Kinetics of sea buckthorn juice concentration by freezing

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Abstract. Concentration of sea buckthorn juice by freezing on a batch crystallizer with plate-fin heat exchange elements having the area of 0.08 m² per cycle equal to 3600 s was studied in the work. An increase in the initial content of soluble substances in the concentrated juice with freezing reduces the specific amount of ice formed on the heat exchange surface. Increased release of frozen ice was observed at the refrigerant lower boiling point. An increase in the initial content of soluble substances in sea buckthorn juice reduces the value of the ratio of soluble substances in the concentrated juice in relation to the initial value. An increase in the refrigerant boiling point causes a decrease in the concentration level. The degree of sea buckthorn juice concentration also decreases with an increase in the content of soluble substances in the initial juice supplied for concentration by freezing.

1. Introduction

The composition of sea buckthorn juice and its berries has high nutrients content. The content of vitamins and minerals in the composition of sea buckthorn juice is perfectly balanced. It is supplemented by the presence of moderate amounts of phospholipids, phytosterols, amino acids, mono- and polyunsaturated fatty acids. Sea buckthorn juice concentrate is rich in fatty oil containing glycerides of oleic, stearic, linoleic and palmitic acids, as well as in flavonoids, folic acid, carotenoids, betaines, cholines, coumarin, glucose, fructose and phospholipids [1].

Concentration of sea buckthorn juice by freezing makes it possible to preserve these compounds in their maximum native state.

The mechanism of the process of liquid media concentrating by moisture freezing is mainly determined by the crystallization mode and the peculiarities of the connection between moisture and the material or by the conditions for the water molecules migration to the surface of the crystallization front and embedding in the ice crystal structure. Moisture freezing mode, which determines the mechanism of the process, is characterized by the medium or heat exchange surface temperature, the latest receiving the heat of moisture crystallization. Moisture freezing mode is also characterized by the speed of movement and the contact surface area of the heat exchanging media as well as by the type and concentration of dissolved substances. The process of moisture freezing is a complex phenomenon of liquid heat and mass transfer with a heat exchange surface and mass transfer inside liquid itself.

2. Materials and methods

The process of concentration by freezing of sea buckthorn juice on a batch crystallizer with plate-fin
heat exchange elements, created on the basis of the Blexmatic V41 electronic ice maker, shown in figure 1 was investigated in the work [2-5].

![Figure 1](image1.png)

**Figure 1.** Crystallizer with plate-fin heat exchange elements: a) - scheme; b) - general view; c) - top view 1 - refrigeration unit; 2 - liquid tank; 3 - liquid drainage line; 4 - plate-fin heat exchange element; 5 - cover; 6 - temperature control sensor; 7 - tilting bath; 8 - liquid supply line; 9 - counter - flow meter; 10 - pump; 11 - dashboard; 12 - evaporator pin.

The crystallizer consists of a refrigeration unit 1 with a refrigerant supply and removal system, which ensures the achievement of the required boiling point of the refrigerant in the pins 12 of the evaporator, a bath 7, a liquid tank 2. The surface of moisture freezing is made in the form of pins 12 of the evaporator with plate-fin heat exchange elements 4 immersed in the bath 7 with liquid. The heat exchange elements are galvanized metal plates of 30×10×0.1 mm, soldered to the cylindrical half-ring on three sides. The angle between them is 120 degrees. Heat exchange elements are fixed on vertical cylindrical pins 12 of evaporators with a diameter of 8 mm, the number of which in the unit is 100 pcs.

The thermocouple was used to measure the temperature of the evaporator pins with a soldering diameter of 0.2 mm. The connecting end of the thermocouple was joined to the input connector of the TRM202 instrument, measuring range being 73 ... 1073 K and a division value of 0.1 K. The thermocouple was periodically checked for accuracy.

The tilting bath 7, made of 0.2 mm stainless steel sheet, was thermally insulated. When concentrating the liquid, the bath was in the raised position. The liquid level in the bath was measured with a measuring ruler, its scale division being 1.0 mm.

Concentration cycle duration was controlled by an electronic stopwatch.

The release of the evaporator pins from the frozen ice was carried out with the lowered tilting bath 7 and the operation of the refrigeration unit 1 in "defrost" mode.

