



Recovery and purification of lactose from whey

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ABSTRACT

One of the main problems associated with the manufacturing of cheese is the production of whey, which causes environmental pollution due to its high concentration of dissolved organic substances, mostly proteins, fat and lactose. The search for economically viable and appropriate alternative uses of whey proteins and lactose is of fundamental importance. This work investigated four integrated processes, incorporating microfiltration, ultrafiltration, ion exchange, reverse osmosis and spray-drying, for the separation and purification of lactose from whey. The recovery and the purity of the final lactose product recovered were evaluated. It was shown that the process comprising of microfiltration (nominal pore size 0.2 μm), ultrafiltration UF3 (molecular weight cut-off 5 kDa), ion exchange and reverse osmosis, a lactose purity of 99.8% (including 4.2% of galactose/glucose) and an overall lactose recovery of 74% were obtained. The lactose left over in the retentate and trapped in the membrane modules and ion exchange columns could be further recovered to improve the overall recovery.

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1. Introduction

Currently, there is growing interest in new applications of whey and its derivatives for various food products with improved quality that are beneficial to health (i.e., as functional foods or nutraceutical products). A good deal of research has been carried out to investigate the effects of these new products on human health, the control and prevention of such diseases as osteoporosis, cancer, and vascular problems [1,2]. The drug and food industries have a great interest in developing products using whey and its derivatives as raw material sources.

Brazil is the world's sixth largest producer of milk and one of the largest producers of cheese, with a production of 580,000 tons in 2007 [3]. On average, about 10 L of milk is used to make 1 kg of cheese, resulting in a by-product of 8–9 L of whey. This is equivalent to 5 million tons of whey produced annually. In spite of the high organic matter content, cheese whey is mostly disposed of as wastewater directly to the sewage or rivers in Brazil, and it is occasionally used for animal feed to avoid the waste treatment costs. The disposal of large amounts of whey without the proper pre-treatment not only poses an environmental problem but also

represents an economic loss of this noble by-product for value-added commodities [4,5].

Some of the most important components of whey are lactose and soluble proteins. Typically, cheese whey contains 5–6% lactose, 0.8–1% protein and 0.06% fat. It is one of the most valuable biological sources of proteins (representing about 20% total proteins in milk), which have attracted interest for potential use in human diet products with specific functional properties. On the other hand, lactose can be used in the food and drug industries. For instance, lactose has found use as a supplement in baby formulas and as an excipient for pharmaceutical products, and it can also contribute to the color and taste in bakery and pastry products. In addition, lactose can be used for production of glucose and galactose by hydrolysis. Compared to other carbohydrates, lactose has a low caloric value, low glycemic index, and low cariogenicity [6]. The favorable characteristics of plasticity, compressibility, lubrication, firmness and light flavor make it apt for use in the manufacturing and coating of pills and tablets in the drug industry [7]. The potential use of lactose and its derivatives as a value-added product is well recognized [8–11]. The patents presented by Clodt and Lehman [15] and Theoleyre [16] demonstrate the feasibility of recovering proteins and lactose from cheese whey. The former patent uses ultrafiltration membrane to separate protein, and the reverse osmosis to increase lactose concentration to facilitate the evaporation of water and the crystallization of lactose. The latter patent is based on the partial removal of polyvalent cations present in the lactoserum to improve evaporation of water and crystallization of lactose.

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Several technologies are available for cheese whey processing to concentrate and fractionate whey proteins and to treat the wastewater, including membrane technologies [12–14]. While most processes focus on concentration and fractionation of cheese whey proteins, one should consider the recovery of lactose as well because of the significance of lactose in the overall activities of whey utilization and waste disposal. Lactose is a major component in whey, and the recovery of lactose from whey will thus help not only improve the economics of whey utilization but also minimize pollution problems as the lactose recovery itself will reduce the biochemical oxygen demand of the waste.

The main objective of the present work was to recover and purify lactose from cheese whey using integrated processes involving microfiltration, ultrafiltration, ion exchange, reverse osmosis, and spray-drying. Different process configurations of membrane processes were examined. The recovery and the purity of the lactose recovered using these processes were evaluated.

2. Experimental

This work was carried out using four different process integrations, as shown in Fig. 1. They are designated as processes P1–P4, respectively. Ultrafiltration was mainly used to remove fat and proteins, and ion exchange and reverse osmosis were used to purify and concentrate lactose, followed by spray-drying to obtain high-purity lactose powders.

