Study the performance of the organic membrane ultrafiltration on whey treatment

Sakina Belhamidi, Majdouline Larif, Hajar Qabli, Soufian Elghzizel, Hicham Jalte, Selma Chouni, Azzedine Elmidaoui

***Abstract*— Treatment of whey using the organic membrane processes namely ultrafiltration (UF) was investigated to recover the proteins for re-use. Membrane module was tested in batch mode operations and multi-stages operations employing the influence of the pH and temperature. The performance of ultrafiltration membrane can be characterized permeate flux and membrane retention, these parameters are determined by pH and temperature. The influence of these parameters on whey protein concentrate is also measured.**

The permeate flux and the protein content in the permeate and in the concentrate fractions were measured during the experimental runs. By comparing the separation behavior of the membrane for the two separation modes it was found that the investigated membrane produced the best results from the point of permeate flux, VRF and protein content in multi-stage modes in optimal condition (pH=6.5 and Temperature 50°C). The filtration characteristics were obviously influenced by the process parameters.

***Index Terms*— Whey, Proteins, Ultrafiltration, Organic membrane, Concentration Factor.**

I. INTRODUCTION

The dairy industry is one of the most important fields of both industrial and developing countries. This industry generates significant liquid waste, such as whey and butter milk whose disposal requires a large amount of capital investment [1]. Whey contains more than the half of the solids of the original whole milk, including whey protein (20% of total protein) and most of the lactose [2,3]. Since it contents organic compounds, whey can not be discharged to receiving environments.

Also, when it is considered that on cheese making about half of the total milk finds its way into the whey, it is more understandable that the processing of whey, in particular its organic constituents, is regarded as very important [3]. Therefore, recovery of valuable compounds in whey, such as protein and lactose, has received intense attention recently. However the utilization of whey towards the production of value-added products will be economical and environmentally desirable [4]. Several methods have been explored for the recovery of whey:

- Feeding cattle [5];
- Concentration and drying of whey [5];
- Energy recovery by production of bioethanol and biogas production [5];
- Obtaining sugar syrup from [6];
- Whey development by lactic yeast production [7];
- Separation and recovery of proteins [8, 9].

However the whey functional properties are usually attributed to the protein fraction [7].The most important functional properties of whey proteins are solubility, viscosity, water holding capacity, gelation, emulsification and foaming [8,9]. Indeed, whey protein fraction is a mixture of different proteins with various functional properties. The major protein fraction composed by β-lactoglobulin and α-lactalbumin account for 70% of the total proteins [10]. They possess both excellent nutritional values that are superior to those of casein and, excellent functional properties. In this case the recuperation and the concentration of whey proteins have led to the implementation of the new technologies of the membrane such as ultrafiltration (UF).

The dairy industry is one of the pioneers in the development of ultrafiltration equipment and techniques [11].Application of ultrafiltration in the dairy industry started with the separation and concentration of whey proteins from whey in 1972. The principal aim of whey ultrafiltration is to concentrate the native or pre-denatured proteins in order to obtain a protein powder with varying protein content and reduced lactose and ash content [12].

In ultrafiltration, the constituents of whey are fractionated according to molecular size. Depending on the retention characteristics of the membranes, there can be a significant difference in the nutritive power of the retentate and permeate. The protein and fat fractions are retained very well (virtually completely) in the retentate, while the lactose, minerals and vitamins are divided between the retentate and the permeate [13].

Among the existing work on whey UF processes, Atra et al. [14] studied protein rejection and permeate flux at operating pressures of 1–5 bar and feed flowrates of 100–400 L/h. Muller et al. [15] studied the purification of α-lactalbumin from acid whey using the tubular ceramic membranes (150–300 kDa). Cheang and Zydney[16] designed a two-stage ultrafiltration process for the purification of both α-lactalbumin and β-lactoglobulin from whey protein isolate.The major purpose of this work is to study the performance of membrane organic ultrafiltration for the whey treatment and protein recovery employing one stage and multi-stages would be economically beneficial for full-scale applications.

