of them slowly turns into an equilibrium mixture of both forms, in which there is 63.8% of the β -anomer and 36.2% of the α -anomer [4].

Sugars in the free state are practically not found in nature. Nature has ensured their existence, giving them the property of hydration – easy interaction with water molecules. The mechanism of hydration is based on ideas about the hydrogen bonds formed between water and hydrophilic polar groups of sugar.

The binding energy between the elementary particles of solute and water depends on the nature of the substance, on its ability to hydration. Hydrogen bonds are formed as a result of donor–acceptor interactions between sugar and water molecules. These hydrogen bonds exceed the strength of the bonds between water molecules [5].

Modern ideas about the energy of intermolecular interactions make it possible to consider water in sugar solutions as being in two different states. In one state it has properties similar to the properties of pure water (free or freezing water). Another state occurs as a result of favorable energy interactions with sugar molecules (hydrated or non-freezing water) [6]. Water associated with dissolved sugar does not undergo a phase change of the first kind when cooled below 0 °C. The method of quantitative determination of free and hydrated water by differential scanning calorimetry (DSC) is based on these differences in the properties of free and bound water [7, 8]. The most complete data on the hydration of sucrose, glucose and fructose were obtained by this method [6, 9–12].

It was found that the amount of hydrated water in the solution depends on the sugar concentration. The proportion of free water in the solution decreases, while the proportion of hydrated water increases with an increase in the sugar content (Fig. 1).

Hydration is probabilistic in nature. Therefore, it is customary to characterize it quantitatively by the average degree of hydration, representing it by means of hydration numbers. Studies and calculations have shown the dependence of hydration numbers on concentration and temperature [10]. Usually, the degree of hydration is expressed as the molar ratio of hydrated water to solute depending on the concentration of the solution (Fig. 2). Studies of hydration of sugars by DSC have shown that the degree of hydration decreases with increasing sugar concentration despite the presence of free water in solutions [10–13].

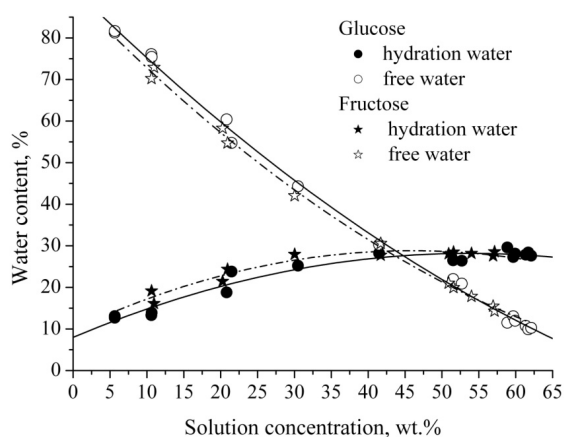


Fig. 1 – The content of hydrated and free water in solutions of D–glucose and D–fructose [12]

The observed nature of the dependence is a reflection of complex intermolecular interactions, which indicate a change in the quantity and quality of hydrogen bonds. Coordination numbers of hydration of D–fructose (5.19 ± 0.15) and D–glucose (4.95 ± 0.15) turned out to be close and coincide with the number of OH groups of these molecules, despite the difference in their isomeric forms in water solutions [12].

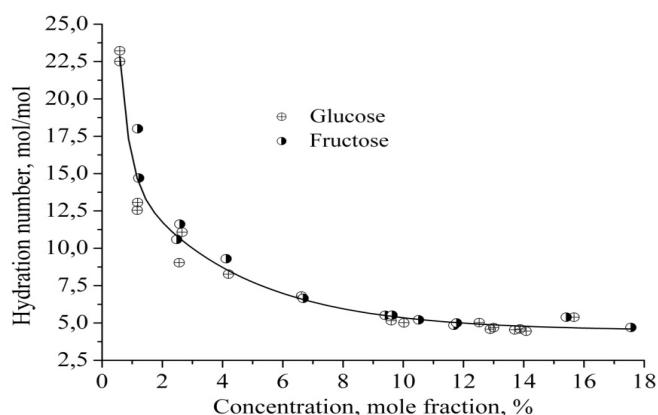


Fig. 2 – Average dependence of hydration numbers of D–glucose and D–fructose on the concentration of solutions [13]