The basic process steps are illustrated in P1. The cheese whey was first centrifugated to remove suspended particulate matter and then subjected to ultrafiltration to remove fat and proteins. The ultrafiltered solution was subjected to ion exchange to remove color and minerals prior to concentration with reverse osmosis. Finally, the concentrated lactose solution was spray-dried to produce fine powders with a mean particle size of 4.54 μm . The concentrated whey proteins retained by the ultrafiltration could be recovered as well, but the protein recovery was beyond the scope of the present study. P2–P4 are modified processes of P1, where a second ultrafiltration stage (including ultrafiltration in the diafiltration mode) or an additional microfiltration pre-treatment was implemented to improve the product yield and purity. In processes P1–P3, after centrifugation, the whey was ultrafiltered with UF1 (MWCO 500 kDa). The ultrafiltered whey was further treated with another ultrafiltration (UF2 or UF3) of smaller molecular weight cut-off in processes P2 and P3, where a diafiltration mode of ultrafiltration was used as well to improve the product recovery. Instead of using UF1, process P4 used microfiltration prior to ultrafiltration treatment with UF3. The characteristics of the membranes used in this study were shown in Table 1. The equipment and procedure for the various process components are shown below:

Centrifugation: The centrifugation was carried out using a CHIBRAS centrifuge (model CB-26Y) at 1500 rpm and at a flow rate of 12.5 L/min.

Ultrafiltration UF1: The centrifuged cheese whey was treated using UF1 ultrafiltration pilot unit with a feed capacity of 1000 L. Hollow fiber polyethersulfone membranes (ROMICON PM-500 – 0720196, Koch Membrane Systems Inc.) with a nominal molecular weight cut-off of 500 kDa and a permeation area of 5.0 m^2 were used in this unit. About 375 L of centrifuged whey was ultrafiltered at room temperature (25 °C) and at a recycle flow rate of 200 L/h to reach a volume concentration of 4. The transmembrane operating pressure was 1.5 bar.

Ultrafiltration UF2: UF2 was employed only in P2. It was an experimental unit equipped with a spiral wound polyethersulfone membrane (HFM-131, Koch Membrane Systems Inc.) with molecular weight cut-off of 5–8 kDa and a filtration area of 0.28 m^2 . The unit UF2 was operated at room temperature and at a transmem-

brane pressure of 1 bar. 8 L of whey solution from UF1 was treated with UF2 to obtain 4 L of retentate and 4 L of permeate, which corresponded to a volume concentration factor of 2. To further recover the lactose present in the UF2 retentate, 4 L of deionized water was added and re-ultrafiltration of the diluted retentate was conducted under the diafiltration (DF) mode at the same operating conditions.

Ultrafiltration UF3: UF3 was a Millipore lab unit equipped with a polyethersulfone membrane (Pelicon XL 50 cassette filter) with a molecular weight cut-off of 5 kDa and a filtration area of 0.005 m^2 . The permeate (0.5 L) from UF1 was ultrafiltered using UF3 in process P3 at room temperature and a transmembrane pressure of 1.4 bar to reach a volume concentration factor of 2, and the retentate was then diafiltered after dilution with an equal volume (i.e., 0.25 L) of water. UF3 was also used in process P4 to ultrafilter the permeate from microfiltration under same operating conditions.

Microfiltration MF: Microfiltration was used in P4 as a pre-treatment prior to ultrafiltration. The MF unit (Netzsch, model 027.06-1C1/07-0005/Al) contained two Filtanium membranes (Tami Industries) in series arrangement with a permeation area of 0.0132 m^2 . The membrane, made of TiO_2 , had a tubular geometry with seven channels. The nominal average pore diameter of the membrane was 0.2 μm . MF was performed with 6 L of whey solution at room temperature and a transmembrane pressure of 1.5 bar to reach a volume concentration factor of 4, and retentate was then diafiltered using the same initial volume.

