v. 4, n. 1, 2024

International Journal of Biological and Natural Sciences

DESIGN, CONSTRUCTION AND CHARACTERIZATION OF A SEMI-PILOT ULTRAFILTRATION EQUIPMENT

Orozco-Alvarez Carlos
Department of Bioengineering
Interdisciplinary Professional Biotechnology Unit
Instituto Politécnico Nacional
Ciudad de México

Palma-Orozco Gisela
Department of Bioengineering
Interdisciplinary Professional Biotechnology Unit
Instituto Politécnico Nacional
Ciudad de México

Camacho-Santos Aranza
Department of Bioengineering
Interdisciplinary Professional Biotechnology Unit
Instituto Politécnico Nacional
Ciudad de México

Osnaya-Domínguez Carlos F
Department of Bioengineering
Interdisciplinary Professional Biotechnology Unit
Instituto Politécnico Nacional
Ciudad de México

García-Salas Sergio
Department of Bioengineering
Interdisciplinary Professional Biotechnology Unit
Instituto Politécnico Nacional
Ciudad de México

Hernández-Sánchez Enrique
Department of Bioengineering
Interdisciplinary Professional Biotechnology Unit
Instituto Politécnico Nacional
Ciudad de México

Abstract: Ultrafiltration equipment was built for a ceramic membrane module with a filtration area of 0.15 m2. Additionally acquiring a 1.0 HP centrifugal pump, a 30 liter tank and a batch of one-inch diameter pipes, valves, instruments and accessories. The equipment was tested with water, obtaining a flux of 120 x10-6 m/s, at 240 kPa and on a 100 kDa membrane, while with an 8 kDa membrane the flux was 50 x10-6 m/s at the same pressure.. When tested with whole milk, the fluxes were 5 x10-6 and 3 x10-6 m/s for the 100 and 8 kDa membranes, respectively, at the pressure of 240 kPa. Whole milk was concentrated five times on 8 and 100 kDa membrane and a total solids content of 50% was reached, equivalent to a protein content of 15 to 17%. It was found that it was better to use the 100 kDa membrane because it reduces the operation time by half.
Keywords: Tangential filtration, flux, transmembrane pressure, membrane module.

INTRODUCTION

Ultrafiltration is a widely useful operation in food areas. [(1)], biotechnology [(2)], environmental and pharmaceutical [(3)], where it is applied in its two basic modalities: concentration [(4)] and diafiltration. In the first modality, the example is the concentration of recombinant proteins with high added value such as human insulin, albumin and human immunoglobulins, microbial and viral vaccines; whole milk concentration [(5)], polysaccharides such as xanthan and arabic gums, enzymes such as glucose oxidase, alpha-amylase, commercial proteases and all types of cellulases; also in concentration of peptides with biological activity, recovery of whey proteins, etc. While diafiltration(6) is applied to eliminate low molecular weight impurities that contaminate all these types of high molecular weight products and that have to meet the required commercial specifications.

On the other hand, the school does not have a team of semi-pilot ultrafiltration (10 to 50 liters) where students, from the different engineering careers taught here, could carry out both practical work in a pilot plant with equipment of these characteristics, as well as apply the basic concepts of unit operation tangential filtration. With this background, this project was developed to design and build a semi-pilot ultrafiltration equipment that would have a much lower cost compared to a commercial equipment with similar characteristics and that, once built, would serve as support for academic, technical and experience training. professional for engineering students.

MATERIALS AND METHODS

Part I. First, all types of ultrafiltration modules were investigated, reviewed and analyzed, to subsequently quote and purchase the selected module that met the stated objectives.

Once this module was acquired, the next step was to determine the flow and working pressure needs for its good performance. Both the maximum work flow and the maximum pressure it supports were calculated.

Then the isometric diagram of the ultrafiltration equipment was prepared to have the exact dimensions and arrangement of the main components, as well as to have the number of accessories, valves and pipe sections to assemble the equipment.