II. APPARATUS AND PROCEDURE

A. UF equipment

The system by which the experiments were conducted, consisted of a 200 L feed tank, stain less teel membrane housing, feed and recycling pumps, pressure indicators, and flow meters (Figure 1). The feed was pumped to the membrane module by means of a feed pump (2), the applied

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pressure over the membrane can be varied from 1 to 10 bars with manual valves (V). Table 1 gives the characteristics of the UF organic membrane.

In order to regenerate the organic membrane after whey ultrafiltration, the membranes were cleaned with alkaline and acidic solutions according to the manufacturer recommendations.

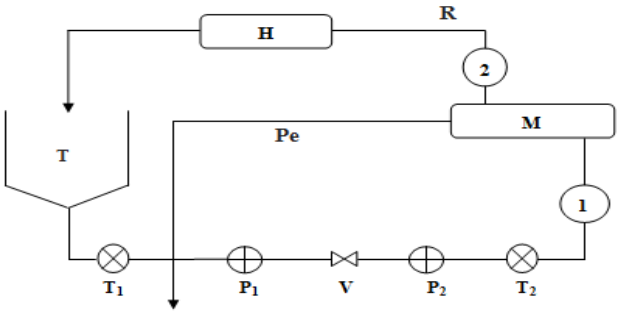


Figure1: Schematic diagram of the ultrafiltration system

T: Tank; **M**: Module of ultrafiltration, **Pe**: permeate recirculation, **R**: retentate recirculation, **E**: Heat exchanger; **P1**: feed pump; **P2**: Recirculation pump; **1** and **2**: Feeding Pump and Recirculation pump, **V**: pressure regulation valves, $\mathbf{T_1}$: Indicator of the fluid temperature inside the tank, $\mathbf{T_2}$: Indicator of the temperature of fluid inside the membrane.

Table 1: Characteristic of the used membrane

Module/type	Material	Module area (m^2)	P_{max}(bar)	pH	T_{max} (C°)
HFK-131	Polyamide	12	10	02-Nov	55

B. Feed solutions

Experiments were conducted by using acid cheese whey, whey as feed for membrane module. The original whey samples used in this study were obtained from a company specialized in the production of cheeses generate approximately 40,000 L of whey. This whey was obtained following an acidic coagulation of pasteurized milk. After milk coagulation, it was separated by centrifugation. The composition depends on milk origin and of the process coagulation of casein. Characteristics of the feed solution are given in Table 2.

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**

C. Filtration experiments

In our study, two different modes for the filtration of whey are supplied by ultrafiltration membrane: bacth mode and muti-stage mode, before membrane treatment in order to remove coarse protein particles and residual lipid, the whey was undergone a filtration on a single cartridge filter of 10 µm membrane fouling. For the batch mode, the operations of membrane module were operated under different operational conditions, namely different temperatures and different pH. Figure 2 and 3 gives the steps for the treatment of whey in the both modes.

The effectiveness of the membrane processes was determined by measuring the permeate flux during the experiments, calculating the protein and lactose rejection of the membrane module. The protein and lactose rejection percentage (R %) of the module was calculated by:

$$\mathbf{R\%= 1- (C_P/C_R)\ 100}$$

Where C_P solute concentration in the permeate (g/kg), C_R solute concentration in the retentate (g/kg). Volume Reduction Factor (VRF) obtained by the membrane module was calculated by:

$$\mathbf{VRF= V_0/V_0 - V_P}$$

Where $V_0$is the initial feed volume, V_R and V_P are the retentate and permeate volumes.

