Scientific Papers. Series D. Animal Science. Vol. LXVI, No. 1, 2023
ISSN 2285-5750; ISSN CD-ROM 2285-5769; ISSN Online 2393-2260; ISSN-L 2285-5750

ELECTROACTIVATION EMERGING METHOD OF PROCESSING OF WHEY WITH HIGH PROTEIN CONTENT

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Abstract

Electroactivation is an emerging method of non-residual processing of secondary dairy products, namely, whey with high protein content, which presents one of the directions that describe the treatment of whey with different initial protein content and the extraction of serum proteins into protein mineral concentrates. The treatment of whey with high protein content was carried out in different electrolyzers with different ratios of the processed whey volume to the electrode/cathode surface and different geometric shapes, different distances between the electrodes and the membrane, which influences the specific energy consumption per unit volume. The main objective of the work was the maximum extraction of whey proteins in protein mineral concentrates and the simultaneous isomerization of lactose into lactulose at low energy consumption and the exclusion of "dead"/inefficient areas for the electroactivation of whey in different diaphragm electrolyzers. The mechanisms of whey protein extraction depending on pH and redox potential values upon electrochemical activation of whey with high protein content are presented.

Key words: *electroactivation, pH, protein mineral concentrates, redox potential, whey.*

INTRODUCTION

The factors that influence industrial development are technological innovations, economic comfort, and environmental safety. In the context of the circular economy, regarding the processing of waste/secondary products, the complex, non-residual treatment is the main factor that satisfies the consumer and governmental requirements, which involves the initiation of extensive research, for the correction of certain actions applied in the technology (Ebrahim, 2020; Wang et al., 2021). Our society is currently facing the negative impact affecting the environment. Sustainable development implies the solution of ecological and social problems that require the implementation of emerging and innovative technologies (Bordean, 2009).
The use of electrochemical methods, in particular, electroactivation, allows solving a number of ecological problems, beneficial in alternative industrial processes, environmental protection, and pollution monitoring. Electrotechnologies are supposed to be among the most harmless ones in the processing of secondary products (Bersier et al., 2011; Ghazouani et al., 2019, Marin et al., 2017).
Electroactivation as a sustainable method for processing dispersed media, in particular, secondary dairy products (whey, buttermilk, etc.) is an alternative to conventional methods and is of growing interest due to its ability to transform electrical energy into chemical energy.
The principle of electroactivation can be defined as the intensification of the electron donor-acceptor properties through the energy exchange of the solution and the substances produced at the electrodes. The electrochemical activation method is a process of activation of water molecules caused by an electric field, which induces their metastable state, causing changes in pH, redox potential (E, mV), and electroconductivity (Karim & Aider, 2020; Kareb et al., 2017; Liato et al., 2015).
The global amount of obtained whey is estimated at approximately 180-190·10^6 tons/year, of which only 50% is processed (El-Tanboly et al., 2017; Dimou et al., 2019).

About 50% of the world production of whey obtained after cheese making is treated and transformed into various food and fodder products. Half of that amount is used directly in a liquid form, 30% as whey powder (obtained by spraying), 15% as lactose and its derivatives, and the rest - as protein concentrates obtained by membrane processes (Buchanan, 2023).

Membranes in whey processing play an important role in the separation of proteins and other valuable substances from whey, which have two aspects of use: filtration by the pore size (microfiltration, ultrafiltration, nanofiltration, etc.) and the use of ion-selective membranes at the electrodialysis and electroactivation of whey (Paladii et al., 2021a).

A total of $40 \cdot 10^6$ tons/year is produced in the European Union, the annual surplus of whey is $13 \cdot 10^6$ tons, containing approximately 619,250 tons of lactose (Zotta et al., 2020; Buchanan, 2023).

