The rationale for the method of thermochemical processing of beetle chips before extraction of sucrose

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Abstract. The objective of this thesis is to ensure utmost performance of the diffusion process by choosing the most effective components of chemical heat treatment of beetle chips. Aqueous solutions of ammonia sulphates \((\text{NH}_4)_2\text{SO}_4\), aluminium sulphate \(\text{Al}_2(\text{SO}_4)_3\), calcium sulphate \(\text{CaSO}_4\) have been considered as reagents to process chips. The classical diffusion process method has been used as a benchmarking option. It has been deduced from experiments that beetle tissue treatment with solutions of the proposed reagents heated up to 73 °C enables a high degree of cell plasmolysis, enhances its permeability and intensifies sucrose transfer to the extractant during heavy convectional leaching. The process is strongly enhanced if chips are treated with reagent solutions following electrochemical activation, which is confirmed by the study of the beetle tissue fine structure after its various chemical heat treatments. Rational conditions of solution electrochemical activation for beetle chips chemical heat treatment prior to extraction have been selected experimentally. 3 patents of the Russian Federation for the diffusion juice process have been obtained based on the results of the studies conducted.

1. Introduction
Sucrose is extracted from beetle chips as a result of diffusion, which is spontaneous equalization of the matter concentration at the interface of two phases mediated by heat motion of molecules [1,2]. It is commonly supposed that sucrose extraction from beetle proceeds under certain conditions (temperature of 72-75 °C) and comprises two most important stages: sucrose transfer (convective diffusion) from internal layers of chips to their surface following molecular diffusion laws and from the surface of chips to extractant (mass exchange). Mass exchange is mainly affected by molecular diffusion [3,4,5]. It has been established that the diffusion coefficient when extracting plant tissue matter is fully dependent on its structure and physical properties as well as nature of the matter being extracted [6].

One of the ways to enhance efficiency of the diffusion process in beet sugar production is thermal action on beetle chips with various heat carriers. Workmanship and technology of the diffusion department of a beet sugar factory predetermine operational efficiency of all downstream stations as well as quality and output of finished products [7,8].
The objective of this thesis is to ensure utmost performance of the diffusion process by choosing the most effective components. An important task here is to choose a chemical reagent that ensures the best technology parameters of semi-finished products.

2. Materials and methods
Beet chips obtained on a pilot plant have been used as material. Aqueous solutions of ammonia sulphates \((\text{NH}_4)_2\text{SO}_4\), aluminum sulphates \(\text{Al}_2(\text{SO}_4)_3\), calcium sulphates \(\text{CaSO}_4\) have been considered as reagents to process chips. The classical diffusion process method has been used as a benchmarking option. Sucrose was extracted in laboratory settings.

3. Results and their discussion
Diffusion with chemical heat treatment of beet chips with heating steam and solutions of the proposed saline reagents before extraction (table 1) has been studied.

Table 1. Sucrose diffusion coefficient when using various reagents

| Reagents for beet chips treatment | Diffusion coefficient \(D\), \(\text{m}^2/\text{s} \cdot 10^{-10}\) |
|----------------------------------|----------------------------------|
| Standard                         | 26.0                             |
| Calcium sulphate                 | 37.3                             |
| Aluminum sulphate                | 41.5                             |
| Ammonium sulphate                | 46.4                             |

Analysis of diffusion coefficient values allows drawing a conclusion on the positive influence of beet tissue chemical heat treatment with solutions of the proposed reagents on the coefficient. Beet tissue treatment with solutions of the proposed reagents heated up to 73 °C enables a high degree of cell plasmotosis due to intensive penetration of heated up reagent solutions inside the beet tissue, which enhances its permeability and intensifies sucrose molecule transfer from beet tissue pores to the extractant during heavy convectional leaching. Active interaction between the beet tissue and hot solutions of the reagents triggers cell thermal expansion caused by a high temperature gradient, which eventually results in caving of tissue cell membranes and their subsequent destruction. This phenomenon decreases intracellular pressure (“quasi-diffusion effect”). The highest diffusion coefficient value is attained when the beet tissue is treated with the hot solution of ammonia sulphate \((\text{NH}_4)_2\text{SO}_4\).