The protein fraction of samples was determined by the Kjeldahl method using the known formula [(TN - NPN)* 6.38)] [17]. Fat was determined according to (NF V 04-214) [18]. Lactose concentration was determined by employing the DNS method for reducing sugars [19]. Finely the pH was measured using a pH meter and the conductivity was measured using a conductivity meter.

a. Treatment in batch mode

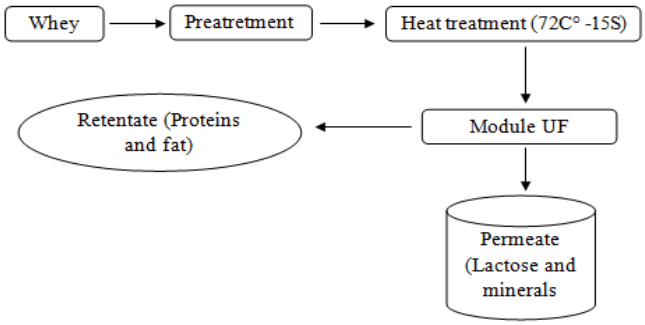
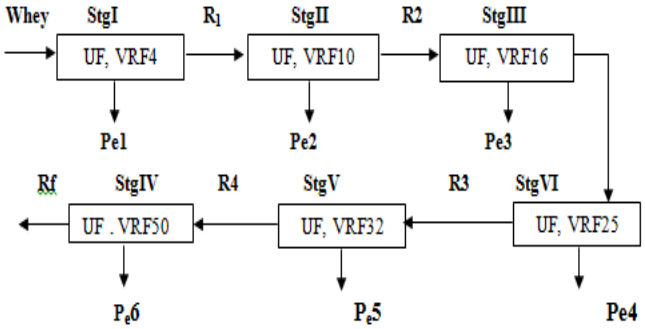


Figure 2: Schematic diagram of the concentration of whey in batch mode

b. Treatment in multi-stages mode



Stg: Stage; **R**: Retentate, **Pe**: Permeate, **Rf**: Finale retentate, **UF**: Ultrafiltration membrane; **VRF**: Volume Reduction Factor.

Figure 3: Schematic diagram of the concentration of whey in multi-stages mode

III. RESULTS AND DISCUSSION

A. Ultrafiltration of whey in batch mode

In order to study the performances of the membrane, the ultrafiltration of whey was operated in batch mode. The operational pH and temperature of UF was tested.

Figure 4 presents the variation of the permeate flux as a function of VRF, the permeate flux decreases exponentially with an increase the VRF. The flux reaches a minimum of 90 (L/h) corresponding to15 VRF. At pH=4.5, near to the isoelectric point of whey proteins, the conformation of the protein molecule and the net charge of the adsorbed protein layers at the interface [20].This phenomena corresponding to the forming of a thick and dense layer of polarization non the membrane surface as a result of fouling[21, 22].
So in these conditions, it was not possible for the raw whey to go beyond 15 VRF. In this case it was decided to study the influence of pH and the heat treatment on the permeability of the membrane (figure 5 and 6).

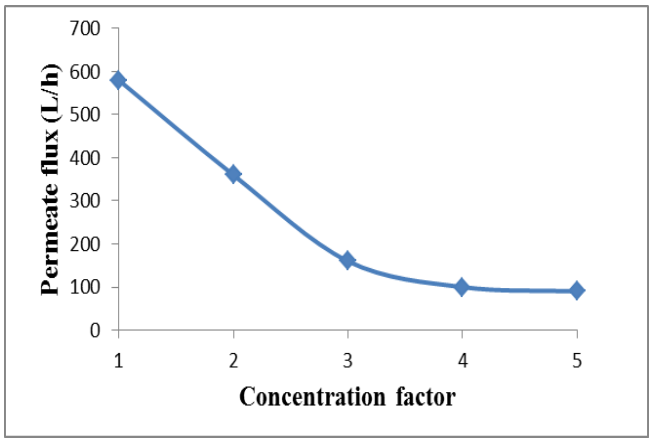


Fig. 4. Whey concentration by UF: influence of concentration factor on the permeate flux (FSK 131 membrane, 50 °C, 1.5 Bar, 600L/h flow rate)

