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Recovery and purification of whey with the concentration, reincorporation and reuse Protein and Lactose

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Abstract
The recovery and purification of whey with the concentration, reincorporation and reuse Protein and Lactose were studied by coupling the two operations: Ultrafiltration with a ceramic membrane and the reverse osmosis with the organic membrane. The two operations were performed in batch mode. In the first step the ultrafiltration, was operated in one stage, and produced the best results from the point of treatment capacity and protein recovery. The ultrafiltration was concentrated the total solids and protein concentrations with around 60% of protein. In the second step the **UF+RO** combination produced the best results of COD; BOD_5 (99%) and a high concentration of the lactose with around 51% in a Volume Reduction Factor 3 (VRF3). The concentration of protein, lactose and reducing the organic load by ultrafiltration coupling with the reverse osmosis operations has zero discharge.

Keywords: Whey, Proteins, Lactose, Pollution load, Ultrafiltration, Reverse Osmosis

Introduction
Cheese Whey (CW) is a by-product of dairy industries, particularly the watery portion that is formed during the coagulation of milk casein in cheese making or in casein manufactures. Whey represents about 85-95% of the milk volume and retains 55% of milk nutrients. Among the most abundant of these nutrients are lactose (4.5-5% w/v), soluble proteins (0.6-0.8% w/v), lipids (0.4-0.5% w/v) and mineral salts (8-10% of dried extract) [1]. They also contains appreciable quantities of other components, such as lactic (0.05% w/v) and citric acids, non-protein nitrogen compounds (urea and uric acid) and B group vitamins [2].
Cheese whey represents an important source of environmental pollution due to its enormous global production rate, approximately the world whey production is over 160 million tonnes per year (estimated as 9-fold the cheese production), showing a 1–2% annual growth rate and high organic matter content, exhibiting the BOD and COD values of 50 and 80 g.L−1, respectively [3,4]. Utilization of whey towards the production of value-added products will be economical and environmentally desirable [5].
A cost-effective disposal of whey is a major problem for cheese manufacturers despite of the variety of available techniques [6]. Possible waste minimisation routes have been described by, e.g., Zall [7];
- Application of techniques minimising whey production during cheese making;
- Usage of whey as a valuable by-product in the food industry;
- Production of petrochemicals such as methane and methanol;
- Production of lactic acid and its derivatives (potentially as green solvents);
- Production of biodegradable plastics and polymers;
- Treatment of whey like a sewage.

Considering whey as a valuable by-product seems to be a very promising future route development in membrane technology [6] .The first step in most procedures for cheese whey valorisation consists in the

recovery of the protein fraction. Whey proteins represent about 20% of the milk proteins, having a high nutritional value as well as reported health benefits and therapeutic potential [8].
Separation of whey proteins is typically achieved by Ultrafiltration or Diafiltration to produce whey protein concentrates (WPC), which have many applications in the food [3-12]. Whey proteins have also non-food uses, mainly in cosmetics and pharmaceutical products [9]. During the processing of whey for the production of WPC, high volumes of a lactose-rich stream, the permeate is also obtained. The permeate remains a major pollutant since it retains the lactose, which represents more than 70% of total whey solids and is largely responsible for the whey polluting load [1]. Therefore, the permeate creates disposal problems, in terms of volumes produced and polluting load, almost equal to the disposal of raw whey [10].
Currently, the whey production in Morocco is estimated at 100.000 l/d, causes the loss of about 700 kg of protein and 4400 kg of lactose. The objectives of this work are multiple it was the coupling several unit operations; in particular membrane processes Ultrafiltration (UF) and Reverse Osmosis (RO) to:
- Collect, concentrate and recycle proteins;
- Collect, concentrate and enhance the lactose;
- Reduce pollution load sent to treatment plants by about 50%;
- Produce water for various uses.)

2. Materials and methods

2.1. Cheese Whey

The used whey is a rejection of the plant of the dairy industry of the Meknes city specialized in the production of yoghurt and fresh cheeses. Whey was obtained following an acidic coagulation of pasteurized milk. After milk coagulation, the whey was separated by centrifugation. The protein content of the obtained whey varies following the production cycle.

