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Milk protein production by a more environmentally sustainable process: bipolar membrane electrodialysis coupled with ultrafiltration†

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The increased demand for food production to nourish the rapidly growing human population raises serious sustainability issues for the food sector. Indeed, conventional food production lines involve processes having a significant environmental burden. Hence, the present study aims to demonstrate an environmentally sustainable process of food production. The milk protein was chosen as a model food ingredient due to its exceptional role in the human diet. The proposed innovative process of milk protein production includes bipolar membrane electrodialysis coupled with ultrafiltration (EDBM-UF). The crucial problem during the EDBM-UF of milk, such as different types of membrane fouling, was successfully solved. Moreover, the life cycle assessment of the novel EDBM-UF protein production process was carried out and compared to a conventional acid/base process. Additionally, a sensitivity test of electricity supply at different geographical locations of the world was performed since electricity is the main energy source for the EDBM-UF process and it could be derived from different sources (renewable and non-renewable). The assessment results demonstrate that the proposed electromembrane process has significant environmental benefits compared to the conventional process using chemicals independently from the electricity supply mix from all considered geographical locations. Thus, EDBM-UF could become a prospective industrial technology taking into account environmental concerns and promoting the development of healthy human society.

1. Introduction

The world population is growing each year. In 2017, we have 7.4 billion people and the population will reach more than 9 billion by 2050. Moreover, 70% of the population will be urban compared to 49% today.[1] The population growth and urbanization raise serious challenges for the agri-food sector since there will be a 70% increase of the global food demand by 2050.[1] To satisfy the demands of the growing population, the food industry should significantly increase its productivity. How can it be performed? The simple expansion of the traditional supply chains and related infrastructure would lead to harmful consequences on the environment due to an increased pressure of the agri-food industries on the eco-systems of our planet, human health and natural resources. Eco-design of the agri-food systems is therefore necessary and inevitable to minimize the unwanted consequences. Eco-design is based on sustainable processing providing healthy food to meet the current food needs while meeting the need to supply food for present and future generations with a minimal negative impact on the environment.[2]

The present paper is about protein production. Proteins are considered as one of the most important food components due to their ability to furnish essential amino acids for the synthesis of organ and tissue proteins and other nitrogen-containing compounds for the normal growth and functionality of an organism. Moreover, proteins are an important source of bioactive peptides having anticancer, immunomodulatory, antimicrobial, hypotensive, cytomodulatory, antidiabetic, opioid and other properties improving human health and emotional conditions.[3,4] The above-mentioned benefits of protein intake in human diets led to the tremendous increase in the demand for food protein production.[5] The current study focusses on the production of proteins from skim milk since dairy proteins have several advantages compared to vegetable ones such as a higher protein efficiency ratio, biological value, net protein utilization and digestibility.[6] This paper aims to present tech-

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nological aspects related to the development of a novel eco-designed process for protein production and the evaluation of its environmental impacts compared to the traditional protein production process.

Firstly, the innovative process for skim milk protein production will be presented. This process consists of the production of milk caseins and whey proteins *via* electrodialysis with a bipolar membrane (EDBM) coupled with ultrafiltration (UF). The EDBM-UF process does not use any hazardous chemicals and does not generate waste like conventional technologies using acids, fermentation or ion-exchange resins.[7,8] An EDBM module comprises a particular bipolar membrane allowing the production of H^+ and OH^- ions from water under the application of current. Thus, milk can be acidified in the EDBM module and caseins can be precipitated and then separated from whey proteins.[8] The base generated by the bipolar membrane could be used for the subsequent conversion of insoluble caseins into their soluble form called caseinates instead of using a chemically produced base. The implication of the EDBM process is limited by the presence of membrane fouling, which is the major problem of all membrane processes.[9] Indeed, cleaning procedures and membrane replacement cost up to 47% of the overall process expenses.[10] There are two types of fouling occurring during milk electroacidification: protein fouling (clogging) inside the EDBM cell and mineral fouling called scaling on cation-exchange membranes. Recently, the protein fouling problem has been solved by coupling an EDBM module with an ultrafiltration (UF) module.[8] Indeed, an UF membrane hampers the penetration of protein fractions inside the EDBM module significantly increasing the performance of milk electroacidification. However, the scaling problem, which hampers the industrial application of the EDBM-UF process, remains unsolved despite the attempts to inhibit the formation of minerals by applying pulsed electric fields, changing the hydrodynamic conditions and other methods.[8,11–13] By knowing the mechanism of scaling formation on ion-exchange membranes, it is clear that the main promoter of mineral precipitation on the membrane surface is the alkaline environment. Indeed, OH^- ions generated by the bipolar membrane or by the water splitting phenomenon, occurring on ion-exchange membranes thanks to the development of concentration polarization, could interact with Ca^{2+} and Mg^{2+} ions (present in milk and released from casein micelles during acidification) forming insoluble hydroxides. Moreover, HCO_3^- ions present in milk[11,14] could be modified into CO_3^{2-} ions and could be found in the form of a $CaCO_3$ precipitate on cation-exchange membranes.[8,14] Hence, in this work, a new design of the EDBM module will be studied aiming to avoid the high concentration of OH^- ions in the compartments containing Ca^{2+} and Mg^{2+} ions and to prevent scaling formation on cation-exchange membranes.