The solid content of whey (7-8%) makes up 50-70% of that of the initial milk. Almost all lactose and the most precious protein fractions (β-lactoglobulins, α-lactalbumins, bovine serum albumin, immunoglobulins, etc.) pass into the whey; they are not retained in primary dairy products, nor are a number of macro- and microelements, and vitamins (Paladii et al., 2021b; Soltani et al., 2017).

Proteins, due to their biological and nutritional properties, show increased interest in their usage in both food and pharmaceutical industries. A high potential of whey as a valuable raw material for obtaining food and bioactive substances with added value generates new directions in the development of technologies for the reuse of by-products of the dairy industry (Tsutsumi, 2014; Patricia et al., 2019).

The non-residual processing of whey and the valorization of its protein content is a complex problem from a technical and technological point of view, however, being of major importance.

The extraction of whey proteins from different types of whey ensures the reduction of the harmful impact on the environment, using the internal forces of the processed product, based on multiple inter- and intramolecular reactions.

Electroactivation is carried out via the electrical action on a technological liquid in the region of the polarized electrode, for example, in a diaphragm electrolyzer. However, unlike electrolysis and electrodialysis, electroactivation does not present a finite chemical process and is used to regulate the reactive capacity of the physico-chemical properties of media in various technological processes, with the aim of optimizing and increasing the effectiveness of the proposed process (Vrabie et al., 2019).

The electroactivation of whey is an amazing treatment process, that allows simultaneously the isomerization of lactose into lactulose and the recovery of protein mineral concentrates (PMC) (Bologa et al., 2009).

The purpose of the present work was to discuss the non-residual processing of whey with a high protein content, which allows the recovery of protein mineral concentrates at different treatment conditions.

MATERIALS AND METHODS

The experimental model of an electrolyzer

The electroactivation of whey with a high protein content imposes certain technical requirements to ensure the command and control of the technological process (see used electrolyzers in Figures 1-2) (Vrabie et al., 2019).

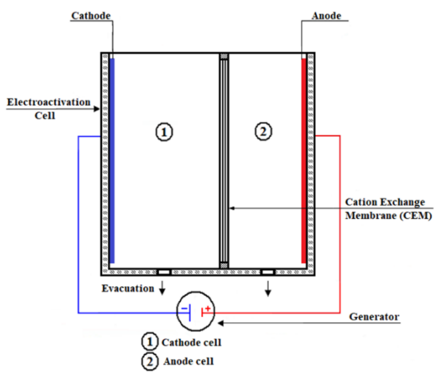

Figure 1. The layout of experimental membrane electrolyzers EDP-2 and EDP-4

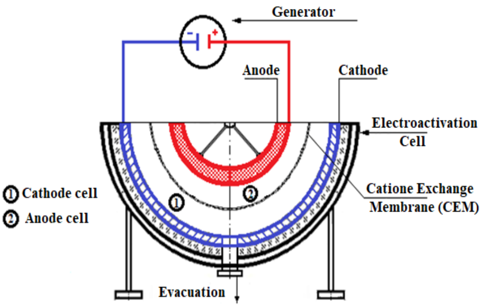

Figure 2. The layout of experimental membrane electrolyzers EDC-3 and EDC-pilot

The results of the research regarding the establishment of the factors that influence the electroactivation of whey, the understanding of the physico-chemical and biochemical processes that take place when the electric current passes through a dispersed medium with a complex biological structure such as whey, allowed the development of the principles and the constructive layouts of certain electrolyzers with different geometrical configurations, adapted to the peculiarities and technological requirements of the processing of secondary dairy products with the extraction of PMC and the simultaneous isomerization of lactose into lactulose.

Different types of electrolyzers (conventionally called EDP-2, EDP-4, EDC-3, and EDC-pilot) with certain geometric parameters, allowing the non-residual processing of whey, were fabricated and used in the research (Figure 3).