Having determined the above, the next step was to calculate the power of the pump through the calculation of the total pressure drop of the system and the work flow of the membrane module, and then proceed to select, quote and purchase the pump that meet these results; All accessories, valves and pipes were also selected and purchased. Finally, the ultrafiltration equipment was assembled following the isometric diagram in detail. The calculation sequence of the pump power (eq. 1) and the NPSH (eq. 2) was as follows:

$$Pot_{pump} = \frac{(\Delta P_{total})(\rho g) flow_{alim}}{Ef};\ W$$

Equation 1

Where:

a) ΔP_{total} *(total pressure drop; m) =* $P_{discharge}$ *+ (z2 - z1) +* h^{l}.

a.1) $P_{discharge}$ *(pump discharge pressure; m); which was considered the maximum pressure at which the membrane module must be fed and according to the technical sheet it must be 45 psi.*

a.2) *z2 - z1* (difference in heights between the pump discharge and the liquid level in the tank; m).

a.3) h_L (pressure drop due to pipes, fittings, valves, etc.;m)= $f_{Darcy} \frac{1}{g} \frac{L}{d} \frac{v^2}{2}$.

a.3.1) f_{Darcy} (Darcy friction factor)

a.3.2)(acceleration of gravity)=9.807 m/s^2.

a.3.3) L/d (equivalent length)

a.3.4) v (speed; m/s)= $\frac{flow_{alim}}{af}$. $Flow_{alim}$ (feed flow to membrane module; m^3/s); af (pipe flow area; m^2)= $\frac{\pi}{4} d^2$; d (pipe diameter; m).

b) ρ (estimated density of whole and concentrated milk) = 1200 kg/m3.

d) Ef (pump efficiency.)

$$NPSH = P_{int} + P_{hid} - pv - h_L; m$$

Equation 2

Where:

P_{int} (internal pressure in the tank; m); which was considered zero because it was a tank open to the atmosphere.

P_{hid} (hydrostatic pressure; m)=$\rho g H_{liq}$. It will be expressed in meters of liquid column; H_{liq} (liquid height from pump suction to liquid level in tank.)

c) pv (vapor pressure of the liquid; m).

d) hL (pressure drop between tank discharge and pump suction, due to pipe, *fittings and valve; m)=* $\frac{1}{g}\frac{L}{d}\frac{v^2}{2}$*. All terms have already been previously defined.*

Part II. The designed and built equipment was first tested for pump performance by obtaining the discharge head vs. flow.

Then the performance of the membrane was tested using water as a fluid to obtain the filtration flux curve vs. transmembrane pressure. The filtrate flux was determined in triplicate by measuring the time necessary to collect a filtrate volume of 100 mL, and the average value was divided by the filtration area of the membrane to obtain the flux value. While the transmembrane pressure (ptm) was determined with equation 3.

$$ptm = \frac{P_{ent} - P_{sal}}{2} - P_f; \qquad \text{Equation 3}$$

Where:

P_{ent} : inlet pressure to the membrane module

P_{sal} : membrane module outlet pressure

P_f : pressure in the filtrate stream

In the end, the team worked using whole milk as a model fluid and with this, carried out the concentration of the protein by a factor of 3 to 5, and thus obtained the filtrate flux curve vs. % total solids. The latter was determined in duplicate by taking a 10 mL sample from the equipment tank and placing it in an oven at 60 °C to dry and weigh every 24 hours until constant weight.

RESULTS AND DISCUSSION

Firstly, the information provided in the membrane module supplier's catalog was analyzed and from this it was possible to prepare the information shown in Table 1. The module acquired corresponds to the 7-channel ceramic material module. [7], with a hydraulic diameter of each channel of 0.006 m and a channel length of 1.178 m. With these data it was possible to calculate the feed flow to the module at each of the speeds indicated in the table, and thus it was found that at the maximum speed of 6 m/s a result of feed flow of 11.88 x10-4 was obtained. m3 /s (71.3 liters per minute); The filtration area was also calculated for the entire module, being 0.155 m2 (7π*0.006*1.178). It must be noted that the calculated feed flow is the highest value, so the pump power, which is later calculated, would also serve for the 19 and 61 channel modules, in case these were purchased in the future. [8, 9]. On the other hand, the pump will not have pressure control because its cost could be 4 to 5 times higher than a centrifugal pump, so the working pressures will be reached with manual control of the closure of the backpressure valve. ; and the resulting flows will be measured with the rotameter that will be placed at the pump discharge.