Secondly, the results of the proposed EDBM-UF process will be used to evaluate the environmental profile of sodium caseinate manufacturing by performing a Life Cycle Assessment (LCA). The LCA is a tool that quantifies the use of resources (*e.g.* fuels, water, land, *etc.*) and emissions (water, soil and air pollutants) throughout the whole life cycle of the evaluated product, service or process and characterizes their adverse effects into relevant impact categories.[15,16] This provides comparative metrics to evaluate the most environmentally harmful steps of the production chain. In this research, we will compare the newly developed EDBM-UF process against a conventional acid/base process, identify environmental hotspots along each life cycle and perform a sensitivity analysis on electricity supply since EDBM-UF uses electricity as a main energy source. This latter evaluation will inform on the impact variability of using both EDBM-UF and acid/base technologies in different regions of the world, each supplied by a distinct electricity mix (renewable and nonrenewable).[17]

2. Experimental section

2.1 Configuration of electrodialysis and ultrafiltration modules

The EDBM module (Fig. 1) used was a laboratory scale cell (Model MP, 100 cm^2 of effective surface) from ElectroCell Systems AB Company (Täby, Sweden). The tested cell consists of six compartments separated by three Neosepta CMX-SB cation-exchange membranes, one Neosepta BP-1 bipolar membrane and one Neosepta AMX-SB anion-exchange membrane: all these membranes manufactured by Astom Ltd (Tokyo, Japan) are food grade membranes. This configuration was compared with the control one described in the literature,[8] consisting of five compartments separated by two anion-exchange membranes, one cation-exchange membrane and one bipolar membrane (Fig. A1†). The four electrolytes: milk ultrafiltered fraction (MUF), containing mainly lactose and minerals (*e.g.* Ca^{2+}, Mg^{2+}, K^+, Cl^- and $P_xO_y^{n-}$), 2 g l^{-1} NaCl (KCl for the control configuration) (500 ml, 150 ml min^{-1}), and 20 g l^{-1} NaCl (500 ml, 500 ml min^{-1}) were circulated using three centrifugal pumps. The anode, a dimensionally stable electrode (DSA), and the cathode, a 316 stainless steel electrode, were supplied with the MP cell. The UF module (Fig. 1 and A1†) was equipped with a spiral wounded membrane with a molecular weight cut-off of 10 kDa and a surface of 2.13 m^2 (GE Water and Process Technologies, Vista, USA). The UF system was run at room temperature (22 ± 1 °C) under a pressure of 25 psi.

2.2 Protocol

EDBM was carried out as a batch process using a constant current density of 20 mA cm^{-2} generated by using a Xantrex power supply (Model HPD 60-5SX; Burnaby, Canada). The permeate from the UF module (MUF) passed directly to the EDBM cell and electroacidification was stopped when the pH in the UF reservoir reached 5.0 in order to obtain a fully precipitated casein fraction.[8] Moreover, three co-products were generated after EDBM-UF milk electroacidification (Fig. 1):

• NaOH solution (consisted of OH^- ions generated by the BPM and Na^+ ions migrated through the CEM);