2.2. Experimental and procedure

The experiments were performed on Ultrafiltration (UF) pilot plant combined with Reverse Osmosis (RO) pilot plant. The two pilots were supplied by TIA Company (Applied Industrial Technologies, France). The two operations were performed in batch mode. The (UF) pilot plant is equipped with one module contains one element. The applied pressure over the membrane can be varied from 1 to 10 bars with manual valves. The (RO) pilot is equipped with two identical modules operating in series. The two spiral wound modules are equipped with two commercial Reverse Osmosis membranes of one type BW30LE-4040. A schematic diagrams of (UF) and (RO) pilot plants are shown in Figures 1 and 2. The table 1 gives the characteristics of the (UF) ceramic membrane and (RO) organic membrane.

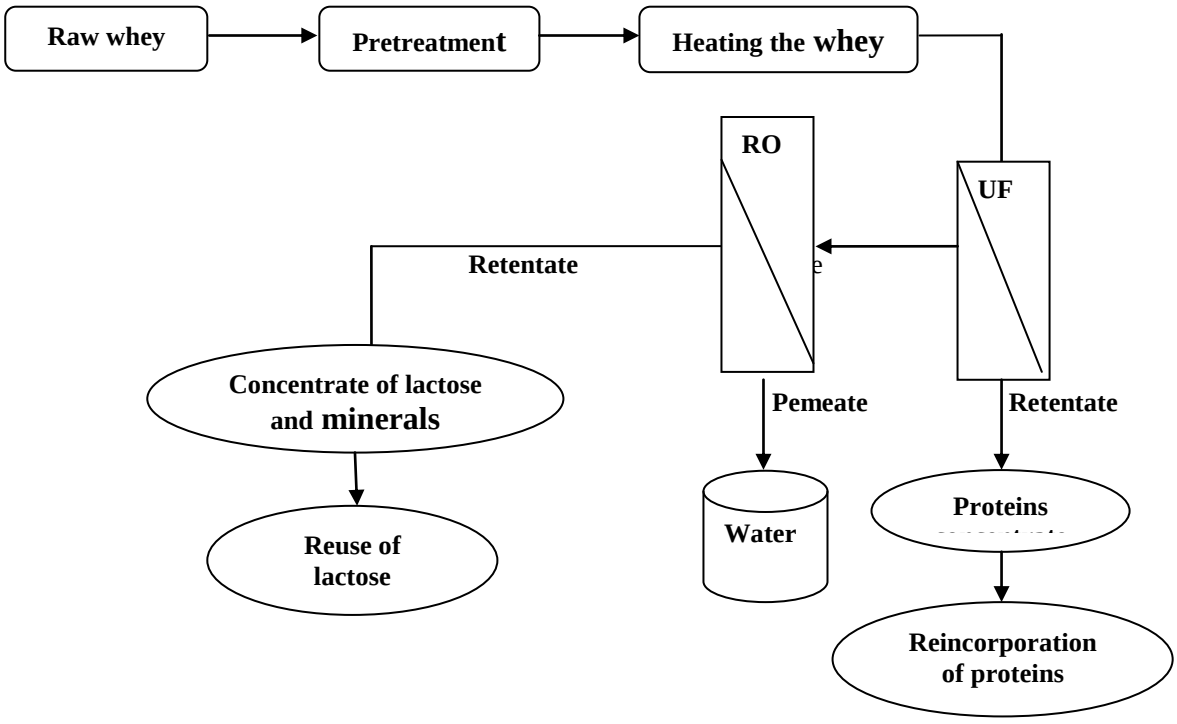

Figure 1: Treatment process

After the run, the membranes were cleaned with alkaline and acidic solutions according to the manufacturer recommendations.
The whey was firstly filtered on cartridge of 10µm to eliminate the fine casein and the residual lipids. The clarified acid whey was treated at desired temperature to increase its viscosity. The UF operation allowed the obtention of a concentrate of proteins and a permeate of lactose. The permeat UF was treated by the RO, the figure 1 show the steps of recovery and purification of whey.

Table 1: Characteristics of the used membranes

Membrane	P.max (bars)	pH	Temp max	Material
UF 50 nm	10	3- 11	100	Ceramic
BW30LE-4040	41	2- 11	45	Polyamide