In order to justify feasibility of application of ECA reagent solutions for the enhancement of beet tissue permeability, aqueous solutions of ammonia and aluminium sulphate with reagent concentration of 0.05% were prepared and subjected to ECA for 60 s with electrode current density of 3 mA/cm\(^2\) and electric field intensity of 1.2 V/cm. Reagent solutions thus electroactivated were heated to 72 °C and used for beet tissue chemical heat treatment. Diffusion coefficient was used as a criterion of beet tissue permeability; it was calculated in line with the method [9] (table 2).

Table 2. Diffusion coefficient depending on the method of chemical heat treatment of chips

| The method of processing beet chips                  | Diffusion coefficient \(D\), \(\text{m}^2/\text{s} \cdot 10^{-10}\) |
|-----------------------------------------------------|----------------------------------|
| Standard                                            | 29.5                             |
| Treatment with a solution of aluminium sulphate without ECA | 41.2                             |
| Treatment with a solution of aluminium sulphate after ECA | 44.1                             |
| Treatment with a solution of ammonium sulphate without ECA | 47.6                             |
| Treatment with a solution of ammonium sulphate after ECA | 50.4                             |

Analysis of the resulting sucrose diffusion coefficient values confirms feasibility of electrochemical action on aqueous solutions of the reagents used for chemical heat treatment of chips. This processing method enables fast denaturation of the protein-pectate membrane of cell walls, which results in its reduced stability that causes destruction and fragmentation [10,11]. This brings about
elevated permeability of the beet tissue. The maximum sucrose diffusion coefficient ($K_D \cdot 10^{-10} 50.4 \text{m}^2/\text{cm}$) is observed during chemical heat treatment of the beet tissue with electroactivated solution of ammonia sulphate.

The second stage of experimental studies determined the influence of reagent solution ECA duration on the diffusion coefficient. Since better qualitative and mass exchange diffusion characteristics are attained in the presence of ammonia sulphate solution, further studies used this reagent only.

In line with the method of the second stage of tests, a series of ammonia sulphate aqueous solutions was prepared and subjected to electrochemical activation during 30, 60, 90 and 120 s with constant parameters (electrode current density of 3 mA/cm$^2$ and electric field intensity of 1.2 V/cm), after which it was heated to 72 °С and used for beet tissue chemical heat treatment. Treated beet tissue samples were placed in the pilot plant and the diffusion coefficient was determined in line with the method [12] (figure 1).

![Figure 1. Sucrose diffusion coefficient with various duration of ECA of the solution for beet tissue chemical heat treatment](image1)

Analysis of the experimental data obtained shows that the maximum effective diffusion coefficient is attained with ECA duration of 120 s. However, with this ECA duration, high values of the diffusion coefficient are associated with high power consumption. Reasonable ECA duration is 60 s.

The third stage of experimental studies addressed the influence of the heating degree of the electroactivated ammonia sulphate solution used for the chemical heat treatment of chips on the sucrose diffusion coefficient. In line with the algorithm of the third stage, ammonia sulphate aqueous solution with reagent concentration of 0.1% was prepared, subjected to ECA and subsequent heating to 60, 65, 70 and 75 °C. Heated solutions were used for chemical heat treatment of the beet tissue samples placed in the pilot plant to determine the diffusion coefficient in line with the method [12] (figure 2).

![Figure 2. Sucrose diffusion coefficient with various temperatures of the ammonia sulphate solution](image2)
It has been established that the maximum sucrose diffusion coefficient is attained with the ammonia sulphate solution ECA temperature of 75 °C. Obviously, this temperature of the solution used to treat the beet tissue causes high-speed denaturation of protein structures and its maximum permeability, which promotes favorable conditions for sucrose intensive diffusion.