It can be seen that the permeate flux slowly decreases with an increase in the pH. The permeate flux of UF was higher: 200L/h (Fig. 5) at higher pH 6.5 compared to low pH4.5near to the isoelectric point. This decline in the permeate flux at the low pH was well expected due to the gel layer formation on the membrane surface as the feed concentration increased and deposition occurred [23].
The effect of the higher operating temperature resulted in an increase in the permeate flux (Fig. 6) until 50 °C, where the viscosity of the processed whey reaches its minimum value and hereafter temperature increase can cause heat denaturation of the whey proteins [23, 22].

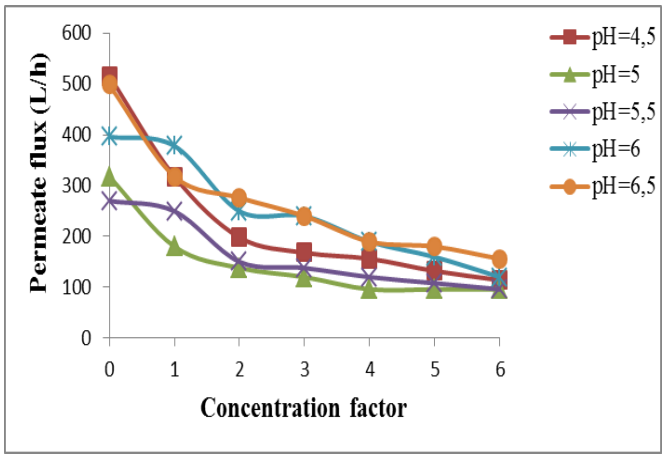


Fig. 5. Whey concentration by UF: influence of the pH and theconcentration factor on the permeate flux (FSK 131 membrane, 50 °C, 1.5 Bar, 600L/h flow rate)

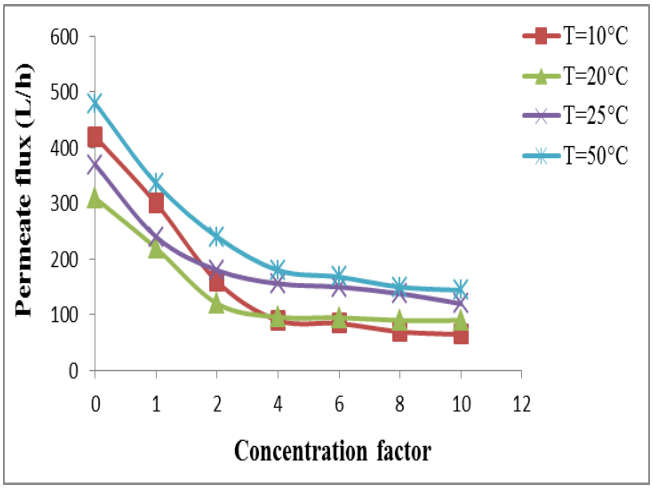


Fig. 6. Whey concentration by UF: influence of the temperature and the concentration factor on the permeate flux (FSK 131 membrane, 50 °C, 1.5 Bar, 600L/h flow rate)

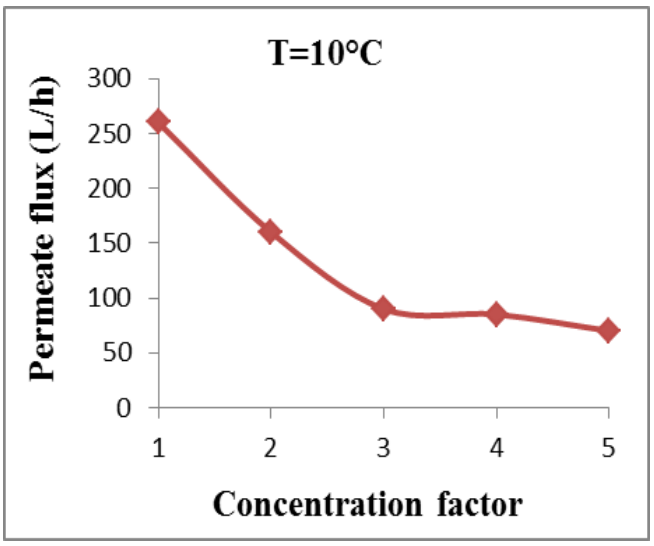
B. Ultrafiltration of whey in multi-stages mode