The EDP-2 and EDP-4 electrolyzers developed in the form of a parallelepiped (Figure 1) have the same distance between the electrodes and between the electrodes and the membrane, but a different ratio of the processed whey volume (V) to the electrode surface (S) V/S; for EDP-2 this ratio is 1.4 and for EDP-4 – 1.0 (Figures 3, 4).

The EDC-3 and EDC-pilot electrolyzers were intended for the processing of whey in a periodic and continuous regime of the flow rate of whey and the secondary liquid (anodic liquid).

They have the semi-cylindrical dielectric housing with the anode and cathode cells, the membrane, located on the semi-cylindrical housing, the electrodes: the cathode, placed on the semi-cylindrical casing and the anode, the inlet and outlet valves of the anodic liquid, the inlet and outlet valves of the whey.

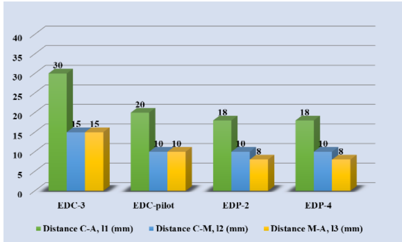

Figure 3. Constructive parameters of electrolyzers EDC-3, EDC-pilot, EDP-2, and EDP-4): l_1 - cathode (C) and anode (A) distance; l_2 - cathode and membrane distance (M); l_3 - membrane and anode distance

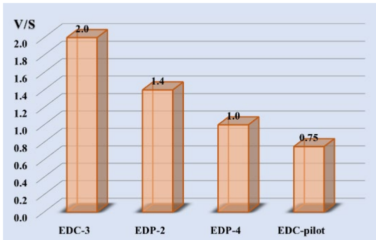

Figure 4. Variations of the ratio of the processed whey volume (V) to the surface of the electrode (cathode) (S) V/S of the electrolyzers EDC-3, EDC-pilot, EDP-2, and EDP-4

They have different distances between the electrodes and between the electrodes and the membrane, and the ratio of the processed whey volume (V) to the electrode surface (S) V/S, so that for EDC-3 this ratio is 2.0 and for EDC-pilot - 0.75 (Figures 3, 4).

The electroactivation of whey was carried out in different electrolyzers at different densities of the electric current: 10 and 20 mA/cm^2, keeping them constant during the processing period. The pumping regime of the working liquid (different types of whey in the cathode cell - CC) and the secondary liquid (2% $CaCl_2$ solution in the anode cell - AC) is periodic in nature. PMC were collected at certain treatment periods. The processed whey was discharged as foam, mixed to destroy the foam formed during processing and separated in the form of PMC as sediment and deproteinized whey (DW) as supernatant, in the field of mass forces. The secondary liquid from the anode cell underwent recycling.

The research was carried out using whey with a high protein content (supplied by the "JLC" Joint Stock Company, Chisinau, RM) obtained after the production of the granulated cheese "Grăuncior". The protein content was

determined by the Warburg method with the СФ-56 spectrophotometer (standard solution - bovine serum albumin, calibration coefficient k=1.72) (Ressler, 1976).
The amount of protein extracted in PMC (or the degree of protein extraction in PMC) (Q, %), was calculated by the difference between the protein content in the initial whey (IW) and that remaining in DW:

$$Q=Q_{IW}–Q_{DW}, \%$$

Where: Q is the protein content in PMC, Q_{IW} is the protein content in the IW, and Q_{DW} is the protein content in the DW.