Speed (m/s)	Number of channels	1	7	19	61
	Internal diameter (m)	0.006	0.006	0.0035	0.002
	Power flow to module ($x10^{-4}$ m^3/s)				
	2	0.57	3.96	3.66	3.83
	2.5	0.71	4.95	4.57	4.79
	3	0.85	5.94	5.48	5.75
	3.5	0.99	6.93	6.40	6.71
	4	1.13	7.92	7.31	7.67
	4.5	1.27	8.91	8.23	8.62
	5	1.41	9.90	9.14	9.58
	5.5	1.56	10.89	10.05	10.54
	6	1.70	11.88	10.97	11.50

Table 1. Determination of the feed flow to the membrane module.

Subsequently, the isometric diagram was prepared to establish the arrangement of the equipment components (prepared in AutoCad Plant 3D), such as the tank, pump and membrane module, as well as to know the arrangement of the pipes, valves, instruments and all the accessories necessary for the complete construction of the equipment distributed in the 2-inch PTR metal structure; Figure 1 shows the proposed arrangement of the equipment.

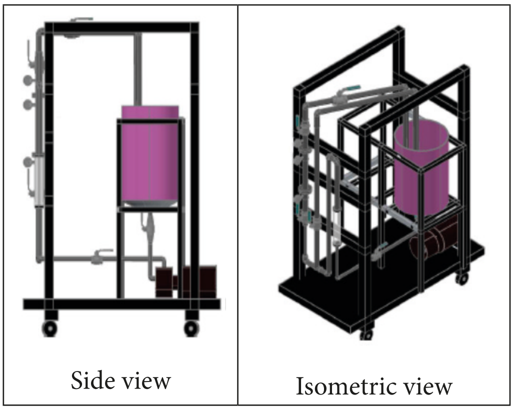

Fig. 1. Isometric diagram of ultrafiltration equipment; own elaboration.

Based on the isometric diagram of the equipment, the total pressure drop was determined, obtaining a value of 39 m of water column equivalent to 460 kPa (67 psi). With this result and the feed flow calculated in Table 1, the power of the centrifugal pump was estimated, giving a value of practically 1.0 HP; This power will be sufficient to provide the flow and pressure necessary for the membrane module to work at speeds, within the channels, of 2 to 6 m/s. Table 2 presents the calculation sequence to obtain the aforementioned results, and also shows the value obtained from the NPSH (net pressure suction head), this being 0.56 m (approx. 2 ft), which is a criterion for pump selection. With this, the centrifugal pump was selected and purchased, and the national supplier provided us with the "power curve" of the pump to verify that it complies with what was calculated in this work (figure 2; masterflex brand centrifugal pump, model 70840-30, Cole-Parmer Catalog).

Also based on the isometric diagram, the rest of the components of the equipment were calculated, quoted and then purchased, the costs of which are presented in Table 3. The total cost to have the equipment assembled was 85 thousand pesos, a value that was 3 5 times lower compared to commercial equipment with similar characteristics.

Component	Cost (M.N.)
Ceramic membrane with 0.155 m2 filtration area, 7 channels, 6 mm diameter	15,000
Stainless steel housing or module for the 1,178 m and 7 cm diameter membrane	25,000
1.0 HP centrifugal pump	20,000
30 L capacity high-density PVC tank	5,000
15 m of 1.5 inch diameter heavy duty PVC pipe, 5 butterfly valves, 3 stainless steel 0 to 60 psi gauges, 12 PVC elbows, 1 0 to 30 GPM rotameter and 2 PVC union nuts.	10,000
2 inch PTR frame	10,000
Total	**85, 000**

Table 3. Cost of ultrafiltration equipment components

Once the equipment was built, the performance of the pump was characterized and the results are shown in Figure 3. The typical behavior of a centrifugal pump is clearly observed, that is, the greater the flow, the lower the head or discharge pressure of the pump. When all valves are fully open the discharge pressure measured on the gauges is practically zero and the measured pump flow is approx. 42 gpm, and a low flow could also be observed in the filtrate pipe. It can also be interpolated from this curve that at a flow rate of 18.8 gpm (1.19 x10-3 m/s, maximum value from Table 1), the pump will provide a discharge pressure of 62 ft, equivalent to 182 kPa.