In solutions with low concentrations, the number of water molecules bound to sugar molecules significantly exceeds the number of hydroxyl groups of this sugar. It can be imagined that each sugar molecule has a water shell, the thickness of which decreases with increasing concentration of the solution [10]. The hydration shell prevents the convergence of the solute molecules as long as the value of the degree of hydration exceeds the number of hydroxyl

groups [14]. It becomes possible the emergence of associates (elementary formations of future solid-phase structures) with increasing concentration of D-glucose solutions above 66.89 wt. % (0.49 g water/g dry matter) and D-fructose above 65.84 wt. % (0.52 g water/g dry matter), which correspond to the above coordination numbers of hydration [13]. Hydration curves confirm the existence of different structures in the sugar–water system and indicate a smooth transition from one structure to another when the concentration changes.

Taking into account the information on the hydration of D-glucose and D-fructose [12] and the results of measurements of the specific heat of evaporation of water from a sucrose solution [14], it can be expected that the specific heat of evaporation of water from solutions of monosaccharides should also exceed the specific heat of evaporation from the free surface of pure bulk water. Therefore, an experimental study of the influence of the degree of hydration of fructose on the energy of evaporation of water from its solutions is of both theoretical and practical interest, since fructose is a component of the juice of many fruits and root crops subjected to drying.

Materials and methods. Measurement of the specific heat of evaporation of water from fructose solution was carried out in the evaporation calorimeter, which was designed and manufactured in the IET of NAS of Ukraine [15]. The developed applied computer programs make it possible to collect information about the change in heat flows and the mass of the solution during the evaporation.

The study was carried out in isothermal mode at 40, 60 and 80 °C with an initial concentration of fructose in solutions of 12.5 wt. %. Chemically pure fructose and distilled water were used to prepare the solutions. The solutions were sterilized by boiling to prevent the possible development of microbiological activity. Samples weighing ~1.1 g were placed into the measuring cell of the calorimeter using a syringe–dispenser. The thickness of the layer of the solution was ~1 mm. The remainder of the solution in the calorimeter cell was carefully checked after each experiment. Needle-shaped crystals of fructose were always detected in it. The studies were carried out at least three times at the indicated temperatures.

After the thermal equilibrium was established in the calorimetric unit, synchronous registration of the heat spent on water evaporation and the mass of the sample was started. Measurement was stopped after the differential heat flow decreased to the resolution level and no change in the mass of the sample. The current values of the specific heat of evaporation r were determined in the process of dehydration of the solution from changes in the mass of the sample m and the differential heat flow q . The calculations were performed according to the algorithm:

$$r_i = \int_{\tau_{i-1}}^{\tau_{i+1}} q(\tau) d(\tau) / (m(\tau_{i-1}) - m(\tau_{i+1}))$$

The calorimeter was calibrated according to the requirements set out in [16] by comparing the obtained values of the heat of evaporation of the reference substance with the recommended values [17]. Distilled water for injections produced by the pharmaceutical company “Darnitsa” was used as a reference substance.

The mathematically expected relative error in determining the specific heat of evaporation of water in the temperature range of 40 – 80 °C was 0.49 %, and the maximum possible error was 1.09 %.

Results of measurements and discussion of the obtained results. The experimental values of the specific heat of evaporation of water r are represented by the reduced specific heat of evaporation as a dependence of the dimensionless parameter $R = r/r_{tab}$ on the concentration of solution C , where r_{tab} is the specific heat of evaporation from the free surface of pure water (Fig. 3).

The thermograms presented in Fig. 3 reflect the thermal processes occurring in a fructose solution during isothermal water removal. Two periods are clearly distinguished on the curves. The first period is purely endothermic. It occupies the concentration interval from the beginning of dehydration to the maximum heat absorption. The second is a reflection of simultaneous running of processes of water evaporation and spontaneous crystallization of fructose. The heat of spontaneous crystallization is generated in a short time and has a capacity greater than the endothermic heat consumption for dehydration. Therefore, the thermogram curves have a maximum after reaching which heat of evaporation it is almost impossible to estimate.