RESULTS AND DISCUSSIONS

The electroactivation of whey with a high protein content obtained after the production of the granulated cheese "Grăuncior" processed in different electrolyzers allows the extraction of whey proteins into PMC and the simultaneous isomerization of lactose into lactulose.
The processing was carried out at different densities of electric current: 10 and 20 mA/cm^2, kept constant during the treatment; the variations of voltage, temperature, and physico-chemical parameters (pH values and redox-potential) were recorded, then the degree of the whey proteins extraction in PMC was specified.
The determination of the specific energy consumption per unit volume is a decisive factor in the study of the electroactivation of high-protein whey processed in different electrolyzers, namely: the extraction of whey proteins in PMC and the isomerization of lactose into lactulose occur simultaneously, so, at the specific consumption of energy, a possibility of obtaining two products at the same time - protein concentrates and the DW, which contains isomerized lactulose, appears.
The variations of the specific energy consumption per volume unit, A/V (Wh/ml), when electroactivating whey with a high protein content, in the investigated electrolyzers, indicate the lowest values when treated in the EDC-3 electrolyzer, which allows the processing of whey with the lowest specific energy consumption per unit volume at both 10 and 20 mA/cm^2 current densities in the processing regimes (Figures 5, 6).

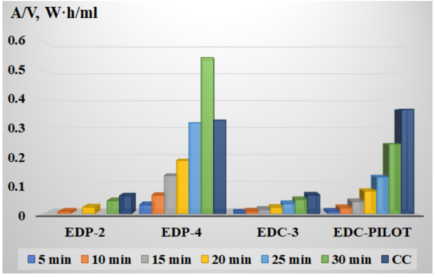

Figure 5. Variations of the specific energy consumption per unit of volume, A/V (Wh/ml), in EDP-2, EDP-4, EDC-3, and EDC-pilot electrolyzers, during the electroactivation of the whey obtained after the production of "Grăuncior", at j = 10 mA/cm^2

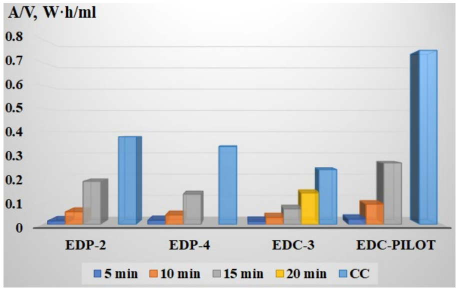

Figure 6. Variations of the specific energy consumption per unit of volume, A/V (Wh/ml), in EDP-2, EDP-4, EDC-3, and EDC-pilot electrolyzers, during the electroactivation of the whey obtained after the production of "Grăuncior", at j = 20 mA/cm^2

The variations of the consolidated specific energy consumption per unit of volume during the electroactivation of whey with a high protein content, in the periodic treatment regime at the electric current densities of 10 and 20 mA/cm^2 in different electrolyzers indicate the rationality of the use of EDC-3 (Figure 7).
The electroactivation of the whey investigated in this article in different electrolyzers with different geometrical parameters influences the specific energy consumption per unit of volume, and, namely, it depends on the processed volume, the initial solid and protein content of this type of whey and is attributed to obtaining two products simultaneously: PMC with different content of whey proteins and the DW containing isomerized lactulose.

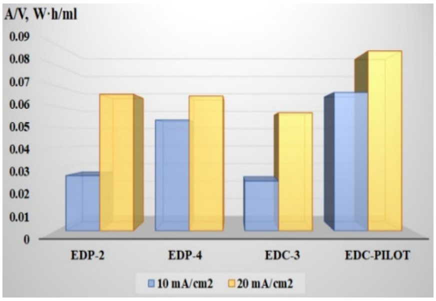

Figure 7. Variations of A/V (Wh/ml) during electroactivation of whey obtained after the production of "Grăuncior" in electrolyzers EDP-2, EDP-4, EDC-3, and EDC -pilot at the electric current densities 10 and 20 mA/cm^2

The variations of thermal parameters during the electroactivation of whey in different electrolyzers requires the correct control of processing regimes, which is caused by Joule heating, dependent on the conductivity of the treated medium.

The necessary conditions for reducing the resistance of the system require: ensuring the presence of charge carriers depending on the solid content of whey, reducing the membrane resistance, maintaining the optimal distance between the electrodes and between the electrodes and the membrane.

The whey proteins have a thermal denaturation point (55-60°C), and their extraction into protein concentrates requires compliance with certain processing regimes.

