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STUDY OF DIFFUSION PROCESSES IN PUMPKIN PARTICLES DURING CANDIED FRUITS PRODUCTION

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Abstract. The production of candied fruits is a priority development area of the food industry. The basic process in candied fruits production is diffusion of sugar syrup into vegetable raw material. Kinetics of the diffusion processes depends on sucrose concentration, medium temperature, particles size and internal structure of the fruits.

The experiments to determine the factors influencing the diffusion processes were carried out using the installation designed by the authors; the experimental dependences of sucrose concentration change in pumpkin candied fruits on time have been determined at temperatures of 20, 40, 60 and 80°C. Cell sizes and diameter of pores between the cells in raw and blanched pumpkin have been determined. This makes it possible to determine the internal porosity of the pumpkin particle, the value of which determines the coefficient of mass transfer.

On the basis of the experiments we derived the dependence, allowing to determine the mass transfer coefficients for sucrose molecules in blanched pumpkin fruits within the temperatures corresponding to the quality of the finished product. The experimental studies and the chosen mathematical model allow us to calculate the change in sucrose concentration in the pumpkin fruits in time at different temperatures and to determine the time required for the candied fruits to reach the equilibrium concentration. Also, the coefficients not depending on temperature, but depending only on the shape of the particles being saturated with sugar syrup, have been determined using the kinetic model of diffusion processes. The obtained theoretical dependences are in good agreement with the experimental data and substantiate the expediency of the chosen temperature and concentration ranges. The designed installation, the obtained experimental and theoretical dependences, and the calculated coefficients allow to create an intensive sucrose diffusion process in pumpkin particles during pumpkin candied fruits production.

Key words: diffusion, temperature, internal surface, pumpkin fruit, sucrose, kinetics, mass transfer coefficient.

ДОСЛІДЖЕННЯ ПРОЦЕСІВ ДИФУЗІЇ В ЧАСТИНКАХ ПЛОДУ ГАРБУЗА У ВИРОБНИЦТВІ ЦУКАТІВ

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Анотація. Для визначення чинників, що впливають на дифузійні процеси на спроектованій авторами установці проведено експерименти та наведено експериментальні залежності зміни концентрації сахарози в цукатах гарбуза в часі за температур 20°C; 40°C; 60°C; 80°C. Також визначені розміри клітин та діаметр пор між клітинами в плодах сирого та бланшованого гарбуза. Це дає можливість визначити внутрішню пористість частинки гарбуза, значення якої визначає коефіцієнт масопровідності.

На основі експериментальних досліджень виведено залежність, яка дає можливість визначити коефіцієнти масопровідності молекул сахарози у бланшованих плодах гарбуза в межах температур, які відповідають якості готового продукту. Експериментальні дослідження та обрана математична модель дають змогу розрахувати зміну концентрації сахарози в плоді гарбуза в часі за різних температур та знайти час досягнення цукатами рівноважної концентрації. Також за допомогою моделі кінетики дифузійних процесів визначені коефіцієнти, які не залежать від температури, а залежать лише від форми частинки, що насичується цукровим сиропом.

Ключові слова: дифузія, температура, внутрішня поверхня, плід гарбуза, сахароза, кінетика, коефіцієнт масопровідності.



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Introduction. Formulation of the problem

Today in Ukraine there is an increasing interest in the production of candied fruits from useful, widely available and cheap domestic raw materials such as pumpkin. The production of candied fruits from pumpkin should be organized in such a way that the substances that are useful for the human body have

been maximally preserved in the finished product. For this purpose it is necessary to improve the processes and production equipment.

Improvement of the candied fruits quality occurs at the stage of studying the influence of various factors on the diffusion process of sugar syrup into vegetable raw material. This material is a capillary-porous matter with a complex structure. That is why the value of the

coefficients of sucrose diffusion in the plant material and proceeding of the diffusion process on the whole will be influenced by the temperature of the sugar syrup, as well as the shape, size and internal structure of the raw material.

Analysis of recent research and publications

G. Giraldo et al. [1] studied the dehydration kinetics of soluble solids leaching from candied mango fruits. Dehydration was carried out under vacuum at 10°C for 8 hours using 25–65% solutions of sucrose in fruits. The sucrose concentration of 45% was found to be the highest content of the dissolved substance in equilibrium state.