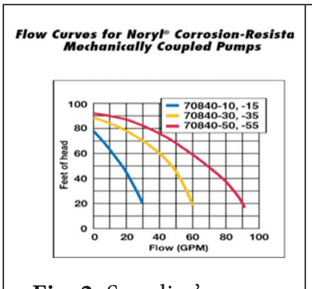

Fig. 2. Supplier's pump duty curve; yellow line

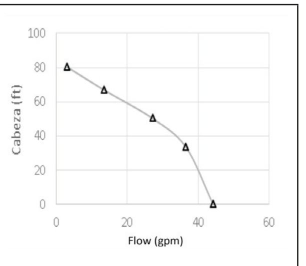

Fig. 3. Acquired pump performance test

Then we proceeded to characterize the

Discharge pressure (45 lb/in^2); $P_{discharge}$	310264	N/m^2
Considering a density of concentrated milk of 1200 kg/m3, and then multiplied by gravity; (ρg)	11,768	N/m^3
Discharge pressure in (m); $P_{discharge}$	26	m
Liquid height difference; (z2-z1)	1.5	m
Pressure drop in pipe, fittings, rotameter and valves; h_L	10.76	m
Total drop in pressure; ΔP_{total}	39	m
Power flow to module; flow $_{alim}$	11.88x10^{-4}	m^3/s
Pump efficiency; Ef	0.73	
Pump power; P pump	740	W
Internal pressure in the tank; P_{int}	0	m
Height between suction and liquid level;P_{hid}	1	m
Vapor pressure of water at 25 °C; pv	0.32	m
Suction pressure drop; $h_{L\ suction}$	0.12	m
NPSH (Net Pressure Suction Head)	0.56	m

Table 2: Determination of power and NPSH of the pump

ultrafiltration membranes of 100, 8 and 0.45 kDa molecular cut-off, through the calculation of the filtrate flux, using water as the working fluid and the results are presented in Figure 4. It is clearly observed that the Water flux in the 100 kDa membrane was 3 to 5 times higher than that of 8 kDa at any pressure that was compared; This was expected since the 100 kDa molecular cut membrane has a larger pore size than the 8 kDa one. In turn, the fluxes obtained in the 8 kDa membrane were 2 to 2.5 times higher compared to the 0.45 kDa molecular cut membrane, since the latter membrane has the smallest pore size of the three membranes that were worked on.

Then we worked only with the 100 and 8 kDa membranes for the tests with whole milk and the results obtained are represented in figure 5. The fluxes are very similar in both membranes, but at the same time they are 10 to 20 times lower in comparison when water was used as the working fluid(10, 11). It is also observed that the flux of the 100 kDa membrane (5 x10-6 m/s) is twice that of the 8 kDa (2.5 x10-6 m/s) when working with whole milk as fluid. It was expected that the flux of the 100 kDa membrane would be higher than that of 8 kDa (and not only double as experimentally obtained), but probably the mixture of proteins-fats-cells, which roughly we can say that they make up the whole milk, could have formed a conglomerate of a larger size than casein (which has a molecular weight of 20 kDa), thus converting this conglomerate into an additional resistance on the surface of the membrane of 100 kDa, then causing this so that the flux was lower than expected, and in these experimental results, similar to the flux values obtained with the 8 kDa molecular cut membrane [13, 14, 15].