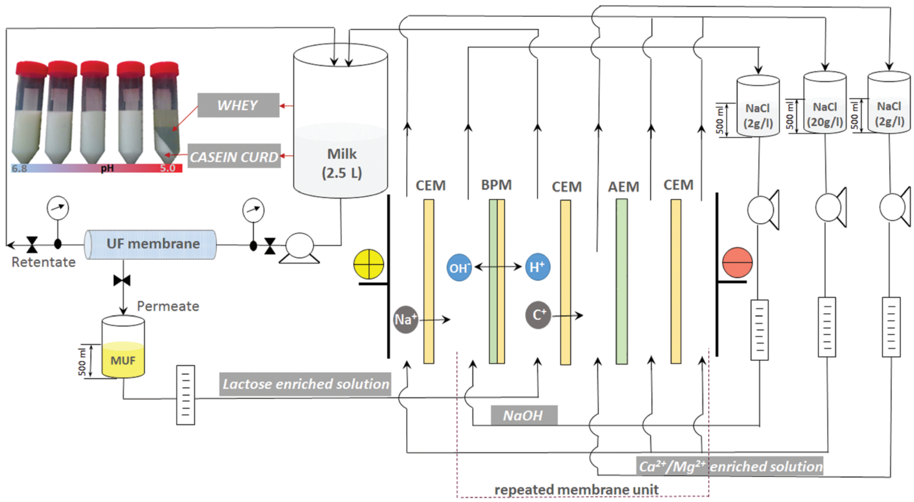

Fig. 1 Scheme of the six-compartment bipolar membrane electrodialysis coupled with ultrafiltration. The final products are indicated inside the grey squares. C^+ represents migrating cations.

• lactose enriched solution (consisted of lactose and minerals separated from milk by the UF membrane);

• Ca^{2+}/Mg^{2+} enriched solution (consisted of Ca^{2+}/Mg^{2+} ions migrated through the CEM from lactose enriched solution (MUF)).

Three replicates of each EDBM-UF treatment were performed. During each treatment, 1.5 ml samples of the acidified milk solution were taken at every 0.4 pH unit decrease. The time required to reach the final pH value, the anode/cathode voltage difference and the temperature were recorded as the treatment progressed. After electroacidification, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) were carried out on a CMX-SB membrane separating the MUF and Ca^{2+}/Mg^{2+} enriched solution (Fig. 1) to evaluate the presence and composition of membrane scaling.

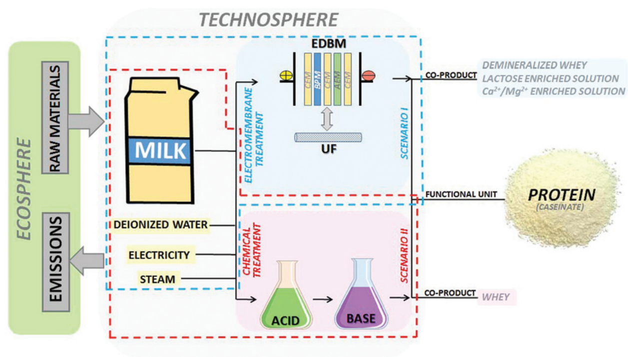

Fig. 2 Scheme of the life cycle of both processes of milk protein (caseinate) production.

2.3 Life cycle assessment

The life cycle assessment (LCA) was carried out according to ISO 14044 [18] to evaluate the potential environmental impacts associated with the production of protein (caseinate) powder from skim milk of two different scenarios: (1) electromembrane treatment (scenario I) and (2) chemical treatment (scenario II) (Fig. 2). Moreover, the sensitivity test of electricity mix supply in different regions of the world was performed since an electromembrane process uses electricity as a main energy source, which can have significant differences in the environmental profile depending on the production process (ESI†). The function defined for both evaluated processes focuses on the production of sodium caseinate powder chosen as the main milk protein product. The functional unit (*i.e.* the reference to which the environmental impacts of a scenario are related) was defined as being the production of 1000 kg eq. of sodium caseinate powder from skim milk at the dairy factory gate, ready to be delivered.

Similarities and differences of both studied systems for caseinate powder production from skim milk[19] with their respective life cycle stages are presented (Fig. 3): electromembrane processing (scenario I) and acid/base processing (scenario II). The casein precipitation process of scenario I occurs through the electrogeneration of H^+ ions by using an EDBM-UF system (data from the present experiment), whereas it is obtained by chemical precipitation *via* the addition of an inorganic acid for scenario II (data from the literature). Note that electricity supply is initially considered for a Quebec context (more than 99% from hydropower[20]). It is known that electrically precipitated casein has a higher purity (91.4–95.0%)[12,21] compared to the chemically precipitated casein (83.0–85.0%).[12,22] This fact is due to the use of a strong acid during chemical acidification, which can affect the integrity of caseins and whey proteins and trigger their interactions.[23,24] Moreover, the ionic strength of chemically acidified milk is significantly higher than that of electroacidified milk, leading to possible contamination with ions of precipi-