The main physico-chemical parameters - pH values and redox potential E, (mV) - during the electroactivation of whey with a high protein content in different electrolyzers vary due to the characteristic reactions of the water dissociation process in the cathode and anode cells at the surface of the electrodes, and they characterize the physical, chemical, and biochemical changes of whey.

Depending on the reaction of the medium and the ratio of the acidic or alkaline radicals of amino acids, proteins have a positive or negative charge.

Simultaneously, upon electroactivation, the dissociation of water at the cathode forms hydroxide ions, which characterizes the alkaline medium and releases hydrogen gas, which imposes ionic flotation through foaming, accompanied by protein salinization due to the migration of bivalent ions from the anode cell, which intensifies the foaming process and the "unfolding" of protein molecules.

The amine groups, in turn, intensify the alkaline medium, imposing a rapid increase of pH values in the cathode cell, which favors the passage of protein fractions through their isoelectric point and a considerable decrease in the redox potential, characterized by intense reduction reactions.

An increase of the pH values upon electroactivation of dispersed media is also accompanied by the passage of aquacomplexes, where polarized water molecules that maintain the colloidal structure serve as ligands together with proteins, in hydrocomplexes, that have hydroxyl ions as ligands (Vrabie et al., 2011).

Depending on both the protein content of the whey and the geometric shape of the electrolyzer and the volume processed, the transition of aquacomplexes into hydrocomplexes is different.

In the same way, the redox potential decrease characterizes the redox reactions which have intensely negative values in the cathode cell, where, respectively, reductive reactions characteristic of the processed medium take place.

A slower increase of the pH values in electrolyzers EDP-4, EDC-3, and EDC-pilot upon electroactivation of whey with a high protein content, at electric current density j = 10 mA/cm^2, denotes a slower transition of aquacomplexes into hydrocomplexes compared to those in EDP-2 in which this passage is practically absent, and the formation of hydrocomplexes occurs very quickly (Figure 8).

The redox potential decrease has the same character in the four electrolyzers, indicating the rate of formation of active reductants (Figure 9).

The analysis of the physico-chemical parameters (pH and E, mV) allows the investigation of the multiple transformations of the main components of whey, first of all, of the protein fractions, as well as the activation state of the amino acids, which depends on the energy delivered to the system and their state activation.

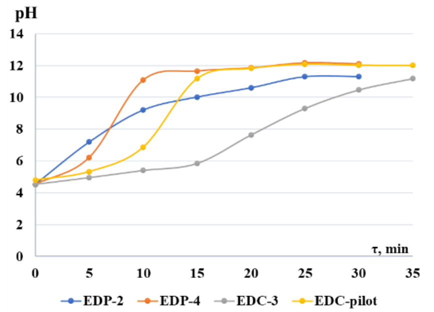

Figure 8. Variations of pH in electrolyzers EDP-2, EDP-4, EDC-3, and EDC-pilot during electroactivation of whey obtained after the production of "Grăuncior", at j = 10 mA/cm^2

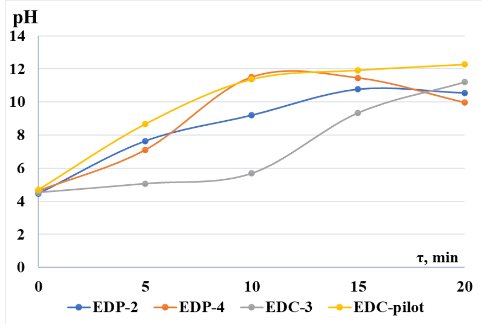

Figure 10. Variations of pH in electrolyzers EDP-2, EDP-4, EDC-3, and EDC-pilot during electroactivation of whey obtained after the production of "Grăuncior", at j = 20 mA/cm^2