The frozen ice mass was measured in one concentration cycle [6-8].

Frozen ice was placed in a pre-weighed flask, and the total weight of the flask with the ice was measured on an analytical balance. To determine the dry matter value of the liquid captured with the ice, the lastest was melted, thermostated at a temperature of 293 °C, and the dry matter content was determined with an IRF-22
refractometer.

The system for liquid supplying to the bath 7 consisted of a pump with the flow rate of 1200 ... 4000 dm$^3$/h and a power of 40 W, a drainage line 3 and a liquid supply line 8 with control valves and a counter-flow meter 9.

The refrigerant supply system into the cavity of the evaporator pins 12 consists of a refrigeration unit containing a piston hermetic compressor, a condenser, a receiver, a filter drier, a temperature control sensor 6, a pressure switch, and solenoid valves. Freon 134a was used as a refrigerant in a refrigeration unit. Its capacity made it possible to carry out the experiments with the boiling point of the refrigerant from 262 to 250 K by changing the compressor refrigerating capacity. The pressure in the compressor suction and discharge lines was monitored by pressure gauges on the dashboard 11.

The following experimental technique was used in the research. After an external unit inspection the refrigeration machine was turned on and the refrigerant supply valve was opened in the evaporator pipe. The refrigerant boiling point was controlled with the thermocouple. Pump 10 was turned on after reaching the required evaporator temperature. It provided the supply of liquid to the bath 7, being in a horizontal position, and the stopwatch was turned on as well. The specified liquid flow rate was controlled by a valve by preliminarily changing of the flow cross-section of the liquid supply line.

Evaporator boiling point, compressor suction and discharge pressures, flow rates and freeze cycle times were recorded during the experiment. At the same time, we measured the voltage and current in the motor circuit.

At the end of the freeze cycle time equal to 60 minutes, the pump motor was turned off, the remaining liquid was drained from the bath into the supply tank, the bath was brought to the "down" position, and the refrigeration unit was switched to the "defrost" mode. In this case, when switching the solenoid valves, hot refrigerant vapor was supplied to the evaporator pins and plate-fin elements. The ice layer in contact with them partially melted and the ice freely slipped from them into the ice collector.

After the freezing cycle, the productivity of the unit for frozen ice was calculated, the dry matter content in the concentrated liquid and the solution obtained after thawing of the frozen ice was determined [9-11].

An experimental study of the change in the average value of the amount of frozen ice from a unit of heat exchange surface of the freezing unit in time is an important characteristic of the moisture freezing process.

![Figure 2](image1.png) ![Figure 3](image2.png)

**Figure 2.** Curves of growth and ice growth rate on the surface of heat exchange elements with an area of 0.08 m$^2$, at the refrigerant boiling point of 258 K and the initial content of soluble substances in sea buckthorn juice: 1 – 12,0 %; 2 – 16,0 %; 3 – 20 %; 4 – 24 %.

**Figure 3.** Curves of growth and ice growth rate on the surface of heat exchange elements with an area of 0.08 m$^2$, at the refrigerant boiling point of 253 K and the initial content of soluble substances in sea buckthorn juice: 1 – 12,0 %; 2 – 16,0 %; 3 – 20 %; 4 – 24 %.
Studies of the growth and ice growth rate on the surface of heat exchange elements with an area of 0.08 m$^2$, for 3600 s, depending on the heat exchange surface temperature and the initial content of soluble substances in sea buckthorn juice (figure 2-5) were carried out in the work.

**Figure 4.** Curves of growth and ice growth rate on the surface of heat exchange elements with an area of 0.08 m$^2$, with an initial content of soluble substances in sea buckthorn juice of 12.5% and the refrigerant boiling point: 1 – 262 K; 2 – 257 K; 3 – 255 K; 4 – 253 K.

**Figure 5.** Curves of growth and ice growth rate on the surface of heat exchange elements with an area of 0.08 m$^2$, with an initial content of soluble substances in sea buckthorn juice of 18% and the refrigerant boiling point: 1 – 262 K; 2 – 257 K; 3 – 255 K; 4 – 253 K.

The change in the concentration degree of sea buckthorn juice during freezing from the refrigerant boiling point and the initial content of soluble substances in it (figure 6, 7) was studied in the work.