Ion exchange: Two packed columns filled with strongly acidic cation exchange resin (Amberlite 252Na) and weakly basic anion exchange resin (Amberlite IRA96SB), respectively, were used for demineralization and decolorization of the lactose recovered. Both resins were supplied by Rohm and Haas, and their harmonic mean particle sizes are 590–840 and 440–590 μm , respectively. The packed columns measured 50 cm long and 3 cm in diameter. The ion exchange was carried out by running the solution first through the cationic column and then through the anionic column at a mean percolation flow rate of 12 mL/min. During this step, the pH, °Brix and absorbance were measured at every percolated 0.5 BV (bed volume). The ion exchange resins were regenerated periodically with a 10% HCl and 10% NaOH for the cationic and anionic resins respectively according to the manufacturer's specifications. Before regeneration, the resins were thoroughly rinsed with distilled water and kept in 4% salt solution for at least 40 min. The regeneration was carried out for every 2 L of treated solution.

Reverse osmosis RO: The lactose solution was concentrated by reverse osmosis using a Millipore Helicon RO-4 spiral wound polyamide membrane module, which had a filtration area of 0.4 m^2 . The reverse osmosis was carried out at room temperature and 10 bar.

Spray-drying: The concentrated lactose solutions (i.e., the retentate from reverse osmosis) were dried in a Buchi B-191 spray drier at a circulation rate of 1.0 mL/min. The inlet and outlet temperatures during drying were maintained at 170 and 105 °C, respectively.

In this work, acid cheese whey from a local dairy company was used the raw material. In the evaluation of various process steps, the protein and lactose contents, color, the contents of ash and soluble solids were all measured. All the sample analyses were carried out in triplicate unless specified otherwise.

The protein concentration was determined by the Lowry protein assay method [17], which is based on the interaction of proteins with a phenol reagent and copper in alkaline conditions. The lactose content was evaluated by measuring the contents of the reducing sugar using the dinitrosalicylic colorimetric method [18]. The pH was measured with a pH meter (Digimed DM 20) and the soluble solid content was determined with a Shimadzu refractometer. The percent color removal was determined using a Micronal spectrophotometer at a wavelength of 420 nm, and the ash content was measured in duplicate according to the standards of Adolfo Lutz