The first period is characterized by an almost linear increase in the heat of dehydration. The specific heat of evaporation is close to the tabular values ($R \approx 1$) only at the beginning of dehydration. The degree of hydration in the initial solution is ~12.5 mol water/mol fructose. As dehydration, the specific heat of evaporation increases monotonically until the solution reaches a concentration of ~65 %. In this case, the hydration number reaches ~5, that is, it becomes equal to the coordination number of fructose hydration [12]. Further concentration of the solution leads to a steeper increase in the specific heat of dehydration, which reaches an increase of 6 – 7 % by the time crystallization begins. That is, the removal of water from the hydrated coordination sphere of fructose occurs with large increasing specific heat consumption.

Taking into account that the degree of hydration is higher in solutions of lower concentration than in concentrated ones, it was possible to expect higher values of the specific heat of evaporation at the initial stage of dehydration. The obtained experimental fact was registered and described earlier in [14] and found an explanation in the following assumption. The binding energy between water molecules and the hydroxyl groups of sugar (strongly bound hydrated water) is higher than that of the bonds formed in solution at some distance from the sugar molecule (weakly bound hydrated water). This assumption arises as an analogy with the existing idea of water weakly and strongly bounded to the hydrophilic surface. It agrees with the information about the existence of an ordered first

coordination layer in the range of 0.18 – 0.35 nm and a disordered second hydration shell at a distance exceeding 0.4 nm [6, 18].

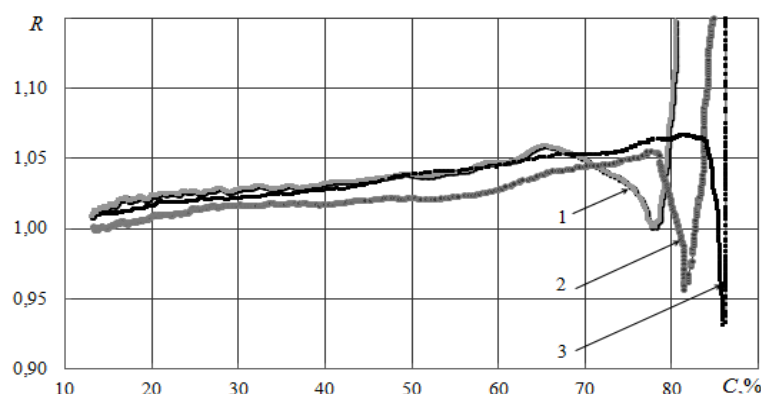


Fig. 3 – Curves of dependence of the reduced specific heat of evaporation of water from D-fructose solutions on the concentration at 40 (1), 60 (2) and 80 °C (3)

This assumption explains the steeper rise in the parameter R after the fructose solution reaches a concentration of ~65 wt. %. The degree of hydration of fructose becomes < 5 in excess of this concentration. Dehydration occurs when there is no free water in the solution. Hydrated water, which is strongly bound to fructose molecules, is removed.

Judging by the thermograms (Fig. 3), the degree of fructose hydration, which can be calculated from the data of the total concentration of solutions in the calorimeter cell (70.0 % at 40 °C, 82.4 % at 60 °C, and 87.5 % at 80 °C), is 4.3, 2.1 and 1.4 respectively at the maximum points. Comparing the obtained data with the solubility of fructose 84.34 % (40 °C), 89.16 % (60 °C) and 92.46 % (80 °C) [19] and the degree of its hydration 1.86, 1.21 and 0.81 respectively, it can be concluded that the nucleation of crystals does not occur in the entire volume of the solution. That is, the concentration of the solution does not correspond to the actual concentration in the crystallization zone at the points on the curves where the inflection is fixed (transition from heat absorption to heat release).

Evaporation of water is carried out from the surface of the solution. The diffusion of water into the surface layer is complicated by the high viscosity [20]. The gradient of fructose concentration occurs along the height of the solution. This leads to the appearance of supersaturation in a thin surface layer and involuntary spontaneous crystallization. As can be seen from the thermograms, the kinetics of crystallization depends on temperature. The crystallization rate increases slowly at 40 °C due to the higher viscosity of the solution compared to the ballistic crystallization processes at 60 and 80 °C. Reaching the maximum heat release, the curves are sharply directed upwards. Such course of thermograms in the crystallization zone is due to the closure of the evaporation surface with a layer of the solid phase of fructose. During this period, heat generation still continues, but the intensity of evaporation decreases. And when the change in the mass of the sample reaches values less than the sensitivity of the balance, the calculated value of the specific heat of evaporation increases sharply, which is represented as an ascending curve of the parameter R (Fig. 3).