B.1.The effect of the temperature on acidic whey

In the first step, the concentration of whey was conducted at acidic pH of the influence the temperature and VRF. Figure 7 and figure 8 present respectively the effect of the temperature and VRF on the permeat flux and the proteins content.
The study show at the lower temperature (10 and 25°C) (figure 7)it was not possible to conduct the stages operations, the membrane is quickly fouled. The VRF is reached the maximum of 10C° for 4 and 6 to 25°C. The decreasing of the temperature results in an increase in the viscosity of whey, resulting in a decreasing in the permeate flux. This phenomenon is in agreement with literature data [24].

The higher operating temperature (50°C) (figure 7) resulted in an increase in the permeate flux and the VRF. The flux decrease exponentially for the two stages and tends towards the bearings. It was not possible to go beyond the second stages and beyond the VRF 16 due to the fouling of the membrane.

At the higher temperature it can be seen that the protein content increases with the VRF in the retentate. The protein content of UF was higher: 60 (g/kg) but the leak protein in the permeat remains low whatever the VRF it's about 3g/kg (figure 8).



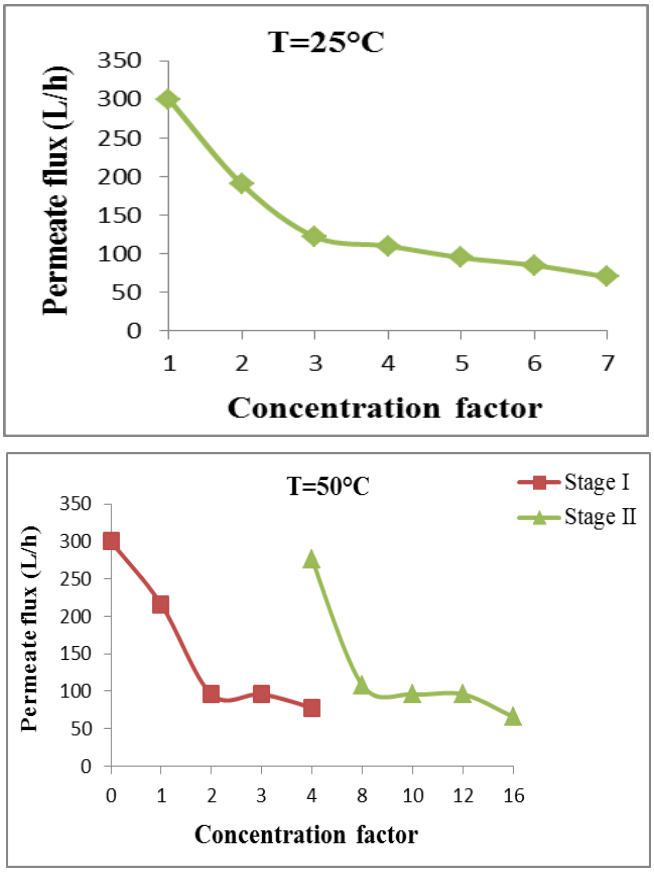


Fig. 7. Whey concentration by UF: influence of temperature and concentration factor on the permeate flux (FSK 131 membrane, 600L/h flow rate, 1.5 Bar, pH=4.5)