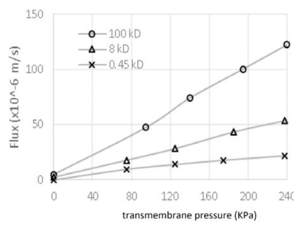

Fig. 4. Flux behavior with water

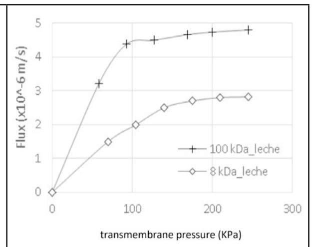

Fig. 5. Flux behavior with whole milk

After working with whole milk, we now proceeded to concentrate it using a factor of 5 times, and using the 100 and 8 kDa molecular cut membranes and the results obtained are indicated in figures 6 and 7.

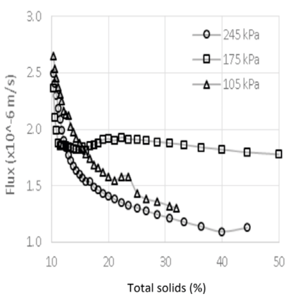

Fig. 6. Ultrafiltration of whole milk at 8 kDa.

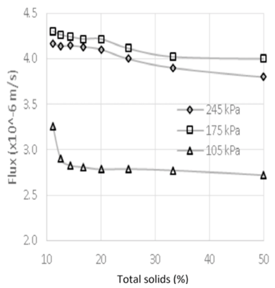

Fig. 7. Ultrafiltration of whole milk at 100 kDa.

As it can be seen, the filtrate flux decreases with the increase in the concentration of total solids, in both membranes, due to the "growth" of the polarizing gel layer that reduces the flux(16, 17). The transmembrane pressure with which the highest flux value was obtained was 175 kPa in both membranes. In theory it would be expected that with a higher transmembrane pressure the flux value would also increase, however and based on the results this did not occur, which could be due to the formation of the polarizing gel layer on the surface of the membrane. which can be said to be "pressed" to transmembrane pressures greater than 175 kPa, and therefore caused a greater reduction in the filtrate flux values(18). The polarizing gel theory is the most used to model the behavior of the flux as a function of the concentration of the retained solute, and it indicates that the solute, larger than the pore diameter of the membrane, cannot filter through The membrane is retained on the surface of the membrane, forming a gel layer that in turn conglomerates as the concentration of the suspension or working solution is being carried out.

Finally, in Figure 8, the results of the cleaning of the membrane are presented once it was used. The results indicate adequate cleaning since the filtrate flux values presented a similarity of up to 90% with respect to the respective values of the flux when the membrane is new and tested with water, that is, there was a complete recovery with respect to the original value. Cleaning is a very important aspect to maintain the high performance of the membrane and it can remain that way for one or two years of work.

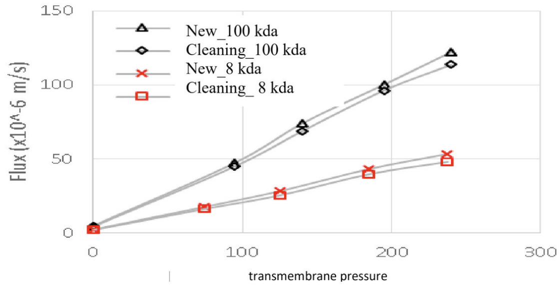

Fig. 8. Recovery of the membrane filtration flux after cleaning.

CONCLUSIONS

Ultrafiltration equipment was built based on the characteristics of the membrane module acquired, additionally purchasing a 1.0 HP centrifugal pump, a 30-liter high-density PVC tank and a batch of one-inch pipes, valves, instruments and accessories. nominal diameter. The total cost of the equipment was 85 thousand pesos, a value that is 3 to 5 times lower than a commercial equipment of the same capacity. The equipment was tested with water and then with whole milk. The latter was concentrated five times on an 8 and 100 kDa membrane to reach a total solids content of 50%, equivalent to a protein content of 15 to 17%. It turned out that it is better to use the 100 kDa membrane because it halves the time to carry out the same level of concentration.

FINANCING AND ACKNOWLEDGMENTS

Project: SIP 20200109. INSTITUTO POLITÉCNICO NACIONAL.