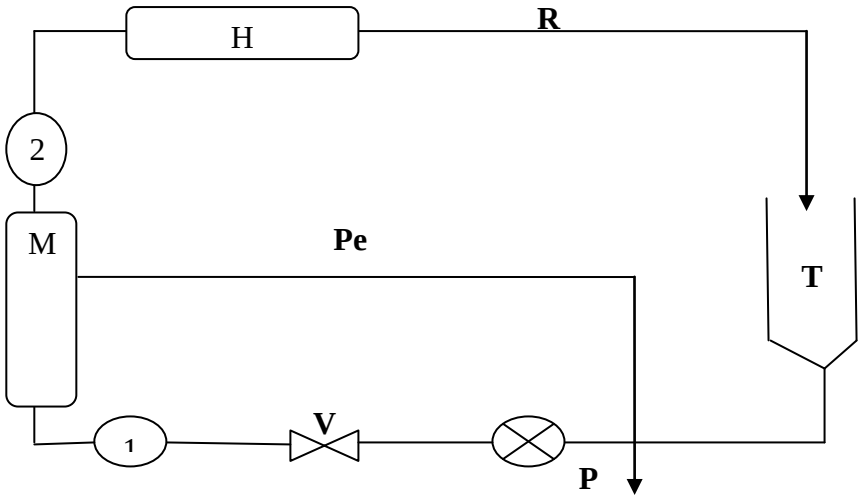

Figure 2: Diagram of the Ultrafiltration pilot plant

T: tank; **P**: feed pump; **V**: pressure regulation valves; **M**: module of ultrafiltration; **Pe**: permeate recirculation; **R**: retentate recirculation; **H**: heat exchanger; **1**: pressure sensor; **2**: temperature sensor.

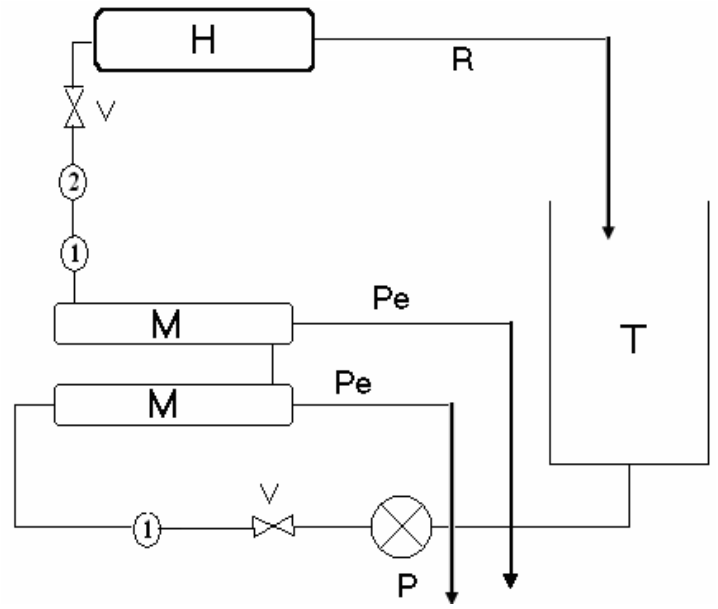

Figure 3: Diagram of the Reverse Osmosis pilot plant

T: tank; **P**: feed pump; **V**: pressure regulation valves; **M**: module of reverse osmosis; **Pe**: permeate recirculation; **R**: retentate recirculation; **H**: heat exchanger; **1**: pressure sensor; **2**: temperature sensor.

The principal followed parameters are pH, proteins, fat, conductivity, lactose, BOD5, COD, dry extract and Volume Reduction Factor (VRF). The VRF is defined as:

$$\mathbf{VRF = V0 / VR = V0 / V0\text{-}Vp}$$

Where V0 is the initial feed volume, VR and VP are respectively the retentate and permeate volumes.

2.2. Analytical methods

The proteins was determined by the Kjeldahl method using the known formula [(TN - NPN)* 6.38)] [14]. Fat was determined according to (NF V 04-214) [15]. Lactose concentration was determined by employing the DNS method for reducing sugars [13]. The Biochemical Oxygen Demand (BOD5) and Chemical Oxygen Demand (COD) were determined according standard methods [17]. Finely the pH was measured using a pH meter and the conductivity was measured using a conductivity meter.

3. Results and discussion

3.1*. Composition of acid whey*

The original whey samples used in this study were obtained from a company in the centre of Morocco which uses raw milk for production of fresh cheese. The production cycle generates approximately 70.000 L/d of whey with a loss of 18% of protein. The mean composition of the used whey is shown in Table 2. Each value in the table is a mean of triplicate assays. The protein content is of 5 g/kg and the lactose content is of 43 g/kg. The pollutant load of whey is very high corresponding to 70 g/l of COD and 47 g/l of BOD_5.