Intensity of sucrose diffusive extraction is mainly determined by beet tissue properties, such as structural integrity of cells, degree of plasmolysis, deformative changes, etc. It has been established that the maximum sucrose release from beet tissue cells requires utmost denaturation of protein components of pectate and cellulose walls of cells that will ensure the required degree of beet tissue permeability. It is also important to ensure that the main barriers that hamper sucrose extraction are destroyed: cytoplasms and surrounding systems of cell membranes [13].

Studies have been conducted to analyze the influence of ECA of solutions of the saline reagents used to treat beet chips on the fine structure of beet tissue cells.

Figure 3 shows photos of the study beet tissue samples obtained with Altami Studio 3.3.0 software system with ×960-fold magnification.

![Figure 3. The microstructure of beet tissue cells under various processing conditions: a - without treatment; b - thermochemical treatment with a solution of (NH₄)₂SO₄ without ECA; c - thermochemical treatment of ECA with a solution of (NH₄)₂SO₄](image)

Figure 3 (a) shows the fine structure of the beet tissue not subjected to heat chemical action before diffusion. One can clearly see almost intact cellular system (membranes are broken only in some cells). Under these conditions, cell content does not undergo changes: cytoplasmic membrane bears again the membrane thus preventing sucrose molecules from diffusion. Permeability of this sample tissue is low, which makes sucrose extraction very difficult.

When examining the image of the fine structure of the tissue (see figure 3 (b)) treated with inactivated ammonia sulphate solution, one can clearly observe thinning of cell walls and subsequent irreversible destruction of plasma membrane – a peripheral part of cell walls. As a result of this, many pores and vesicles are formed that are penetrated by the reagent solution. Its interaction with protein-pectic complex substances results in complex formations of various shapes that settle on the beet tissue surface.

Clear illustration of influence of ammonia sulphate solution ECA on the tissue fine structure is shown in figure 3 (c). When this tissue sample is treated with steam and electroactivated ammonia sulphate solution, the degree of destruction is at its highest. These conditions trigger fast denaturation of cell wall protein components and their destruction. The cytoplasm is isolated from destroyed cell walls and is exposed to direct action of the electroactivated reagent solution, as a result of which the plasmic bag is torn into clots that aggregate to form improperly-shaped bodies. Besides, there is intensified aggregation of the cell content caused by polarization of protein and lipid components of cell membranes [14]. This results in a great deal of aggregated conglomerates that feature large size and lower mobility and are retained inside walls and, thus, do not hamper sucrose release. Results obtained are confirmed by experimental studies of the diffusion process with chemical heat treatment of chips with electroactivated reagent solutions.
Thus, electrochemical activation of solutions of the reagents used for chemical heat treatment of beet chips produces significant influence on the fine structure of beet tissue cells, as a result of which some structural and functional processes occur in cells that allow to attain high permeability of the beet tissue, which intensifies sucrose diffusion transition. When beet chips are treated with electroactivated solutions of the reagents that actively penetrate resulting vesicular pores, cell content is actively aggregated and conglomerate compounds are formed. As a result of chemical heat treatment of chips with electroactivated reagent solutions, transfer of HMC and HOA from beet tissue pores to extractant is blocked, which results in much better quality of diffusion juice.

4. Conclusion
Analytical study of the experimental data obtained has resulted in the elaboration of methods of diffusion juice production (Patents of the Russian Federation No 2553234, 255155, 2603829) with chemical heat treatment of beet chips with steam and aluminium and ammonia sulphate solutions before extraction [15,16,17].

Combination of heat and chemical treatment allows heating up beet chips to the optimal diffusion temperature of 72 °C outside of the beet diffuser. Heated chips are fed to the beet diffuser, which helps decrease the process time. Steaming period is 30-60 s. Temperature of chips after steaming is 72 °C.

Qualitative indicators of the semi-finished products obtained using the method with prior chemical heat treatment of chips before sucrose extraction are much better than those of conventionally obtained juices. This evidences feasibility of the proposed method with thermal and chemical preparation of beet chips prior to sucrose extraction.

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