REFERENCES

1. Chacón, V. (2006). Tecnologías de Membrana en la Industria Láctea, Revisión Bibliográfica. *Agronomía Mesoamericana*, 243-248.

2. Ferreira, M. (2012). Improvement in the Purification Process of the Capsular Polysaccharide from Haemophilus influenzae Type b by Using Tangential Ultrafiltration and Diafiltration. *Appl Biochem Biotechnol,* 2068-2075

3.Bo, S. (2013). Large-scale purification of Pharmaceutical-grade plasmid DNA using tangential flow filtration and multi-step chromatography. *Journal of Bioscience and Bioengineering,* 1-6.

4.Glover, F. A. (1984). Principles of ultrafiltration and the concentration and fractoniation of cow´s milk. England: Nestle, Nutrition.

5.Yan, S., Hill, C., & Amundson, C. (1979). Ultrafiltration of whole milk, Vol. 62 Article 1. Journald of Dairy Science, 23-40.

6.Tessier, L. (2005). Separation and purification of benzylpenicilin produced by fermentation using coupled ultrafiltration and nanofiltration technologies. *Journal of Biotechnologu*, 79-89.

7.Tolosa, S., J., B., & Cadenas, A. (2006). Producción de Yogur Utilizando Membranas Cerámicas para Incrementar el Porcentaje de Sólidos de la Leche. Revista iencia e Ingeniería No 5 Artículo 2, 4-9.

8.Cheryan, M. (1986). Ultrafiltration Handbook. Technomic Publishing Co., Lancaster Pennsylvania, U.S.A. p. 73-125.

9.Yeh, H.M., Wu, H.P. and Dong, J.F. (2003). Effects of design and operating parameters on the declination of permeate flux for membrane ultrafiltration along hollow-fiber modules. *J. Membrane Sci*, 213, 33-44.

10.Dragoljub, D., Marijana, C., & Miloslav, k. (1989). Effects f Protein Concentration in Ultrafiltration Milk Retentates and the Type of Protease Used for Coagulation on the Microstructure of Resulting Gels. *Food Structure: Vol. 8: No. 1, Article 8.*, 53-66.

11.Lewis, M., Grandison, A., & Ramachandra, R. (1995). Effect of pH on flux during the ultrafiltration of sweet whey and buttermilk. *Journald of Dairy Research, Vol. 62 Article 3*, 441-449.

12.Abhiram Arunkumar, Mark R. Etzel. (2014). Fractionationof α-lactalbumin and β-lactoglobulin from bovine milk serum using staged, positively charged, tangential flow ultrafiltration membranes. *Journal of Membrane Science, 454, 488-495*

13. Rinaldoni, A., Campderrós, M., & Pérez, M. (2010). Yogures Deslactosados Elaborados con Concentrados de Leche Bovinas y de Soja obtenidos por ultrafiltración. *Ingeniería y Competitividad*, 19-30.

14. Agnel Muni, Gisela Páez, José Faría, José Ferrer, Eduardo Ramones. (2005). Eficiencia de un Sistema de ultrafiltración/nanofiltración tangencial en serie para el fraccionamiento y concentración de lactosuero. Revista científica, FCV-LUZ/ Vol. XV, No. 4, 361-367.

15.José Faría, Aleida García, Aiza García. (2005). Eficiencia en la concentración de la proteína de lactosuero con una planta móvil de ultrafiltración y nanofiltración. Revista científica, FCV-LUZ/ Vol. XIII, No. 5, 347-351.

16. Brazuelo, A., & et al. (1995). Protein-Enriched yogur by ultrafiltration of skim-milk. Journald of the Science of food and Agriculture, Article 69, 283-290.

17.Tamime, A., & et al. (1991). Microstructure and Firmness of Labneh (High Solids Yoghurt) Made From Cow's, Goat's and Sheep's Milks by a Traditional Method or by Ultrafiltration. Food Structure: Vol. 10: No. 1, Article 4, 37-44.

18.Chaufer, B. (1992). Tetracycline removal or concentration with an inorganic ultrafiltration membrane modified by a quaternized polyvinylimidazole coating. Trans Tech Publications, 249-254.