Table 2: Characteristic of acid whey

pH	4,3
Conductivity (µs/cm)	7550
Lactose (g/ kg)	43
Fat (g/ kg)	0,5
Protein (g/ kg)	5
Dry Extract (g/kg)	60.26
BOD_5 (mg/l)	47260
COD (mg/l)	70890

The performance of Ultrafiltration membranes ceramic to valorised the whey can be characterized in terms of permeate flux and yield, which parameters are determined by pressure, Volume Reduction Factor and temperature. The influence of these parameters on permeate flux was measured during the experimental runs.

3.2*. Influence of pressure*

The ceramic membrane employed was able to run with pressure maximal to 10 bars, because the limited pressure for the membrane was given by manufacturer. The influence of operating pressure ranging between 1.2 and 2.2 bars at constant temperature (45°C) and Volume Reduction Factor, on permeate flux were studied. Figure 4 give the effect of pressure on flux during ultrafiltration.

The figure 4 shows that, the permeate flux of whey increased with an increase in pressure. However the flux decreased respectively as function VRF. This phenomenon can be explained by formation the gel layer on the membrane surface, which represents an additional resistance to permeate flux [19]. This decrease is important for low pressure (1.2 bars and 2 bars).

3.3*. Influence of temperature*

The effect of temperature on the permeate flux can be understood from its effect on the properties of the feed stream. Figure 5 gives to a constant pressure (1.2 bars) the variation of flux at different VRF for the temperature, 30 C°, 40 C° and 45 C°. The analysis of the profile shows that a increasing the temperature results in a decrease in the viscosity of whey, resulting in an increase in permeate flux. Generally, the permeate flux increases with an increase in temperature; this phenomenon is attributed to high temperature increases the solute diffusivity and the rate of transport of solutes from the membrane surface [18].

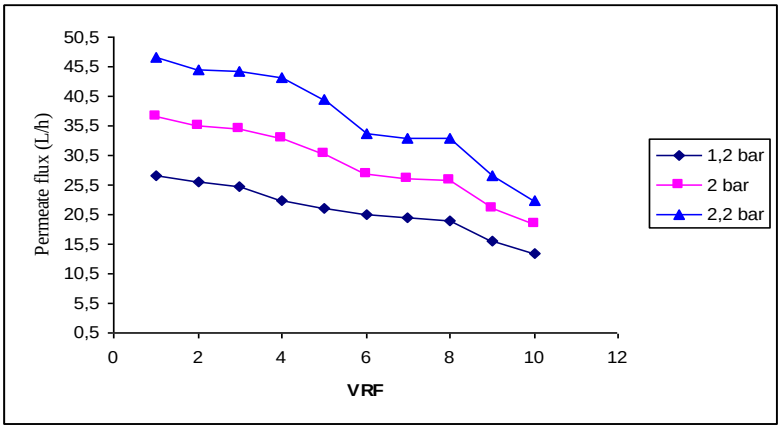

Figure 4: Influence of pressure and volume reduction factor on the permeate flux at constant temperature 45°C

However, the permeate flux decreases respectively as a function VRF, due to the presence of proteins, which may represent a significant source of membrane fouling. At higher Volume Reduction Factor (VRF) a thicker and denser deposit layer in protein is formed which reduces permeate flux until it reaches the steady-state condition [18].This decrease is important for low temperature (30 C°and 40 C°).

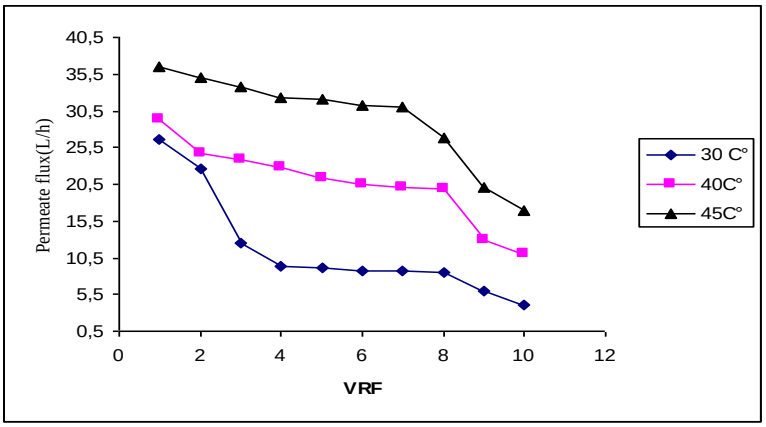

Figure 5: Influence of temperature and volume reduction factor on the permeate flux at constant pressure 1.2 bars

3.4*. Influence of the volume reduction factor (VRF)*

The study of influence of VRF was conducted on whey retentate and permeates in the experimental conditions (45C°, 2.2 bars). Figure 6, 7, 8 and 9 gives the variation of physico-chimical parameters of the whey retentate and permeate according to VRF for the membrane ceramic UF. Figure 6 shows that, lactose, minerals and pH value remained constant in the retentate for all VRF. But protein, Fat increases with increase VRF in figure 7. The protein rejection of UF was 62% at VRF9.

