World Applied Sciences Journal 7 (5): 632-636, 2009
ISSN 1818-4952

Separation of Sugar from Molasses by Ultrafiltration and Nanofiltration

[1]S.M. Mousavi and [2,3]M. Tousi Moghadam

[1]Department of Chemical Engineering, Faculty of Engineering,
Ferdowsi University of Mashhad, Iran, P.O. Box: 91775-1111
[2]Membrane Processes and Membrane Research Center, Department of Chemical Engineering,
Faculty of Engineering, Ferdowsi University of Mashhad, Mashhad, Iran
[3]Department of Chemical Engineering, Quchan Azad University, Quchan, Iran

Abstract: Molasses, the main material of this project was diluted and processed, according to the type of the used ultra and nano membranes (UF10kDa, UF5kDa, N30F). Permeate flux was determined at different times for membranes and steady time was registered. By changing transmembrane pressure, permeate flux, which is dependent on the pressure, was registered. Final permeate flux was registered in each transmembrane pressure and for each membrane and the results were compared finally. The comparison was completed by the rejection calculation for three membranes. The results show that both the ultra and nano membranes have clarified the permeate solution but the rejection for N30F membrane was the greatest, around 0.8 and after that UF5kDa membrane had higher rejection than UF10kDa membrane. For N30F membrane, Brix, viscosity and sucrose concentration in permeate flow decreased. So by evaluating used membranes on separation of sugar from molasses, it was resulted that N30F membrane has been more appropriate.

Key words: Molasses · Ultrafiltration · Nanofiltration · Membrane · Sugar

INTRODUCTION

The membrane processes are modern technologies that have been involved with major revolutions in industry branches such as petroleum, gas, pharmacy, biotechnology, water, wastewater, food processing and medicine. These processes in comparison with conventional methods are led to quality improvement, increase of product yield, novel products and decrease of energy consumption. Sugar industry is one of the most energy-intensive processes in the food chemical industries; therefore, it has been extensively investigated by membrane scientists in the last decade.

The application of new methods in sugar beet factories have been investigated in four classes:

- Purification of raw syrup
- Concentration of diluted syrup by reverse osmosis
- Separation and extraction of color compounds
- Achieving product with high protein percent

Purification and desugarization can be accomplished by nanofiltration. It is clear that ultrafiltration and nanofiltration can be more effective and useful for separating non-sucrose compounds from other compounds that can crystallize directly. Using these methods leads to decreasing energy consumption and environmental pollution. Different investigators have carried out some experiments on sugar beet. These investigations are described as follows. According to the modeling of ultrafiltration of non-sucrose compounds in sugar beet processing, it is stated that concerning sugar syrup purification might be that permeate flux depends on the linear change of flow rate, temperature and transmembrane pressure on the quadratic change of the concentration factor [1]. By improving thin juice quality through ultrafiltration, it is well established that the obtained permeate by ultrafiltration possesses better clarity and purity in comparison with conventional method, it was also proved that better separation can be performed at 30°C [2].

In separation of fructose from a mixture of sugars using supported liquid membranes, Hollow Fiber Supported Liquid Membrane (HFSLM) was compared to Flat Sheet Supported Liquid Membrane (FSSLM) and it is indicated that HFSLM has worked better than FSSLM [3].

Corresponding Author: S.M. Mousavi, Department of Chemical Engineering, Faculty of Engineering,
Ferdowsi University of Mashhad, Iran, Postal code: 91781 35839 Tel:+98 511 8816840

Significant part of non-sucrose compounds of the green syrup has been separated with polymer membrane. An increase of permeate quality quotient (ratio between sucrose content and dry matter) about 1-3% feed purity quotient has been noticed [4].

Separation of colored matter from green syrup was examined by ultra- and nanofiltration of a solution with 39.2% d.m. content. In the process of ultrafiltration (UF), two polymer membranes were used: membrane I with an MWCO (molecular weight cut-off) value of 15–20 kDa and membrane II with MWCO of 6–8 kDa. The highest separation by UF on the examined membranes was achieved for the transmembrane pressure in the range of 2.5–4.0 bar at flow rates ranging from 220 to 360 l/h [5].

The juice treated with micro- and ultrafiltration achieved such quality that direct crystallization was possible. Proper membrane for NF of raw juice should be sought on the dense side of the NF membrane spectrum where some tested membranes exhibited higher retention for sucrose than for non-sugars [6].