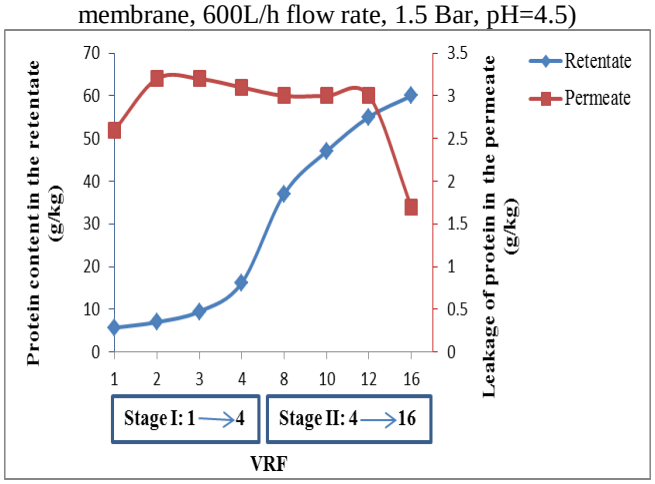


Fig. 8. Whey concentration by UF: influence of concentration factor on the protein content in the retentate and the permeate (FSK 131 membrane, 600L/h flow rate, 1.5 Bar, pH=4.5, 50°C)

B.2.The effect of the temperature on neutral whey

To achieve satisfactory operational performances and higher VRF (higher than 16), the concentration of whey was operated at the same conditions as in the case of the acidic pH of whey but in this case at the neutral pH (6.5).

The concentration at 10 C° was conducted in three stages to achieve maximum VRF 10.Beyond the VRF 10, the membrane fouls almost irreversibly. The number of stages is reduced to two with a maximum of 16 VRF when the permeate flux was decreasing to 100 (L/h) (figure 9).

At the higher temperature (50 °C), the optimizing operation achieves about 4 stages with a maximum of VRF 50. At this temperature, the viscosity of the concentrated whey is minimum, which improves the flow velocity (figure 9). We cannot work beyond this temperature because the membrane does not allow it, and due to the risk of protein denaturation [22].So, the achievement of higher temperature is limited by the properties of the ultrafiltered solution and of the investigated membranes.

The proteins content increased with the operating temperature in the retentate, but in the permeate it decreases (figure10): At the higher temperature (50 °C) the proteins content was maximal about 48 g/kg in the retentate). These results are mainly related to the viscosity of whey which decreases with temperature and which assists permeate flow rate. At higher temperature effect the transfer of water through the membrane increases causing an increase in protein content. The viscosity of the retentate is increased and therefore the leakage of protein decreases.