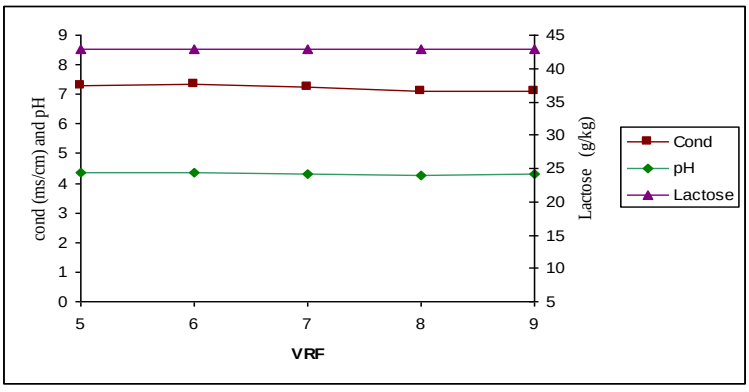

Figure 6: Evolution of lactose, conductivity and pH containing in the retentate compartment at different values of VRF

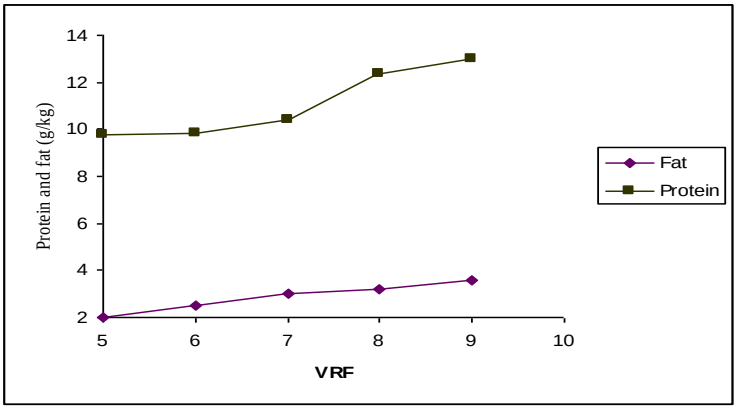

Figure 7: Evolution of protein and fat content in the retentate and Fat as function of VRF

A similar behaviour was observed in figure 8, the conductivity, pH and lactose content remained constant for different VRF in permeate. A small increase in COD and BOD_5 was observed with an increase in the VRF (fig 9). From our experiments it can be concluded that, the valorisation of whey by Ultrafiltration was optimised with used temperature 45C°, pressure 2.2 bars and VRF 8. The table 3 gives the chemical composition for the retentate and permeate after Ultrafiltration of whey at VRF8.

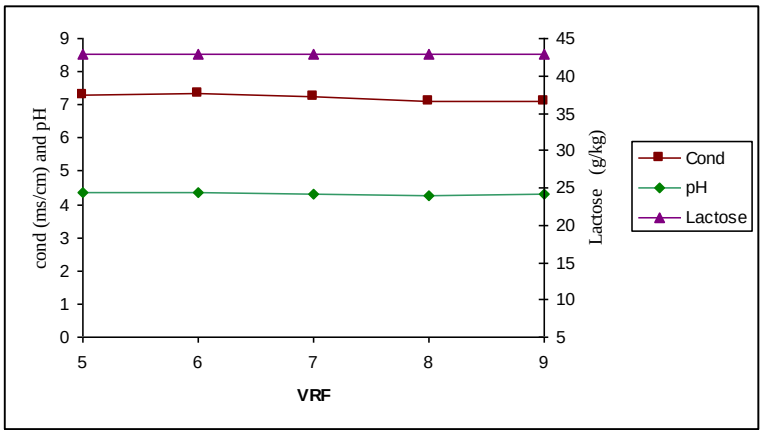

Figure 8: Evolution of lactose, conductivity and pH content in the permeate at different values of VRF