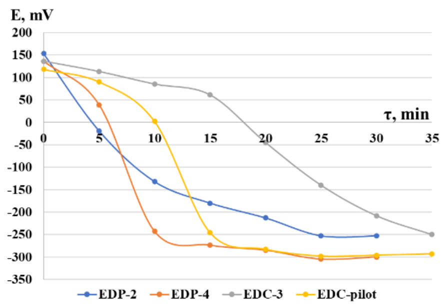

Figure 9. Variations of redox potential (E, mV) in electrolyzers EDP-2, EDP-4, EDC-3, and EDC-pilot during electroactivation of whey obtained after the production of "Grăuncior", at j = 10 mA/cm^2

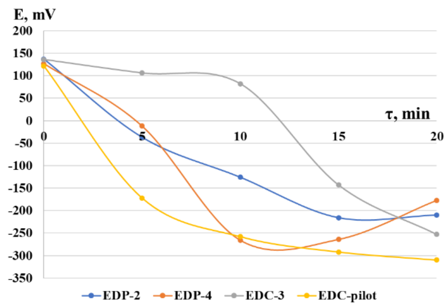

Figure 11. Variations of redox potential (E, mV) in electrolyzers EDP-2, EDP-4, EDC-3, and EDC-pilot during electroactivation of whey obtained after the production of "Grăuncior", at j = 20 mA/cm^2

The variations of the pH values in electrolyzers EDP-2, EDP-4, EDC-3, and EDC-pilot during the electroactivation of whey with a high protein content, at the electric current density j = 20 mA/cm^2, indicate to a faster transition of both aquacomplexes into hydrocomplexes (Figure 10), as well as the rate of formation of active reductants, characterized by redox potential variation (Figure 11).

The exception is EDC-3 which has a larger processing volume and a longer distance between the electrodes and between the electrodes and the membrane.

The electroactivation of this type of whey with a high protein content requires the specific energy consumption to be lower, thus ensuring the achievement of the maximum necessary for the extraction of whey proteins under optimal processing conditions.

The extraction of whey proteins in PMC (Q, %) from the IW varies depending on the initial solid content, the processing regime (different current densities, the amount of IW processed), the variations of electrical, thermal, and physico-chemical parameters, for the duration of the treatment. The intense foaming in the first minutes of processing is an indication of an intense salinization of the protein fractions, which leads to the formation of PMC.

The salinization of whey proteins upon electroactivation is one of the multiple mechanisms that contribute to the extraction of protein fractions in PMC.

The variations of the degree of extraction (Q, %) of whey proteins in PMC upon electroactivation of whey with a high protein content, at the electric current density j = 10 mA/cm^2, in electrolyzers EDP-2, EDP-4, EDC-3, and EDC-pilot, indicates the highest values for electroactivation in EDC-3, making up about

80% both in the foam phase (30 min of treatment) and in the liquid phase - the content of the cathode cell, and is considerably higher compared to that in three other electrolyzers (Figure 12).

The variations of the degree of extraction (Q, %) of whey proteins in PMC upon the electroactivation of whey with a high protein content, at the electric current density j=20 mA/cm^2, shows that in the EDP-2, EDP-4, and EDC-pilot electrolyzers many whey proteins in PMC are extracted, but in EDC-3, during the entire treatment period, a lower percentage of extraction occurs compared to electroactivation at 10 mA/cm^2, which is possibly influenced by the hydrolysis of serum proteins, which are not extracted in PMC, but remain in the DW and allows us to conclude that at a higher electric current density under these treatment conditions protein mineral hydrolysates (PMH) in EDC-pilot could be obtained (Figure 13).