Conclusions. Changes in the specific heat of evaporation of water from fructose solutions were studied in isothermal mode at 40, 60 and 80 °C during continuous dehydration by calorimetry.

The influence of the degree of hydration of fructose on the specific heat of evaporation is revealed. It is shown that the measured specific heat of evaporation of water increases and reaches the excess over the tabular values of the heat of evaporation from the free surface of pure volumetric water by 6 – 7 % despite the decrease in the degree of hydration of fructose with increasing concentration of solutions.

Water has a strong hydrogen bond with fructose in solutions in which the degree of hydration of fructose is less than ~5 mol water/mol fructose. In solutions containing free water, hydrated water outside the first coordination sphere is bound to fructose by less strong bonds, the energy of which decreases as the solution is diluted.

Taking into account the concentration dependence of the degree of hydration of glucose, which is almost the same as that of fructose, it can be assumed that glucose solutions will have the same dependence of the specific heat of evaporation of water on concentration, as in fructose solutions.

The dependence of the specific heat of evaporation of water from solutions of fructose and sucrose [14] on the concentration makes it necessary to take them into account in the existing methods of thermal calculation of drying processes of sugar-containing raw materials and corresponding equipment.

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ДОСЛІДЖЕННЯ ТЕПЛОТИ ЗНЕВОДНЕННЯ РОЗЧИНУ D–ФРУКТОЗИ

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Анотація. Теплота випаровування води є основною складовою витрат при сушінні біосировини. Доля її в витратах залежить не тільки від вихідної вологості об'єкту сушіння. Як показали наші попередні дослідження, на загальні витрати теплоти впливає стан води в матеріалі. А стан води визначається його хімічним складом та структурою. Особливу увагу заслуговують об'єкти сушіння з багатим вмістом цукрів, які, маючи високу схильність до гідратації, зосереджені в соку і визначають як кінетику так і теплоту зневоднення. Проаналізовано та наведено найбільш надійні дані щодо гідратації глюкози та фруктози. В калориметрі випаровування здійснено вимірювання питомої теплоти випаровування води r з розчинів фруктози з початковою концентрацією 12.5 мас.%. В процесі вимірювання було забезпечено безперервну реєстрацію зміни теплових потоків та маси зразка. Отримано залежності приведеної питомої теплоти випаровування води ($R = r/r_{lab}$) від концентрації фруктози в ізотермічному режимі за 40, 60 та 80 °C. Представлені криві відображають теплові процеси, що відбуваються в розчині при видаленні води. Процеси характе-

ризуються ендотермічним періодом зневоднення та екзотермічним періодом одночасного протікання зневоднення та спонтанної гомогенної кристалізації фруктози. Встановлено, що зміна ступеня гідратації фруктози в розчині від 12.5 до 5 моль/моль відбувається зі зростаючими, практично лінійно, питомими витратами теплоти. Видалення води з першої координаційної сфери, коли ступінь гідратації зменшується до значень <5 , зростання питомої теплоти випаровування є більш крутим і досягає перевищення над табличними значеннями теплоти випаровування чистої води r_{tab} на 6 – 7 %. Теплота спонтанної кристалізації фруктози має потужність більшу за потужність ендотермічних витрат теплоти на зневоднення тому реєструється загальний екзотермічний ефект. Показано, що незважаючи на зменшення ступеня гідратації фруктози, при збільшенні концентрації розчинів, виміряна питома теплота випаровування води зростає. Встановлено, що у розчинах, в яких ступінь гідратації фруктози менша за ~ 5 , вода має сильний водневий зв'язок з цукром. Обґрунтовано припущення про сильно і слабо зв'язану гідратну воду в розчинах моноцукрів.

Ключові слова: теплота випаровування, розчин, глюкоза, фруктоза, гідратація, вільна та гідратна вода.

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