T. Nepochatykh [2] considered theoretical and practical approaches to the production of candied fruit based on vegetable material. According to [2], the quality of candied fruits increases at the stages of studying the influence of various factors on the sugar syrup diffusion into vegetable raw material. This raw material is a capillary-porous matter with different forms of bonding moisture, which influences the cooking time. The reason for this is the different forms of sugar syrup diffusion: molecular diffusion, accompanied by volumetric (molar) movement of sugar syrup and selective diffusion, caused by osmotic phenomena.

The most suitable raw material for the production of candied fruits should have the maximum coefficient of the molecular diffusion and the minimum value of the selective one.

The aim of the work [3] was to investigate the sorption properties of candied orange peel.

Candied orange peels were kept in sucrose and glucose-fructose syrups. Sorption isotherms of the candied orange peel corresponded to the isotherms of type III, according to the Brunauer classification; whereas the sorption isotherms of the native orange peel had a sigmoid form characteristic of the isotherms of type II. For the products with high sucrose content (above 40%), the sorption isotherms showed a gap in the range of water activity of 0.3–0.4. The sorption properties of the studied products depend on the level of individual sugars presented in them. The analysis of the main components showed significant differences between the candied and native peels and between products candied in sucrose and glucose-fructose syrups.

The purpose of the investigations described in [4] was to make candied Malay apple (pomerec) with a long shelf life. Ripe fruits were washed with water (0.5% chlorinated solution) and cut into 2 and 3 cm thick slices. The slices were permeated with sucrose solutions (50 and 60° according to the Brix scale) and dried to A_w 0.65. Products were packed in low density transparent polyethylene bags and stored at 5°C for four weeks, during which the physico-chemical properties were constant. Sensory and microbiological

analyses were performed. Fresh pomerec contained moisture (91.3%) and ascorbic acid (0.06%). Humidity of candied pomerec slices varied from 56.9 to 60.1%, whereas the content of ascorbic acid ranged from 0.0185 to 0.0203%. The resulting candied slices (0.5–1.8 mm) were analyzed depending on different drying temperatures, sugar concentrations, and the thickness of the slice. The color, taste and texture of the candied Malay apples have been taken in accordance with the standards.

I. Belenkaya and Ya. Golinskaya [5] considered the technological aspects of candied fruits production from non-traditional raw materials. They analyzed the confectionery market in Ukraine and described the main technological operations associated with the processing of non-traditional candied products – celery and parsnip roots. To improve diffusion and osmotic processes in order to soften the roots, the fruits were kept in steam before boiling in sugar syrup. The constructed Gantt diagram proved that the developed technology could reduce the period of candied fruits cooking. The biochemical parameters of the new products have been studied. The results confirmed that candied fruits had corresponding physico-chemical parameters and original organoleptic properties leading to the growth of demand.

W. Buntaran et al. [6] studied the effect of sugar various concentrations in syrup on the characteristics of dried tomatoes (*Lycopersicon Esculentum*). They determined the optimal concentration, which would have the most positive effect on the quality of candied tomato. For this purpose the tomato was immersed in sugar solution A (40%), B (50%), C (60%) and D (70%), and kept for 18 hours. Then the content of water, ash, vitamin C, as well as organoleptic properties, such as taste, color and texture were determined. The data were analyzed using the ANOVA (Dispersion Analysis) test followed by the DMRT (Duncan Multiple Test Range). The results confirmed the effect of sugar concentration on the above-mentioned properties. The best results were found for candied tomatoes, which was kept in syrup A (40%). Their composition was: water content 24.20%, ash content 0.62%, content of vitamin C 31.15 mg/100g. The maximum permissible water content for candied fruits is 25% (SII No.0718-2003) and maximum permissible content of ash for food materials is 1.0% (SII 0272.90). The content of vitamin C did not essentially decrease compared to the ripe tomatoes, i.e. 30–40 mg/100g. The organoleptic analysis showed the high score for this product: 3.98 for taste, 3.89 for aroma, and 3.98 for color.

A. Abraão et al. [7] investigated the effect of the dehydration osmotic parameters on the efficiency of water loss and the increase of sucrose in pumpkins. Concentrations of water and sucrose affect the quality of the final product and are important for the production of candied pumpkins of the highest quality. The kinetics of mass transfer during osmotic

dehydration of pumpkins was modeled on the assumption of Fick's diffusion of sucrose and water under non-stationary conditions. They were in good agreement with experimental results for water loss and sugar increase. The effective diffusion coefficients of water and sucrose considerably increase with the increasing temperature. The temperature and concentration of sucrose affects both the diffusion of water and sugar. A significant increase in the effective diffusion of water and reduction of sucrose effective diffusion were observed at sucrose concentration of above 40° according to the Brix scale. Candied pumpkins with higher sucrose content have a higher tensile strength, a lower breaking load and fracture energy. Moreover, the increase in sucrose content resulted in the growth of weight and reduction of elasticity and thus the product became more breakable.