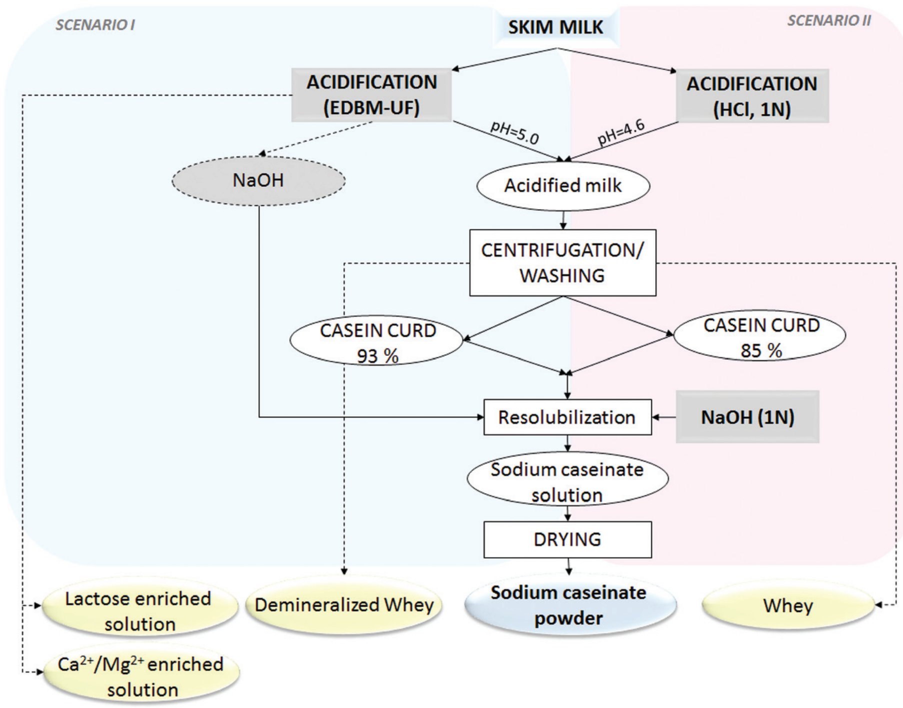

Fig. 3 Description of sodium caseinate powder production by EDBM-UF (scenario I) and acid/base (scenario II) treatments.

tated casein.[12] After precipitation, caseins are centrifuged and washed several times. The whey is separated from the casein curd after the first centrifugation. After the centrifugation/washing step, the casein curd is resolubilised with NaOH. At this step, the alkali generated by EDBM-UF or conventional alkali is used. Eventually, the sodium caseinate solution is dried to obtain the caseinate powder. Data for the EDBM-UF acidification step were generated from the experimental part of the present paper assuming the industrial scale of EDBM-UF modules (see the ESI†). All data concerning other processing steps were obtained from multiple literature sources.[12,19,25–29] The detailed description of the reference flow quantification is given in the ESI.† The cleaning-in-place operation was out of consideration for the present study.

The life cycle impact assessment (LCIA) of both scenarios was performed using Impact 2002+ method. The climate change impact category was updated with the IPCC 2013 characterization factors[30] to identify the environmental hotspots of caseinate powder manufacturing. All impacts were attributed to the same functional unit (production of 1000 kg eq. of sodium caseinate powder). The cut-off approach was applied to deal with the multifunctional character of caseinate powder production. Therefore, no environmental burdens are associated with the co-products generated by the respective product systems described in Fig. 3; they are all attributed to the caseinate powder. This approach is justified by the impossibility, at this stage of development, to define the market values of the different co-products. The open-LCA software (GreenDelta, Berlin, Germany) with the ecoinvent 3.2 database was used to carry out the LCA.[31]