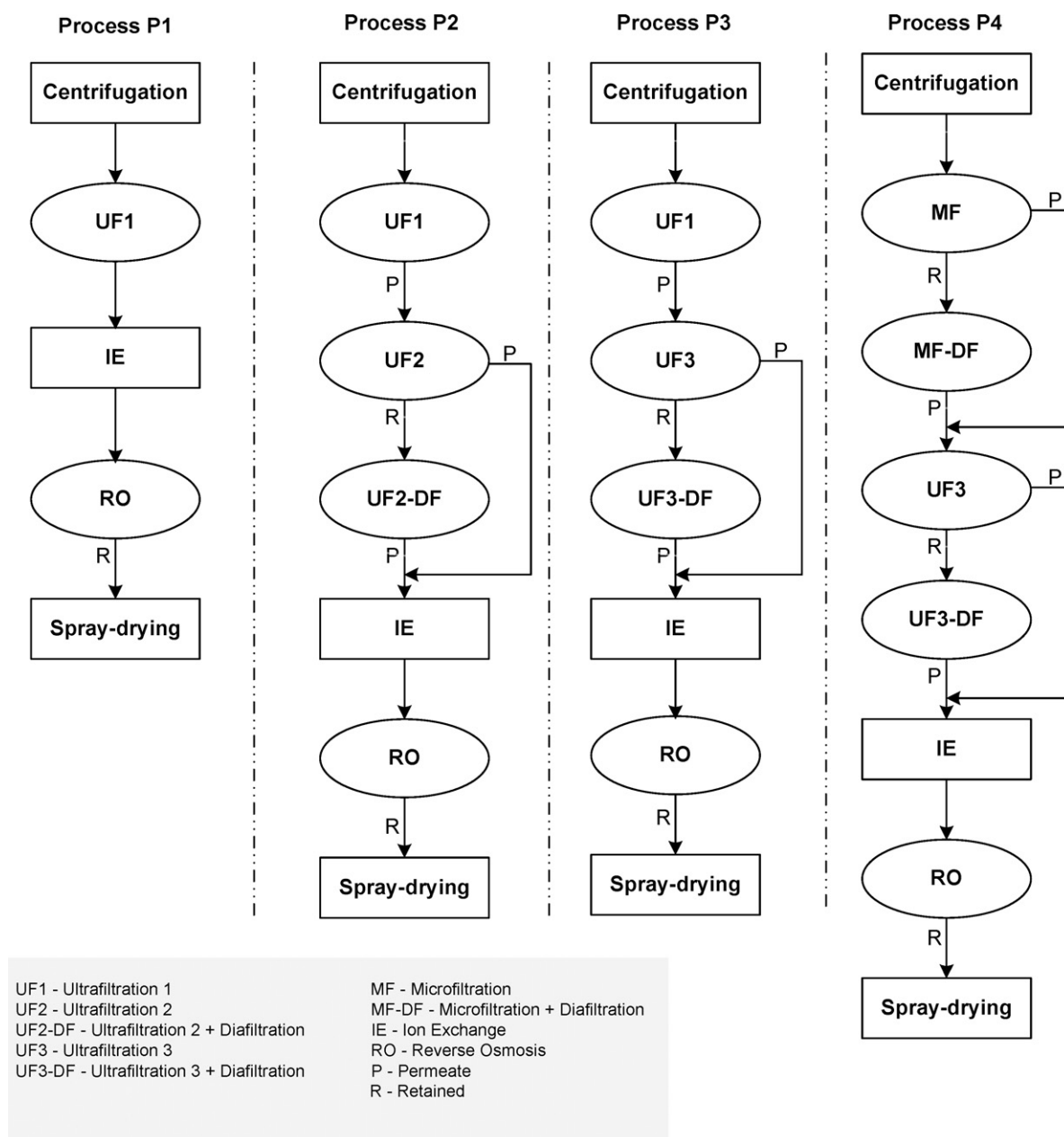


Fig. 1. Production of lactose from cheese whey using different process integrations.

Institute [19]. The fat content was determined in duplicate by the ether extraction method in an acid medium [20]. The turbidity was measured using a turbidity meter (HACH DR/2010) at a wavelength of 860 nm.

Powder lactose was also analyzed for moisture content following the standard methods [19]. The purity of powder lactose was determined by a high performance liquid chromatography (HPLC)

equipped with a Merck NH_2 LiChrosorb column (150 mm \times 4.6 mm I.D.) with an acetonitrile/water solution (80:20 by volume) as the mobile phase (flow rate 1.0 mL/min).

For ultrafiltration and microfiltration, the volumetric concentration factor (F_C) was defined as:

$$F_C = \frac{V}{V_C} \quad (1)$$

Table 1
Specifications of membranes used in the study.

Membrane	Membrane materials	Module configuration	Membrane rating	Manufacturer and model	Operating temperature (°C)	Operating pressure (bar)
UF1	Polyethersulfone	Hollow fiber	MWCO 500 kDa	Koch (PM-500)	25	1.5
UF2	Polyethersulfone	Spiral wound	MWCO 5–8 kDa	Koch (HFM-131)	25	1.0
UF3	Polyethersulfone	Cassette	MWCO 5 kDa	Millipore (Pelicon XL 50)	25	1.4
MF	Titanium oxide	Tubular (with 7 channels)	Nominal pore diameter 0.2 μm	Tami Industries (Filtanium)	25	1.5
RO	Polyamide	Spiral wound	–	Millipore (Helicon RO-4)	25	10

Table 2
Performance of UF1 for lactose separation from protein.

	Proteins (g/L)	Lactose (g/L)
Feed	6.62	40.90
Permeate	2.47	34.75
Retentate	12.66	40.65
Rejection coefficient	0.805	0.145

where F_C is the concentration factor, V is the initial feed volume, and V_C is the final volume of the retentate. In order to maintain high flux of permeate the volumetric concentration factors used in experiments were lower than 4.

The retention or permeation of the solute component (i.e., protein and lactose) by the membrane during filtration was characterized by the rejection coefficient when a determined volumetric concentration factor was reached [21].

$$R = \left(1 - \frac{C_P}{C_R}\right) \quad (2)$$

where C_P and C_R are the solute concentrations in the permeate and retentate, respectively.

The percent color removal by the ion exchange process was evaluated by:

$$\% \text{ Color removal} = \left[1 - \frac{(A_{420})_a}{(A_{420})_b}\right] \times 100\% \quad (3)$$

where $(A_{420})_b$ and $(A_{420})_a$ are respectively the absorbance of the sample at a wavelength of 420 nm before and after the ion exchange process.