Taking into account literature data it is obvious that the concentration of sugar syrup during the production of candied fruits significantly affects their quality, shelf life and organoleptic properties. It has been proved that the most optimal concentration of sucrose in syrup varies within 40–70%. At higher concentrations the product becomes breakable and harder. In addition, during the drying of such candied fruits sugar particles appear on their surface, which worsens both the nutritional value of the product and its appearance. It has been established that desorption properties of water (removal of water molecules from candied fruits during their saturation with syrup) show their best qualities at the sugar concentration 40% in syrup. The raw material treatment with steam or hot water increases the diffusion coefficient in the system "syrup–candied fruit". The temperature and concentration of sucrose affects both the diffusion of water and sugar.

Thus, it can be concluded that diffusion processes are the basic ones during candied fruits production, the rate of diffusion depends on external factors (temperature, pressure, syrup concentration), and the nature of vegetable material. However, there are no sufficient data about the effect of the external structure, the shape of the particles and the internal structure of the fruits from which candied fruits are produced. The mass transfer coefficients of sucrose in pumpkin fruits, which depend primarily on the kind, form and internal structure of the fruits are also absent in literature. The values of mass transfer coefficients allow to determine the saturation time.

The **purpose** of the work were: to study the effect of temperature on the diffusion process of sugar syrup into pumpkin fruits; to analyze the internal structure of raw and blanched fruits; to determine the diffusion and mass transfer coefficients of sucrose molecules in porous media of candied fruits; to choose the mathematical model, which allows to determine the saturation time of pumpkin particles with sugar.

To achieve these aims, the following **tasks** must be solved:

1. To investigate the change of sucrose concentration in pumpkin fruits at different temperatures.

2. To obtain analytical dependences for calculating the mass transfer coefficients of sucrose in pumpkin fruits at different temperatures.

3. To summarize the results of diffusion processes study on the basis of the chosen mathematical model. To compare experimental and calculated values.

4. To determine the coefficients which depend on the particles shape and can be used to calculate the saturation time of pumpkin particles with sugar at different temperatures and concentrations.

Research Materials and Methods

Investigated materials and equipment used for the experiment. The object of the research was particles of identical sizes of cubic shape (10 mm wide) from the pumpkin fruits of the "Stofunvotka Vassma" kind grown in the western regions of Ukraine. Pumpkin particles were saturated with sugar in syrup with sugar initial concentration of 70 wt.% using the installation, the scheme of which is shown in Fig. 1

The sugar syrup, heated to a given temperature, is loaded to an enameled cylinder body 1 through the inlet 2. The compressed air is supplied with a temperature equal to the temperature of the syrup through the pipe 3 in the cylinder. The air is fed by a fan 4, heated by a heater 5 and taken out of the system through the outlet 6. When the device 7 fixes the given temperature by means of the thermocouple, particles of blanched pumpkin are loaded into the enameled body 1 through the inlet 2, providing a pneumatic intensive mixing of the particles with sugar syrup in the cylinder. When the particles are saturated with sugar to the required concentration, the supply of air is stopped, and the syrup is poured out through the outlet 8. To unload the finished candied fruits, it is necessary to open the lid 9.

The temperature is recorded by an eight-channel thermoelectric converter PT-108. Slice of the pumpkin fruit was analyzed under a microscope.

Method of conducting experimental studies of the influence of temperature on the change in the concentration of sugar in syrup and in the fruits of pumpkin. Preparation of raw materials was carried out as follows: the pumpkin was divided into parts, the peel and inner skin were removed, and then the pumpkin was cut into cubes of 10x10x10 mm size. After the sugar content was determined the fruit was blanched for 10 minutes. The content of sugar was determined again.

The syrup was prepared in the following way: 700 g of crystalline sugar and 300 CM³ of water were mixed and heated to 75°C until complete dilution. Thus, the concentration of sugar in the syrup was 70 wt.%. The syrup

weight and the content of sugar in syrup were determined. The syrup was cooled to the required temperature, and then the weight of syrup and the content of sugar in it were determined once more time.