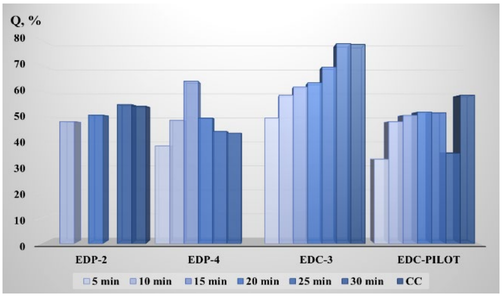

Figure 12. Variations of the degree of extraction (Q, %) of whey proteins in PMC upon electroactivation of whey obtained after the production of "Grăuncior", at j=10 mA/cm^2, in electrolyzers EDP-2, EDP-4, EDC-3, and EDC pilot

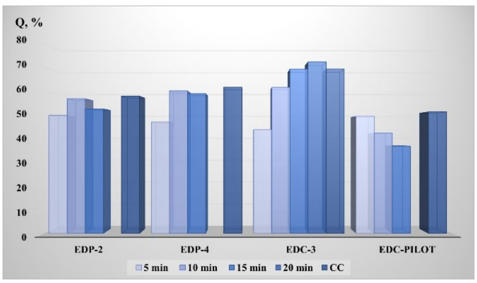

Figure 13. Variations of the degree of extraction (Q, %) of whey proteins in PMC upon electroactivation of whey obtained after the production of "Grăuncior", at j = 20 mA/cm^2, in electrolyzers EDP-2, EDP-4, EDC-3, and EDC pilot

The different and inhomogeneous extraction of serum proteins in PMC during the electroactivation of whey with a high protein content in different electrolyzers is primarily conditioned by the properties of each individual fraction and their behavior during electrochemical activation, the initial solid content, especially the protein one, of the constructive/geometrical parameters, of the processing regimes, of the electrical, thermal, and physico-chemical parameters that denote the individual approach of the investigated whey depending on the initial solid content, which requires the stipulation of the technical and technological treatment conditions.

It is important to adjust the technical and technological parameters in accordance with the characteristics of the specific energy consumption per unit of volume, which denotes the efficiency and profitability of the electroactivation of different types of secondary dairy products.

CONCLUSIONS

The need for non-residual processing of whey and the valorization of its protein content presents a complex technical and technological problem of major importance.

The electroactivation of whey with a high protein content, at electric current densities j = 10 and 20 mA/cm^2, in electrolyzers with different constructive/geometric configurations (named EDP-2, EDP-4, EDC-3, and EDC-pilot) allowed the formulation of the following conclusions:

1. The main factors that influence the electroactivation of whey are: constructive/geometrical parameters (with parallelepiped or semi-cylindrical casing); the volume of the processed whey and the V/S ratio; the distance between the electrodes and between the electrodes and the membrane; the electric current density (j = 10 and 20 mA/cm^2); the solid content of the IW, which characterizes the specific energy consumption per unit of volume A/V (Wh/ml).

2. The different and inhomogeneous extraction of whey proteins in PMC at the electroactivation of high-protein whey in different electrolyzers and different processing regimes is conditioned by the properties of each

individual fraction and their behavior at the electrochemical activation, the initial solid content, in particular, of the protein one.

3. The maximum extraction of whey proteins in PMC takes place at the electric current density j = 10 mA/cm^2 in electrolyzers with a semi-cylindrical casing, which allows the exclusion of "dead"/inefficient areas (EDC-3 and EDC-pilot) - about 80% and 60%; at the electric current density j = 20 mA/cm^2, the extraction is less and, possibly, influenced by protein hydrolysis and allows us to conclude that at a higher electric current density under these treatment conditions PMH could be obtained in the EDC-pilot electrolyzer.

4. The exclusion of "dead"/inefficient areas when using the EDC-3 electrolyzer allows the maximum extraction of whey proteins in PMC at a specific energy consumption twice lower.

5. The recovery of whey proteins at different pH and redox potential values at the electrochemical activation of high protein whey is due to the sedimentation of different protein fractions at their isoelectric point and salinization of whey proteins.

ACKNOWLEDGEMENTS

This research work was carried out with the support of the state program ANCD 20.80009.5007.06 (2020-2023).

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