3. Results and discussion

3.1 Skim milk electroacidification and membrane fouling

The images obtained from scanning electron microscopy and EDS demonstrate that the original non-treated CMX-SB membrane (Fig. A1†) has a flat homogeneous surface. This membrane contains carbon, oxygen, chlorine and sulfur coming from the membrane polymeric matrix and ion-exchange groups, sodium coming from ion-exchange groups as a counter-ion, and gold and palladium elements from the covering layer serving for the improvement of the membrane surface conductivity and consequently image quality. However, the CMX-SB membrane after skim milk electroacidification in a five-compartment EDBM module contains a crystalline deposit on its surface (Fig. 4A). This fact could be understood from the EDS indicating the appearance of high intensity peaks of Ca and Mg elements. Indeed, it is known that milk contains Ca^{2+} and Mg^{2+} ions, which could be found as mineral fouling (scaling) on the surface and inside the CEM in the carbonate and/or hydroxide forms. The CEM after the treatment of MUF in the EDBM module having a new six-compartment design does not contain any visible deposit on its surface (Fig. 4B). Moreover, EDS does not indicate any scaling agents (Ca and Mg elements). The differences between the results obtained from SEM and EDS analyses could be understood from the mechanisms of membrane scaling formation. There are several parameters affecting membrane scaling formation such as the concentration of scaling ions, pH, temperature, hydrodynamic conditions and water splitting phenomenon occurring in ED systems approaching or exceeding the limiting

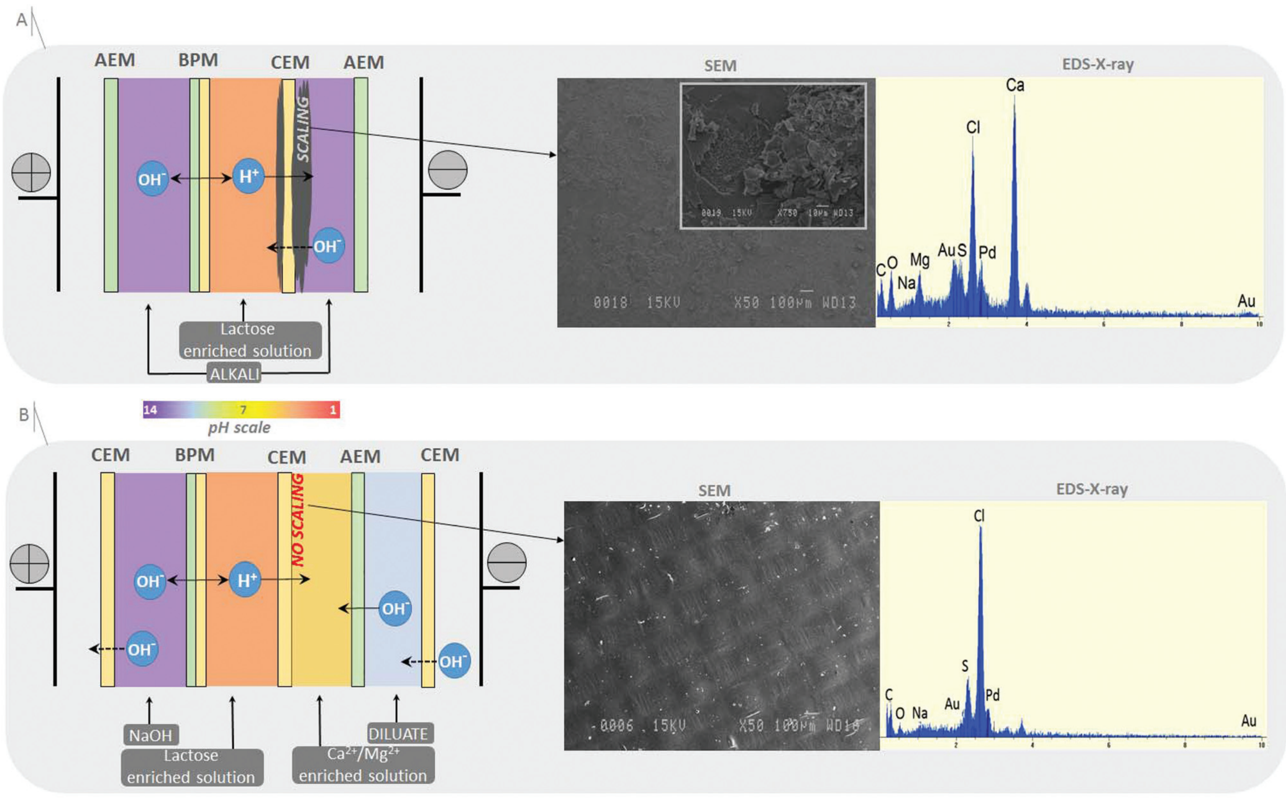

Fig. 4 Scheme of both EDBM configurations tested for skim milk electroacidification with the respective Scanning Electron Microscopy (SEM) images and Energy Dispersive X-ray Spectroscopy (EDS) images of the CMX-SB membrane: (A) conventional five-compartment stack and (B) new six-compartment stack design.