3. Results and discussion

The decrease in permeate flow rate with time due to concentration polarization and membrane fouling were described in [22,23], where the fouling coefficient of the membranes were determined. This work focuses on the performance of the integrated processes for lactose recovery and purification. Table 2 shows the protein and lactose contents of the feed, permeate, and retentate samples of ultrafiltration UF1. The protein and lactose coefficient of rejection were 0.805 and 0.145, respectively. A lactose to protein mass ratio of 14.1 was obtained in the resulting permeate.

In process P1, the UF1 permeate was directly treated by the ion exchange columns. The percent variations in the contents of soluble solid, proteins and lactose content after the sequential ion exchange processes were 14%, 53% and 11% respectively. Not only was a high color removal rate of 94% observed, the ash content was also reduced significantly (about 80%). This is understandable because the cation and anion exchange steps would remove the mineral salts that constitute the ash effectively. Note that the ion exchange also resulted in reductions in the protein and lactose contents. The ion exchange eluate (about 3.5 L) was concentrated by the reverse osmosis unit at a volume concentration factor of 3.4, achieving a soluble solid content (°Brix) of 11.7 in the retentate. A white lactose powder with 88.5% purity was obtained by spray-drying of the reverse osmosis concentrate.

In consideration that the molecular weight of lactose (342 g/mol) is much lower than the molecular weights of the whey proteins with the most prevalent proteins being β -lactoglobulin, α -lactalbumin and immunoglobulins, it was decided to refine the UF1 permeate using a second stage of ultrafiltration with a membrane having a “tighter” structure in an attempt to improve the purity of the lactose product recovered. In process P2, the permeate solution from UF1 was further treated by ultrafiltration UF2 (molecular weight cut-off 5–8 kDa), followed by an additional diafiltration step (UF2-DF) to improve the lactose recovery. The contents of protein and lactose in the feed, permeate and retentate, and retention

Table 3
Performance of UF2 for lactose separation from protein in normal filtration and diafiltration modes in process P2.

	Proteins (g/L)	Lactose (g/L)
<i>Normal ultrafiltration—UF2</i>		
Feed	2.47	34.75
Permeate	0.93	33.76
Retentate	2.12	34.90
Rejection coefficient	0.561	0.032
<i>Diafiltration—UF2-DF</i>		
Feed	1.06	17.45
Permeate	0.76	19.75
Retentate	2.14	20.03
Rejection coefficient	0.645	0.047

Overall concentration of permeate: protein 0.85 g/L and lactose 26.73 g/L.

coefficients in the normal ultrafiltration mode (UF2) and diafiltration mode (UF2-DF) are shown in Table 3. It was shown that in the normal operating mode the rejection coefficient for lactose and protein were 0.03 and 0.56 in UF2, respectively. In the diafiltration operation these coefficients were similar. The effectiveness of UF2 is clearly demonstrated as the lactose to protein mass ratio in the overall permeate collected from the UF2 ultrafiltration/diafiltration has reached to 31.5, which is substantially higher than that of the original UF1 permeate (i.e., 14.1). The color and ash contents were significantly reduced by the ion exchange, 96% and 70% respectively. The percentage reduction in the content of the proteins, lactose and soluble solids were 80%, 9% and 33% respectively. When 5.5 L of the ion exchange eluate with a °Brix of 3.1 was concentrated in the reverse osmosis system a final volume of 0.88 L, a °Brix of 11.6 was obtained. The lactose purity was determined to be 97.6%, which was indeed higher than the purity of the lactose product from process P1 (i.e., 88.53%). The increased purity is attributed to the incorporation of UF2 in the process, which contributed to the refining of lactose.

A previous study showed that membrane UF3 (molecular weight cut-off 5 kDa) generally retained proteins better (with a rejection coefficient of 0.83) than UF2 [22]. Thus, it would be of interest to evaluate whether the use of the smaller molecular cut-off membrane UF3 would improve the purify of the lactose recovered. In another process, ultrafiltration membrane UF3 was substituted for UF2 in process P2. This process, designated as P3, was essentially the same as process P2 except that the second stage of the ultrafiltration had a smaller molecular weight cut-off. Table 4 shows the performance of UF3 for lactose separation from protein in both the normal ultrafiltration and the diafiltration modes. Comparing with the data in Table 3, it appears that UF3 indeed tends to show a better retention for protein (high rejection coefficient) than UF2, which has higher cut-off than UF3.