The next step: all pre-weighed cubes of blanched pumpkin were loaded into syrup. The ratio fruit/syrup was 1:5 (w/w). So, the syrup concentration changed slightly. Every 30 minutes the samples were withdrawn.

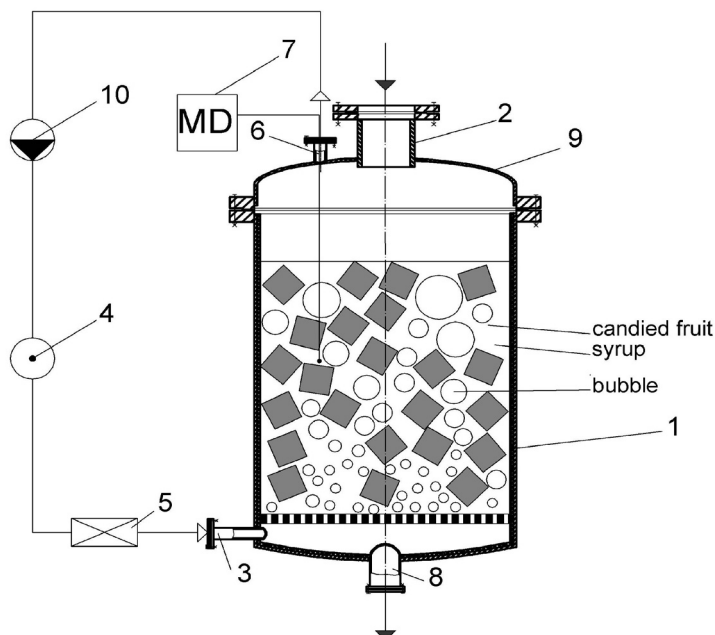


Fig.1 Scheme of installation for saturation of pumpkin particles with sugar

1 – cylinder body; 2 – inlet for supplying sugar syrup and pumpkin particles; 3 – the supply pipe of compressed air; 4 – fan; 5 – heater; 6 – air outlet pipe; 7 – a device for measuring temperature; 8 – outlet for syrup; 9 – lid; 10 – entrainment separator

The research was conducted at temperatures of 20, 40, 60 and 80°C. The temperature range was selected to achieve the high quality of the finished product.

The content of sugar in the fruits was determined by refractometry according to the procedure described in [8] using IRF-454 B2M refractometer. A slice of pumpkin fruit with a thickness of 500 microns was analyzed under a microscope.

Results of the research and their discussion

Dependence of the change in the sucrose concentration in candied fruits at 20, 40, 60 and 80°C is shown in Fig.2.

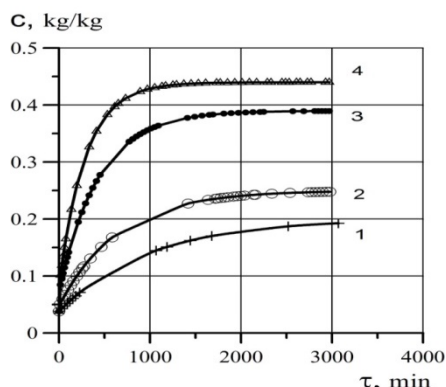


Fig. 2. Dependence of change in the sucrose concentration in candied pumpkin fruits on time at

the temperatures 1 – 20°C; 2– 40°C; 3 – 60°C; 4 – 80°C

It is evident from Fig. 2 that during keeping candied fruits in syrup, the concentration of sucrose in candied fruits increases with increasing temperature.

So at the temperature of 20°C the saturation time till the equilibrium state is 3000 minutes (Fig. 2, curve 1), at 40°C – 2500 minutes (Fig. 2, curve 2), at 60°C – 1500 minutes (Fig. 2, curve 3), and at 80°C – 1000 minutes (Fig. 2, curve 4).

At the temperatures of 60 and 80°C the equilibrium concentration in candied parts is achieved rather fast compared with that at 20 and 40°C. This phenomenon is explained by the fact that when a pumpkin is heated to 60 °C, a protoplasm (protein) surrounding the cell vacuole is not destroyed and badly transmits sucrose molecules and other dissolved substances. At the temperatures above 60°C protoplasm is destroyed, which contributes to a significant increase in the diffusion rate.