current.[9] The literature dedicated to the scaling problem during milk electroacidification revealed that the main factor affecting the formation of CMX-SB scaling appearing in $CaCO_3$, $Mg(OH)_2$ and $Ca(OH)_2$ forms is the alkaline pH of the concentrate stream (alkali in Fig. 4A and Ca^{2+}/Mg^{2+} enriched solution in Fig. 4B).[8,12]

Recent studies attempted to prevent fouling formation by the application of pulsed electric fields, addition of potassium ions and changing the hydrodynamic conditions inside the EDBM cell.[8,11] Indeed, pulsed electric fields can decrease the concentration polarization and water splitting as well as prevent the nucleation and crystal growth of membrane scaling.[9] Moreover, the addition of K^+ ions having a much higher ionic mobility compared to Ca^{2+} and Mg^{2+} ions[32,33] would inhibit the migration of scaling ions and scaling formation. Additionally, the increase of the solution flow rate inside the EDBM cell would inhibit the attachment and growth of scaling. All the above methods demonstrated a high efficiency for scaling inhibition (up to 50%) though they could not completely prevent the membrane scaling formation. Thus, the new design of the EDBM cell was proposed to answer this problem. Indeed, an additional CEM membrane was installed into the EDBM cell to prevent the migration of OH^- ions generated by the BM towards the concentrate stream (Fig. 4B) and consequently to prevent the occurrence of alkaline pH near the surface of the CMX-SB membrane coming in contact with the acidified milk fraction MUF. The application of the proposed EDBM cell design allowed the prevention of pH growth above the neutral values in the Ca^{2+}/Mg^{2+} enriched solution during milk electroacidification (Fig. 5). One could notice that during the first 20 min of electroacidification, the pH in the Ca^{2+}/Mg^{2+} enriched solution remains acidic. This fact is due to the migration of H^+ ions generated by the BM

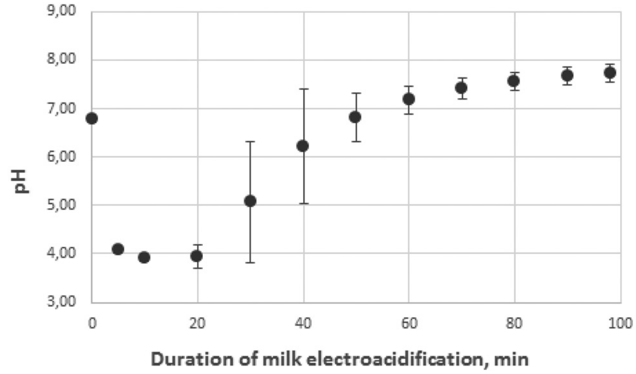

Fig. 5 pH evolution in the concentrate compartment of the EDBM cell (Ca^{2+}/Mg^{2+} solution in Fig. 1) during skim milk electroacidification.

from the acidification compartment towards the concentrate stream. However, after 20 min, the pH increases slightly to values higher than 7.0 and remains constant until the end of the treatment. The pH increase relates to two phenomena. Firstly, there is a leakage of OH^- ions generated by the BM through the CEM due to the fact that membrane permselectivity is less than 100 percent and highly mobile OH^- could penetrate even through the membrane having cationic selectivity.[34] Secondly, during milk acidification, there is a release of weak anions (mainly phosphates and citrates)[35] increasing the milk buffering capacity and consuming a large part of free protons generated by the BM. Hence, the leakage of OH^- ions and reduced migration of H^+ ions create a neutral pH in the concentrate stream, which is unfavorable for scaling formation on the CEM. Hence, the CEM after milk electroacidification in this special six-compartment EDBM configuration looks like the original one (Fig. 4B and A1†). The new six-compartment design of the EDBM stack allows the complete elimination of membrane scaling, which is the crucial breakthrough in skim milk electroacidification due to the significant improvement of the process efficiency and decrease in process costs.[9]