The effectiveness of UF3 in process P3 in terms of lactose to protein mass ratio in the overall permeate has reached to 33.4, which is substantially higher than the correspondent (31.4) in.

Table 4
Performance of UF3 for lactose separation from protein in normal filtration and diafiltration modes in process P3.

	Proteins (g/L)	Lactose (g/L)
<i>Normal ultrafiltration—UF3</i>		
Feed	2.47	34.75
Permeate	0.81	33.19
Retentate	3.70	33.80
Rejection coefficient	0.78	0.018
<i>Diafiltration—UF3-DF</i>		
Feed	1.85	16.90
Permeate	0.62	14.96
Retentate	2.95	15.50
Rejection coefficient	0.79	0.035

Overall concentration of permeate: protein 0.72 g/L and lactose 24.08 g/L.

Table 5

Performance of MF for lactose separation from protein in normal filtration and diafiltration modes in process P4.

	Proteins (g/L)	Lactose (g/L)
<i>Microfiltration—MF</i>		
Feed	7.31	50.84
Permeate	6.10	46.83
Retentate	8.42	50.07
Rejection coefficient	0.275	0.065
<i>Diafiltration—MF-DF</i>		
Feed	2.11	12.52
Permeate	1.98	12.38
Retentate	2.47	12.55
Rejection coefficient	0.198	0.013

Overall concentration of permeate: protein 4.00 g/L and lactose 29.61 g/L.

As a result, the ion exchange performance similar to that in process P2 was observed in terms of color and ash removals, protein and lactose losses as well as the soluble solid content. The final spray-dried lactose powder had a purity of 98.3%, which is statistically the same as the lactose product from process P2. Therefore, process P3 is not superior to process P2 from a product purity point of view. It may be mentioned that the lactose/protein mass ratio in the UF3 permeate can be increased by eliminating the diafiltration step, which could help improve the product lactose purity provided the remaining proteins could be removed by the ion exchange, but the lactose recovery will be compromised.

Note that UF1 used in processes P1–P3 had a considerably large molecular weight cut-off (i.e., 500 kDa). Process P4 examines what the overall process performance would be if UF1 was substituted with a microfiltration MF while keeping UF3 in the process. In process P4 a different batch of whey solution was used where the protein and lactose concentrations were 10–24% higher than the whey solutions used in processes P1–P3. This difference was due to seasonal characteristic of milk, which is reflected on the whey.

Table 5 shows the microfiltration performance for lactose/protein separation under the normal filtration and diafiltration modes. As expected, the protein retention by MF is much lower than that achieved by UF1. Unlike the ultrafiltration process where a significant protein retention was achieved in the diafiltration mode, the microfiltration appears to exhibit a lower protein retention in diafiltration than in normal filtration mode. Nevertheless, when the MF permeate was subsequently treated with UF3 (in process P4), the protein and lactose rejection coefficients were respectively 0.92 and 0.11, as shown in Table 6. The lactose remaining in UF3 residue was recovered after the diafiltration step (UF3-DF). In this process P4, the lactose to protein mass ratio in the overall permeate collected from the UF3 ultrafiltration/diafiltration has reached to 44.5. The mixed permeate of UF3 and UF3-DF was subjected to ion exchange treatment, and Table 7 summarizes the

Table 6

Performance of UF3 for lactose separation from protein in normal filtration and diafiltration modes in process P4.

	Proteins (g/L)	Lactose (g/L)
<i>Normal ultrafiltration—UF3</i>		
Feed	4.00	29.61
Permeate	0.50	27.54
Retentate	6.12	30.77
Rejection coefficient	0.92	0.11
<i>Diafiltration—UF3-DF</i>		
Feed	3.06	15.39
Permeate	0.43	14.27
Retentate	5.40	15.65
Rejection coefficient	0.92	0.088

Overall concentration of permeate: protein 0.47 g/L and lactose 20.91 g/L.

Table 7

Protein and lactose contents, pH, °Brix and color before and after ion exchange treatment in process P4.