The general view of raw and blanched pumpkin cells is shown in Figs. 3a and 3b, respectively. Raw pumpkin cells are almost square, pores are clearly defined. After blanching the pumpkin cells are destroyed and the pores are much wider, which will contribute to a better diffusion of sucrose molecules to the middle of the pumpkin particle.

From the picture of blanched pumpkin (Fig. 3b), the average diameter of the pores between the pumpkin

cells ($d=280 \mu\text{m}$) and the average cross-sectional area

of pumpkin cells ($250,000 \mu\text{m}^2$) were determined.

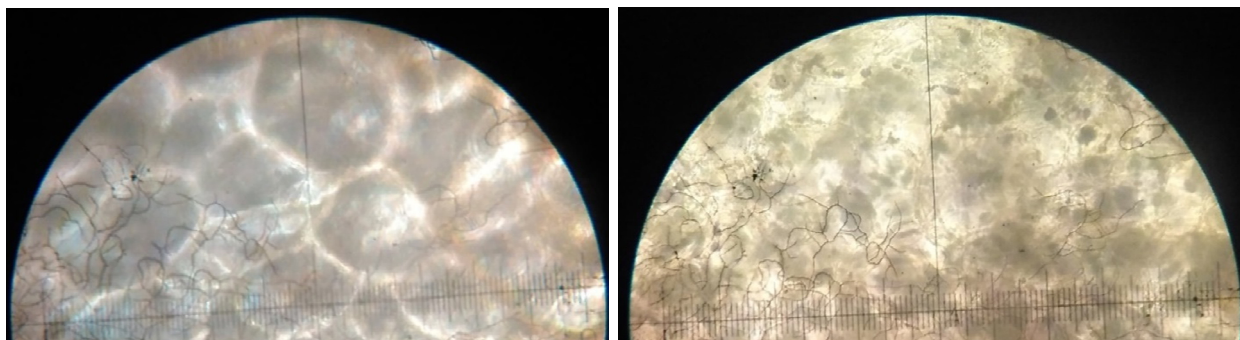


Fig. 3. Photos of the raw (a) and blanched (b) pumpkin cells

We propose to determine the coefficients depending on the particles shape and to calculate the diffusion and mass transfer coefficients of sucrose particles in the porous medium of candied fruits.

The diffusion coefficient depends on the concentration, temperature, nature and shape of the particle. The diffusion coefficients were found based on experimental data (Fig. 2), within a certain range of concentrations.

Well-known Einstein's equation (1) determines the diffusion coefficient of large circular molecules in a liquid [2]:

$$D_c = \frac{k \cdot T}{6 \cdot \pi \cdot \mu \cdot r} \quad (1)$$

where k is Boltzmann constant, J/K; T is an absolute temperature, K; r is the radius of diffusing particles, m; μ is the viscosity of the liquid medium, Pa·sec

The radius of diffusing particles is the radius of the sucrose molecule.

Taking into account that D_c is the coefficient of isothermal diffusion in a liquid continuum, in porous bodies, a coefficient of mass transfer D_m is used instead of D_c .

As a rule, the coefficient D_m is less than the diffusion coefficient D_c . The first reason – the mass transfer area is less compared with that for free diffusion. The ratio of these areas is equal to the matter porosity m . The second reason is the capillary structure of the porous matter. The movement of substance molecules in a capillary, the diameter of which is comparable to the size of diffusing particles, will be slower than that in an infinite liquid medium. This is due to the inhibitory effect of the capillary walls. In our case the size of the cross-area of such capillaries is steadily varied.

On the one hand, the mass transfer coefficient is proportional to the diffusion coefficient, and on the other hand, it depends on structural factors: porosity, capillary geometry, the ratio between the molecules size and the capillary radius.

Thus, the mass transfer coefficient will be equal to

$$D_m = m \cdot D_c \quad (2)$$

where m is the porosity m^2/m^2

The porosity is determined from the photograph of the sample slice (Fig. 3), $m=0.65 \text{ m}^2/\text{m}^2$.

The mass transfer coefficient depends on the medium temperature, the nature of the diffusing molecules, and the nature of porous matter.