The proposed EDBM-UF process has several important advantages apart from the possibility to produce caseins without the addition of chemicals. Indeed, the EDBM-UF process generates several co-products such as a partially demineralized whey, a solution enriched in lactose, a solution enriched in Ca^{2+} and Mg^{2+} ions and a NaOH solution (Fig. 1). Could these co-products be valorized? Definitely, they do have important values. Whey proteins possess a high nutritional value and could be used in different food sectors (*e.g.* dairy, confectionary, infant formula, meat, *etc.*), pharmaceutical sectors (*e.g.* tablets, inhalers and drugs) and nutraceutical sectors (*e.g.* bioactive peptides, probiotics and prebiotics).[36,37] The EDBM-UF process generates the whey fraction with a low mineral content due to demineralization in the EDBM module. Moreover, this whey fraction contains less lactose due to its separation by the UF membrane. These advantages of the EDBM-UF whey fraction positively affect its following transformation (*e.g.* reduced risks of fouling on filtration membranes and evaporators) and properties (*e.g.* bioactivity and functionality). The same situation happens with the lactose-enriched solution, which could be further transformed to lactose having food and pharmaceutical applications.[38] The Ca^{2+}/Mg^{2+} enriched solution could be used directly as a stabilizer of sterilized milk, cheese and yogurt or Ca^{2+} and Mg^{2+} ions could be precipitated from the EDBM concentrate stream and used as food supplements.[39,40] Finally, NaOH generated in the basification compartment of the EDBM module could be used as a solubilizing agent for casein and in the cleaning operation of dairy equipment. Hence, one could see that EDBM-UF allows obtaining several valuable co-products along with the main casein product, while in the case of conventional chemicals there are just two products: casein and whey with high mineral, lactose and water contents.

Solving the main problem of the electromembrane process for casein production, membrane fouling, along with the generation of valuable co-products allows the industrialization of the proposed innovative approach. However, knowing the current global tendencies focused on the improvement of sustainability of food production lines, the next part will be dedicated to the estimation of environmental impacts of the proposed EDBM process and its comparison with the conventional process using chemicals.

Life cycle assessment

The life cycle assessment profile of the sodium caseinate powder production by the conventional (acid/base) process and the innovative (EDBM-UF) process demonstrates that this latter process has about 10% less impacts on all damage categories compared to the acid/base process (Fig. 6A). More than 95% of the overall impacts of the sodium caseinate powder production are related to the skim milk supply. For instance, the greenhouse gas emissions for the production of milk at farms are mainly due to methane release during enteric fermentation, to nitrous oxide emissions from the usage of nitrous fertilizers and manure, and to carbon dioxide emissions from tractors and trucks.[41] The on-farm operations significantly affect non-renewable energy use (*i.e.* animal feed production, drying fodder, milking, ventilation, *etc.*), water consumption and acidification (mainly due to the use of ammonia fertilizers). The high impact intensity of milk production allows the conclusion that the caseinate production process consuming more milk is likely to have a higher environmental burden. Indeed, the acid/base process of caseinate production demands 3723 L more of skim milk compared to the EDBM-UF process, which is due to the lesser purity of the chemically precipitated casein curd.[12] To facilitate the interpretation, Fig. 6B focuses on the results that only take into account the additional milk required by the acid/base process compared to the EDBM-UF process (Δ = $milk_{HCl-NaOH} - milk_{EDBM-UF}$), *i.e.* excluding the equal amount of milk supplied to both systems. The higher efficiency of the electromembrane precipitation process, *i.e.* the quantity of precipitated casein/quantity of skim milk, appears to be the key parameter for an improvement of eco-design compared to the chemical process: 93% *vs.* 85% for the EDBM-UF process compared to the acid/base process, respectively. The next hot spot of the caseinate powder manufacturing process is the steam flux for the drying of sodium caseinate solution. However, the quantities of steam to produce 1000 kg eq. of caseinate powder were equal for both studied scenarios (Table A1†). The reasons for relatively high impacts allocated to steam relate to its production, which implies the combustion of non-renewable energy sources (76% of natural gas and 24% of oil) resulting in CO_2, SO_2, and NO_x emissions, ozone depletion, marine and fresh water eutrophication, *etc.* These results corroborate with other investigations of milk protein powder production[42–44] reporting the necessity of optimizing steam consumption by the improvement of heat recovery and condensate return, elimination of steam leaks, insulation of pipes and steam lines, improvement of equipment design and eventually the use of renewable energy sources (*e.g.* solar and geothermal energy).