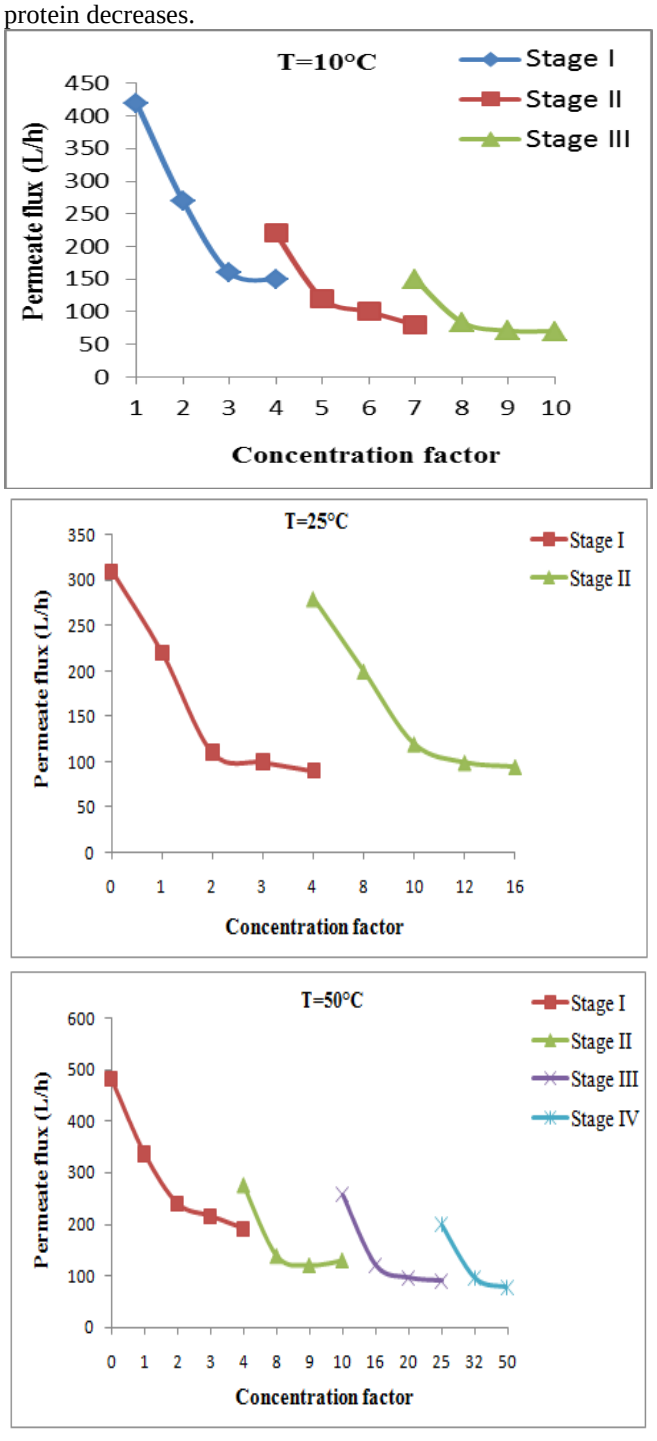


Fig. 9. Whey concentration by UF: influence of temperature and concentration factor on the permeate flux (FSK 131 membrane, 600L/h flow rate, 1.5 Bar, pH=6.5)

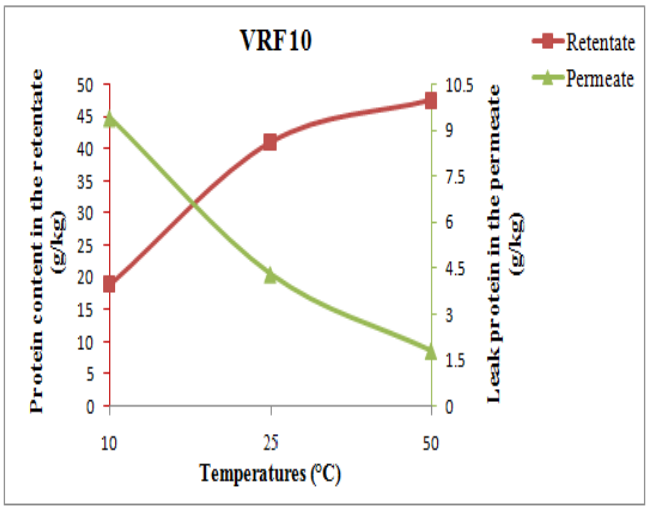


Fig.10. Whey concentration by UF: influence of temperature on the protein content in the retentate and the permeate (FSK 131 membrane, 600L/h flow rate, 1.5 Bar, pH=6.5, VRF 10)

IV. CONCLUSION

It can be concluded, from our experiments, that the concentration of whey protein by ultrafiltration, can be successfully achieved using the investigated membrane (FSK) with a high efficiency.

- The experimental results using UF membrane in multi-stages mode were positive, the protein content reached 98%, while the permeate flux was acceptable, 80 L/ h, using low pressure 1.5 bar.
- For whey proteins the suitable temperature of UF is 50 °C, where the viscosity of the solutes has a low value. Further increase in the temperature is limited, because of decomposition of the proteins and damage of membrane material.

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