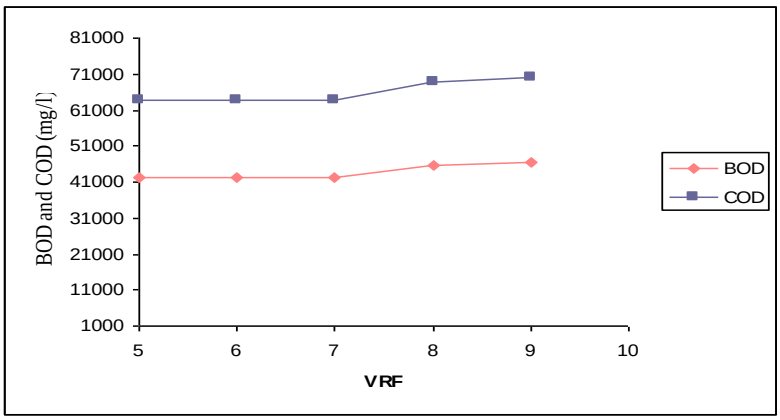

Figure 9: Evolution of BOD_5 and COD in the permeate as function of VRF

Table 3 show that, the protein rejection of UF membranes ceramic reached a values 12.34 (g/kg) at FRV8, this proteins will be denatured by heat treatment (90C°) to obtain a gelatinous product which will be reincorporated into food products. The permeate is consisted principally of lactose and minerals. This sugar is responsible for

this high load responsible for the high BOD (46 g/l) and COD (69 g/l). However, the protein recovery by Ultrafitration reduces the COD of whey only by about 3%. To reduce the polluting load, the UF permeate will be treated by Reverse Osmosis process.

Table 3: Characteristics of the retentate and permeate samples collected after UF concentration at (VRF8)

Parameters	Whey	VRF_8	
		Retentate	Permeate
pH	4.3	4.3	4.3
Conductivity (µs/cm)	7550	7450	7300
Lactose (g/kg)	43	43	43
Protein (g/kg)	5	12.34	0
Fat (g/kg)	0.4	4	0
BOD_5 (mg/l)	47260	1483	45777
COD (mg/l)	70890	2224	68666

3.5*. Treatment the UF permeate by RO*

Reverse osmosis (RO) is used for UF permeate processing including salt removal, recover the lactose from the deproteinized whey and reduce the polluting load. The table 4 gives the chemical composition for the retentate and permeate after RO operation at VRF_3 in the experimental conditions 30 bars and a temperature 25 °C.

Table 4: Characteristics of retentate and permeate samples collected after RO treatment at (VRF_3)

Parameters	$UF_{permeate}$ (VRF_8)	$RO_{permeate}$ (VRF_3)	$RO_{concentrate}$ (VRF_3)
pH	4.3	4.1	4.1
Conductivity (µs/cm)	7300	459	6841
Lactose (g/kg)	43	0	87
DOB_5 (mg/l)	45777	457	45320
COD (mg/l)	68666	619	68047

The analysis of the results shows that, a constant of pH in permeate and retentate was obtained. The conductivity decreases significantly reached a values 459 µs/cm at VRF3 in the permeate. The lactose concentration was higher in the retentate (87g/kg) .The concentrate of lactose can be used in field deferent industrial as food ingredients in infant formulas, filling or coating agent for tablets in the pharmaceutical industry and raw materials for production lactose derivatives, product value added (such as lactulose, lactitol, lactobionic acid, urea lactosyl, galacto-oligosaccharides and lactosucrose) [11] . Another major application for the lactose in RO permeates involves its use as a substrate for the production of valuable compounds by fermentation. The classical examples are ethanol (see below) and Single Cell Protein (SCP) production in yeast-based bioprocesses, although biotechnologists have proposed a multitude of alternative bioproducts [16]. Finally the values of COD and BOD_5 achieved in the permeate compartment are very low in the order 619 (mg/l), 457 (mg/l) respectively.

Conclusion

The optimisation results operating conditions of the pilot plant UF (2.2 bars, 45C° and FRV8) and treatment the UF permeate by RO is proposed in this paper. Experimental results showed that optimization and valorisation of acidic whey by ultrafiltration are feasible. This process can by concentred total solids and protein concentrations with around 60% of protein.

The treatment of whey permeates by Reverse Osmosis (RO) module (BW30LE4040) product very good results in terms of COD and BOD_5 (99%) and a high concentration of the lactose with around 51% in a Volume Reduction Factor 3 (VRF3). The valorisation of protein and reducing the organic load by ultrafiltration coupling with the reverse osmosis has zero discharge.

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