	Inlet	Outlet
pH	4.39	10.69
°Brix	2.40	1.50
Color (420 nm)	0.008	0.000
Proteins (g/L)	0.51	0.062
Lactose (g/L)	20.91	16.84

The ion exchange columns were arranged in series: cationic column + anionic column.

protein and lactose concentrations as well as other quantities for the inlet and outlet streams. It is shown that a complete color removal was achieved, and the removal rate of soluble solids was approximately 38% (°Brix). At the same time, the protein and lactose loss, which were 88% and 19%, respectively, were shown to be slightly higher than the previous alternative processes. After the ion exchange, 4.2 L of the eluate was concentrated by reverse osmosis to a final volume of 0.74 L with 5.9 °Brix. The concentrate lactose solution was spray-dried, producing a white powder with 99.8% purity.

For the sake of comparison, Table 8 summarizes the composition details of the final lactose powder produced in the four processes; the industrial-grade lactose obtained by evaporation and crystallization was also included in the table [24]. In this table standard deviation are presented and means are compared according to Tukey test with 95% of confidence. It should be pointed out that the overall content of lactose obtained in process P2–P4 are statistically equal but, higher than the correspondent value for process P1 and even slightly higher than the lactose form industrial-grade.

Data demonstrated that process P1, which is the simplest process with less separation units, produces a poor lactose product. The inclusion of a unit UF2 with its diafiltration in process P2 improves the quality of the lactose, reaching the industrial-grade of purity for lactose as well as decreasing the proteins, fat and ashes contents in lactose.

It can be seen that although the whey proteins had been largely removed in process P3, the fat content was still relatively high when compared to that of industrial-grade lactose. The substitution of UF1 with MF plus DF in process P4 resulted in improved the quality in the lactose product in terms of the impurities (i.e., protein, fat, ash, and calcium). In all the processes the chromatogram of the powder lactose showed two peaks; the first peak is related to galactose and/or glucose, as these two oligosaccharides have similar retention times. The presence of hydrolyzed lactose in whey is very common because of the action of natural enzymes or other enzymes used in cheese production. Because of this in all analysis, these two compounds were considered together with plain lactose.

The results presented in Table 8 demonstrate clearly the effectiveness of using the integrated processes for lactose separation and purification from cheese whey, especially process P4 for producing the best quality of lactose among the four processes investigated. This is evident by the lower values of fat, ashes and calcium present in the lactose recovered using process P4. The high quality of lactose recovered using the separation process steps in P4 allows for lactose to be used directly as a raw material to produce such highly value-added products as lactitol and tagatose [6,10].

From a practical application point of view, besides lactose purity, the recovery of lactose from whey is also important. Tables 9 and 10 show the amounts of lactose lost at various steps of the process for processes P3 and P4, which produced the best purity of the finished product. These results, calculated from mass balance, were based on 100 kg of lactose present in the centrifuged cheese whey solution, which corresponded to a volume of 2.0–2.5 m³. Theoretically, micro- and ultrafiltration membranes should not retain lactose if

Table 8
Properties and purity of powder lactose obtained in P1–P4.

Components (%)	Process				
	Industrial	P1	P2	P3	P4
Lactose*	98.0 (min)	88.53 ± 1.91 ^a	97.67 ± 1.01 ^b	98.33 ± 0.42 ^b	99.83 ± 0.21 ^b
Proteins	0.50–1.00	6.63 ± 0.47 ^c	0.94 ± 0.15 ^b	0.59 ± 0.09 ^{a,b}	0.00 ^a
Fat	0.10	0.75 ± 0.15 ^b	0.66 ± 0.11 ^b	0.47 ± 0.11 ^b	0.11 ± 0.04 ^a
Ashes	0.10–0.50	0.75 ± 0.13 ^b	0.64 ± 0.12 ^b	0.58 ± 0.08 ^b	0.10 ± 0.04 ^a
Calcium	–	–	0.083 ± 0.042 ^b	0.060 ± 0.044 ^{a,b}	0.002 ± 0.002 ^a

Note: For a row, the same letter does not show significant difference among means ($p > 0.05$), according to Tukey test. Mean ± standard deviation (triplicate).

* Hydrolyzed lactose (galactose/glucose) included.

Table 9
Lactose loss (in kg) for process P3 based on 100 kg of lactose present initially in whey.