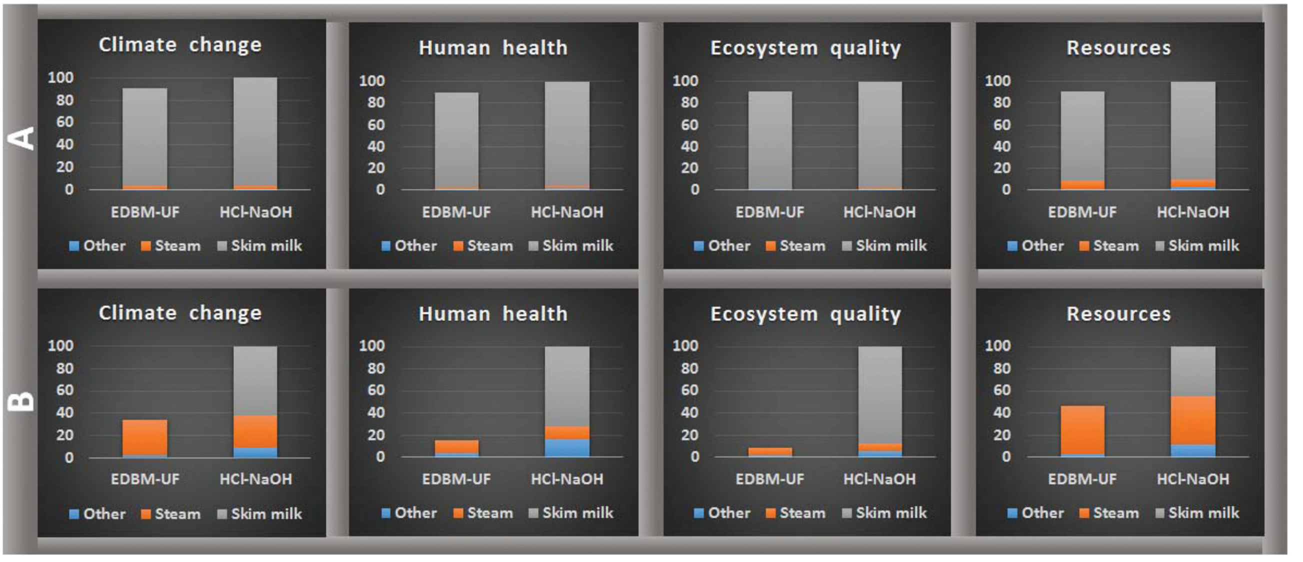

Fig. 6 Relative contributions (in %) of caseinate powder manufacturing by EDBM-UF and acid/base technologies to the impact categories: climate change (IPCC 2013), human health (Impact 2002+), ecosystem quality (Impact 2002+) and resource depletion (Impact 2002+). The A row reports the results including all processes needed to fulfill the functional unit (production of 1000 kg eq. of sodium caseinate powder). The B row reports results of the same scenarios assessed in A excluding the equal amount of skim milk supplied in both systems and therefore, only accounting for the difference of skim milk impacts between acid/base and EDBM-UF technologies.

4. Conclusions and perspectives

• This research work demonstrates the feasibility of manufacturing casein (the main milk protein) by an innovative electromembrane process (EDBM-UF) without any membrane fouling meaning the substantial improvement of the process efficiency and the decrease of its costs;

• EDBM-UF acidification of skim milk allows obtaining, in addition to the casein main product, several valuable co-products such as demineralized whey, lactose enriched solution and Ca^{2+}/Mg^{2+} enriched solution having great potential for the food and pharmaceutical sectors. Moreover, the EDBM-UF process generates NaOH, which could be used to solubilize the casein or in the cleaning operation of milk manufacturing equipment;

• A Life Cycle Assessment of the electromembrane process demonstrated that this novel process has the potential to reduce the environmental burdens of caseinate powder manufacturing by about 10 units of percentage compared to the conventional chemical (acid/base) method, mainly due to a more efficient precipitation process, *i.e.* higher quantity of precipitated casein per quantity of skim milk. Nevertheless, milk production accounts for more than 95% of the overall impact of caseinate powder production;

• The performed sensitivity analysis assuming an electricity supply from the region with the most carbon intensive electricity mix (Asia without China) confirmed the conclusion about the environmental preference of the EDBM-UF process over the acid/base process.

Thus, future studies should focus on further improving the eco-efficiency of the EDBM-UF process by better characterizing the co-products of caseinate production (NaOH, lactose solution and Ca^{2+}/Mg^{2+} enriched solution) and therefore offering the opportunity to allocate the environmental burdens of the process across the valuable co-products.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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