**Figure 6.** Change in the concentration degree of sea buckthorn juice depending on the initial content of soluble substances and the refrigerant boiling point: 1 – 262 K; 2 – 258 K; 3 – 255 K; 4 – 253 K.

**Figure 7.** Change in the concentration degree of sea buckthorn juice depending on the refrigerant boiling point at the initial content of soluble substances: 1 – 12.6 %; 2 – 14.1 %; 3 – 16.3 %; 4 – 17.9 %; 5 – 19.8 %.

3. Results and their discussion
Analysis of the dependences of ice growth and ice growth rate obtained by graphical differentiation showed the following. The curves of ice growth on the heat exchange surface of the freezing unit are of
a similar nature, depending on the temperature of the freezing surface and the initial content of soluble substances in the sea buckthorn juice. An increase in the initial content of soluble substances in sea buckthorn juice at the refrigerant constant boiling point in the freezing unit evaporator reduces the specific amount of ice formed on the heat exchange surface.

The boiling point of the refrigerant in the freezing unit evaporator also affects the specific amount of frozen ice. A change in the refrigerant boiling point causes a non-linear increase in the specific amount of ice frozen on the heat exchange surface as well. With a decrease in the refrigerant boiling point, the specific amount of frozen ice increases.

The ice phase growth rate curve has a complex shape. At the moment of sea buckthorn juice contact with the evaporator surface, the crystallization rate increases sharply from zero to the maximum value in a very short period of time, due to thermal "shock" at the moment of the juice contact with the elements of the freezing unit evaporator.

Then the ice crystallization rate curve changes sharply its inclination angle. An almost linear change in the direction of the moisture crystallization rate curve on the heat exchange surface to the second inflection point is observed at this moment. This indicates the formation of a thin layer of ice on the heat exchange surface and the formation of a temperature regime between the sea buckthorn juice and the heat exchange surface.

Then the character of the ice phase growth rate curve acquires a nonlinear form, after which it undergoes a second inflection point and decreases monotonically nonlinearly. This inflection point is borderline. It separates the processes of moisture ice formation, which have a different nature of connection with the dissolved substances of sea buckthorn juice. In the initial period of freezing, moisture crystallizes in the layers that are the closest to the ice formation surface in the form of sea buckthorn juice least linked with soluble substances.

Then, with the displacement of the liquid phase layers, the period of a sharp decrease in the rate of ice formation caused by the thermal resistance of the frozen ice layer on the heat exchange surface begins. In addition, the increasing forces of adhesion of water molecules with soluble substances of sea buckthorn juice have an effect on water molecules migration.

The change in the concentration degree of sea buckthorn juice is nonlinear during freezing from the initial content of soluble substances. The value of the ratio of soluble substances in the concentrated juice in relation to the initial content decreases nonlinearly with an increase in the initial content of soluble substances in the sea buckthorn juice.

The boiling point of the refrigerant in the heat exchange elements of the freezing unit also affects the change in the concentration degree of sea buckthorn juice with different content of dissolved in it substances. An increase in the boiling point of the refrigerant causes a decrease in the concentration degree under other freezing conditions [12, 13].

4. Conclusions

Investigation of the kinetic regularities of ice formation per unit of crystallizer heat transfer surface per concentration cycle showed the following. The described heat and mass transfer processes are complicated by side effects and processes that occur during concentration by freezing [14, 15].

Heat transfer occurs only on the heat exchange surface. Moisture crystallization occurs on the heat exchange surface in the apparatus only at the beginning of the freezing process, and then the heat exchange surface moves from the geometric cooling surface of the apparatus together with the crystallization front. An ice layer is formed. Its thickness is increasing. The heat removed during moisture crystallization has to overcome the thermal resistance of the boundary layer, as well as the ever increasing thermal resistance of the frozen ice layer i.e. the crystallization zone.

Heat transfer in this layer occurs only through thermal conductivity, which means that as the moisture crystallization zone forms and deepens, the intensity of ice formation as well as the freezing rate decreases.

A nonlinear decrease in the specific amount of frozen ice caused by an increase in the refrigerant boiling point and an increase in the initial content of soluble substances in the initial juice were determined in the
work. Periods of moisture crystallization with a different nature of the relationship between water and soluble juice compounds were also revealed [16].

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