Fig. 4 shows the dependence of the mass transfer coefficient determined from equation (2) on the experimental temperature of the medium. The obtained points were approximated by exponential dependence. The approximation was carried out using Grapher 10 software. As a result of the approximation, the equation (3) is obtained:

$$D_{m_T} = 2.2 \cdot 10^{-3} \cdot D_{m_{293}} \cdot e^{0.02T} \quad (3)$$

Equation (3) allows to determine the mass transfer coefficients of sucrose molecules in blanched pumpkin fruits at the temperature corresponding to the quality of the finished product. The values of the coefficients in Eq. (3) depend only on the fruit kind and its internal structure.

The obtained Eq. (3) is in good agreement with the experimentally determined coefficients of mass transfer, and the maximum relative error as a result of approximation by the exponential dependence does not exceed 5%.

The mass transfer coefficient, the shape and size of the porous matter particles affect the saturation time.

The kinetics of diffusion processes is described by the Fick's law [9, 10]:

$$\frac{\partial c_2}{\partial \tau} = D_m \cdot \left(\frac{\partial^2 c_2}{\partial x^2} + \frac{\partial^2 c_2}{\partial y^2} + \frac{\partial^2 c_2}{\partial z^2} \right) \quad (4)$$

Let us add to the differential equation (4) the boundary conditions: $\tau = 0$; $c_2 = c_0 = \text{const}$, $c_{2II} = 0$.

Then Eq. (4) takes form of equation (5):

$$\frac{c_2}{c_0} = A \cdot e^{-s \cdot \frac{D_m \cdot \tau}{R^2}} \quad (5)$$

where R is characteristic size; c_2 is running concentration; A , s is coefficients depending on the particle shape [11].

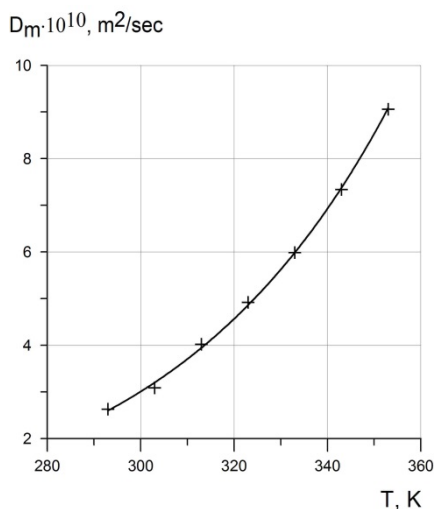


Fig. 4. Dependence of the mass transfer coefficient of sucrose molecules in blanched pumpkin fruits on the medium temperature.

To summarize the results of research on the basis of the mathematical model (5) is possible under conditions implemented in the experiment:

1) Porous matter should have an isotropic structure (for the plate-like form the anisotropy associated with the presence of capillary channels, the axes of which are perpendicular to the plate area, is admissible);

2) The mixture of particles should be monodispersed;

3) The mixing of two-phase system (porous particles-liquid) should be intense.

4) The volume of liquid used for extraction (absorption) should be larger than the pore volume, so that the liquid concentration only slightly increases (decreases).

The latter condition is sometimes difficult to fulfill, therefore, we obtained the equation (6) that determines the extraction kinetics under the conditions of systematic increase of the substance (c) concentration in the porous matter.

$$1 - \frac{c}{c_p} = \sum_{n=1}^{\infty} A_n \cdot e^{-s_n \cdot \frac{D_m \cdot \tau}{R^2}} \quad (6)$$

where c_p is equilibrium concentration.

It is important to determine the coefficients A_n and s_n . Under the experimental conditions all particles were of the same size and the same cubic shape ($n=1$). The process of sucrose diffusion in the porous matter of the pumpkin fruit particles occurs through the surface in the volume of cubic particles. The mass transfer takes place in the volume, the rate of which is limited by the mass transfer coefficient D_m . Thus, the coefficient A depends on the shape of the particle

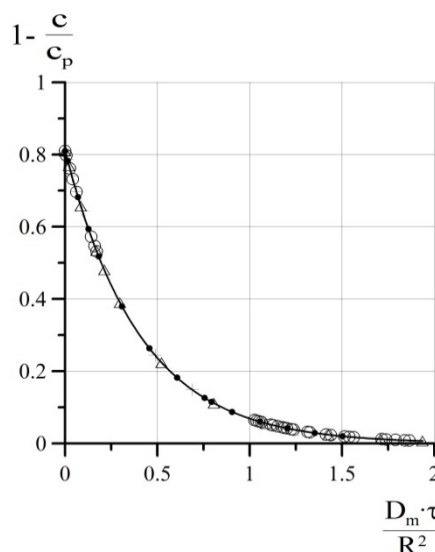


Fig. 5. Summarizing curve of experimental data regarding the change of sucrose concentration in candied pumpkin fruits in time at 20, 40, 60 and 80 °C

external surface, and the coefficient s - on the volume form.