Stages	Basis: initial amount of lactose = 100 kg			
	Retained	Permeate	Membrane/column	Total
UF1 (500 kDa)	26.50	–	11.20	37.70
UF3 + UF3-DF (5 kDa)	13.90	–	2.99	16.89
Ion exchange	–	–	6.79	6.79
Reverse osmosis	–	1.22	2.00	3.21
Total	40.40	1.22	22.98	64.60

the molecular weight cut-off is very sharp. In an ideal membrane, the pore size should be uniform, but in reality there is always a pore size distribution which will lead to diffuse cut-off in membrane filtration. For lactose recovery where the permeate is of interest, the sharpness of the cut-off will directly affect the final product quality. In addition, during the filtration process, the retained protein molecules will accumulate on the membrane surface, which will also affects the passage of lactose molecules. Due to surface fouling and gel layer formation of protein on the membrane surface, molecules such as lactose that are smaller than the membrane cut-off can be partially retained. This issue is particularly important for cheese whey filtration because lactose is readily attached to fats and proteins. It is shown that about 40% and 17% of total lactose remained in the retentate for processes P3 and P4, respectively, and the retentate is the biggest source of lactose loss. For both processes P3 and P4, the lactose left over in the retentate represents about 65% of the total lactose loss. Obviously, the retentate should be further treated, (e.g., with diafiltration) in order to improve the lactose recovery. Lactose trapped in the membrane modules (in particular ultrafiltration modules) and the ion exchange columns are another major source of lactose loss. In general, process P4 performed better than process P3; while the purities of final lactose products from both processes were very similar, the mass balance (Tables 9 and 10) demonstrates that the process P4 had an overall recovery of 74% of lactose, which is much higher than a lactose recovery of 35% achieved in process P3. It is worth noting that the materials lost in the membrane/columns in the processes can be recovered by, for example, rinse and back flushes. For practical applications, the overall lactose recovery can be improved by recycling and refining the lactose retained in the retentate and the membrane/columns.

Table 10
Lactose loss (in kg) for process P4 based on 100 kg of lactose present initially in whey.

Stages	Basis: initial amount of lactose = 100 kg			
	Retained	Permeate	Membrane/column	Total
MF + MF-DF (0.2 µm)	6.17	–	0.19	6.36
UF3 + UF3-DF (5 kDa)	11.29	–	0.62	11.91
Ion exchange	–	–	5.87	5.87
Reverse osmosis	–	0.67	1.58	2.25
Total	17.45	0.67	8.27	26.39

It may be mentioned that a 500 kDa molecular weight cut-off membrane was used in process P3 to retain a large portion of the proteins and fat. If the proteins were also to be recovered as a second by-product, it is unlikely to obtain good protein quality due to the high fat content. Even if the protein-enriched retentate solution had been diafiltrated, the quality of the protein obtained would not be improved because of the presence of fat. In this regard, process P4 was more advantageous as the microfiltration (MF) stage in P4 allowed for the separation of fat from proteins and gave a larger flows in comparison to the UF1 stage.

4. Conclusions

The paper presented a technology to obtain lactose, which is co-product of the protein from whey and for this reason should be integrated with a protein recovery process. In addition, energy savings can be achieved using membranes to concentrate lactose in comparison to conventional process of evaporation to obtain lactose.

Four integrated processes were studied to separate and purify lactose from cheese whey. The processes were comprised of microfiltration, ultrafiltration, ion exchange, reverse osmosis, and spray-drying. The various process configurations were examined, and the recovery and the purity of the final lactose product recovered were evaluated. It was shown that process P1, which employed a single stage ultrafiltration (UF1) with a large molecular weight cut-off (500 kDa), yielded a relatively low purity (88.5%) powder lactose. The incorporation of a second stage of ultrafiltration (UF2) with a smaller molecular weight cut-off (5–8 kDa) had resulted in an increase in the lactose purity to 97.7%. When UF2 was substituted with UF3 that had a molecular weight cut-off of 5 kDa, the impurity (i.e., protein, fat, ash, and calcium) level in the lactose recovered was lowered. Process P4, which consisted of microfiltration (MF) (nominal pore size 0.2 µm), ultrafiltration UF3, ion exchange and reverse osmosis, was shown to be the most effective among the four process configurations investigated in terms of product purity and recover rate. This process yielded a lactose purity of 99.8% (including 4.2% of galactose/glucose) and an overall recovery of 74% of lactose. The recovery could be improved by further extraction of the lactose left over in the retentate using additional diafiltration steps as well as reclaiming the lactose trapped in the membrane modules and ion exchange columns by, for example, rinses and back flushes.

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