Let us assume that the characteristic size R is the equivalent radius of a cubic particle, which is calculated according to the dependencies:

$$a^3 = \frac{\pi \cdot 8 \cdot R^3}{6} \quad (7)$$

$$6 \cdot a^2 = 4 \cdot \pi \cdot R^2 \quad (8)$$

where a is the cube side, according to the experimental conditions.

On the basis of equations (6–8) we obtain the values of coefficients which depend not on the particle size, but only on its shape. For the particles of cubic shape:

$$A = \sqrt{\frac{6}{4 \cdot \pi}} = 0.69 \quad s = 4 \cdot \left(\frac{\pi}{6}\right)^{2/3} = 2.6$$

Using equation (6) let us generalize the obtained results. The dependence of concentration changes on dimensionless time is plotted in Fig. 5. All experimental points within the temperature range of 20–80°C are generalized by one curve. As a result of the approximation carried out with the Grapher 10 software, an analytical exponential dependence has been obtained:

$$1 - \frac{c}{c_p} = 0.8 \cdot e^{-2.5 \cdot \frac{D_m \cdot \tau}{R^2}} \quad (9)$$

The experimental values of the coefficients A and s coincide with the calculated ones; error is 3-10%. It means that the mathematical model we have chosen is adequate within the temperature range of 20–80 °C and concentration range of 2–45 wt.%.

On the basis of the determined coefficients A and s, as well as equations (3) and (9), we derive an analytical dependence:

$$1 - \frac{c}{c_p} = 0.69 \cdot e^{-2.6 \frac{2.2 \cdot 10^{-3} \cdot D_{m293} \cdot e^{0.02T}}{R^2} \tau} \quad (10)$$

Dependence (10) makes it possible to determine the time of cubic pumpkin particles saturation with sugar at the temperatures and concentrations of syrup, corresponding to the quality of the finished product.

Conclusions

- Changes in the concentration of sugar syrup in pumpkin fruits at 20, 40, 60 and 80°C have been investigated.
- Cell size and pore diameter between cells in pumpkin fruits have been determined.
- The dependence (3) was obtained, allowing to determine the mass transfer coefficients of sucrose molecules in blanched pumpkin fruits at the temperatures corresponding to the quality of the finished product.
- On the basis of the chosen mathematical model we determined the coefficients A=0.69, s=2.6, which depend not on the temperature, but only on the particle shape.
- On the basis of generalized experimental data, an analytical dependence (10) was obtained, allowing to determine the saturation time of pumpkin particles and to determine equilibrium concentrations under experimental conditions.
- The obtained theoretical dependences are in good agreement with the experimental data and substantiate the expediency of the chosen temperature and concentration ranges.

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ИССЛЕДОВАНИЕ ДИФFUЗНЫХ ПРОЦЕССОВ В ЧАСТИЦАХ ПЛОДОВ ТЫКВЫ ПРИ ПРОИЗВОДСТВЕ ЦУКАТОВ

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Аннотация. Для определения факторов, влияющих на диффузные процессы, проведены эксперименты на спроектированной авторами установке и приведены экспериментальные зависимости изменения концентрации сахарозы в цукатах тыквы во времени при температурах 20°C; 40°C; 60°C; 80°C. Также определены размеры клеток и диаметр пор между клетками в плодах сырой и бланшированной тыквы. Это даёт возможность определить внутреннюю пористость частицы тыквы, значение которой определяет коэффициент массопроводности.

На основе экспериментальных исследований выведена зависимость, которая дает возможность определить коэффициенты массопроводности молекул сахарозы в бланшированных плодах тыквы в пределах температур, которые соответствуют качеству готового продукта. Экспериментальные исследования и выбранная математическая модель позволяют рассчитать изменение концентрации сахарозы в плоде тыквы во времени при различных температурах и найти время достижения цукатами равновесной концентрации. Также с помощью модели кинетики диффузионных процессов определены коэффициенты, не зависящие от температуры, а только от формы частицы, которая насыщается сахарным сиропом.

Ключевые слова: диффузия, температура, внутренняя поверхность, плод тыквы, сахароза, кинетика, коэффициент массопроводности.

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