There are no researches about separation of sucrose from molasses by membrane processes so far. Thus the main purpose of this paper is to investigate this separation by ultra- and nanofiltration. Permeate flux, rejection, Brix, density and adsorption were determined in each separation test.

MATERIALS AND METHODS

Necessary Materials: Molasses i.e. the final product in the last stage of sugar separation, which its sugar can not be crystallized, is the main material. It was used for the separation of its sugar by ultrafiltration and nanofiltration. The main characteristics of molasses, such as Brix and density, are 75 and 1.6 kg/m^3 respectively. Three types of membranes with different MWCO made in Germany with NADIR mark were used for the experiments. The membranes were composed of polyethersulfone (PES). The range of pH and maximum temperature for these membranes are 0-14 and 95°C respectively. Distilled water for dilution and washing, Fehling solutions A and B for Lane and Eynon test, ferrocyanure with zinc acetate as clarifier, methylene blue for determining the final point of process, concentrated HCl for sucrose hydrolyze and NaOH 0.5 N for neutralizing acid were used too. Majority of the materials are obtained from Merck Co.

Membrane System: The flow diagram of applied pilot is shown in Fig. 1. The installed pump on the pilot is a two stages pump with 2800 rpm and its type is centrifugal. It has capacity 2m^3/h, head 165meter and power 2.2 kW.

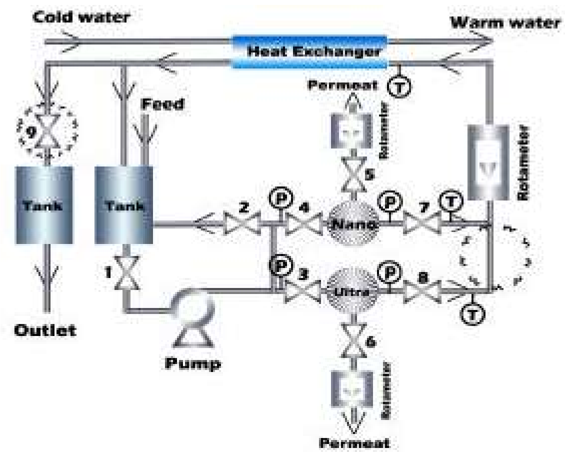

Fig. 1: UF and NF pilot, (1) outlet valve of feed tank, (2) bypass valve, (3) inlet valve to ultrafiltration module, (4) inlet valve to nanofiltration module, (5) permeate valve of nanofiltration module, (6) permeate valve of ultrafiltration module, (7) outlet valve of nanofiltration module, (8) outlet valve of ultrafiltration module, (9) valve for gathering retentate

Experimental: Because the Brix of used molasses is 75, thus it was diluted with distilled water, so that its concentration was reached 140,000 ppm for doing ultrafiltration operation. For fulfilling experiment, first the tank should be loaded by feed. Then, it is necessary to open the aerobatic valve for discharging the air from pump. After that, according to the selective membrane, the valves that are using should be opened and other valves should be closed. For carrying out the tests with each of ultra membranes in several transmembrane pressures, first the pressure was regulated on 4 bar and after 5 minutes sampling was started. It will be repeated after 5 minutes and totally sampling was carried out 9 times till filtration reached the steady state. This test was carried out for transmembrane pressures 5 and 6 bar too. At the end of the test with ultra membranes, samples in each transmembrane pressure were transferred to laboratory for analyzing. Then plant was discharged and loaded by new feed with concentration 70,000 ppm for doing experiments with nano membrane. The experiments in transmembrane pressures 10, 11, 12 and 13 bar were carried out and 9 samples of permeate were collected in period 45 minutes in each pressure. It is noticed that all tests were carried out at 30°C.

Analyze Method: Lane and Eynon method is the best way for measuring the existing sugar in diluted molasses samples such as feed, permeate and retentate.

This method is one of the effective methods for analysing food materials and is based on reclaimable property of sugars [7]. Furthermore picknometric method was used for measuring density of samples. The Brix (percent of solute solid materials in water) of feed, permeate and retentate were measured by refractometer. Absorbance of samples for 420 and 720 nanometer were obtained by spectrophotometer.

Rejection can be obtained with measuring the sugar concentration of solutions. With having the volume fluxes and rejections, it is possible to present the following results.

RESULTS AND DISCUSSION

Permeate Flux: The results of flux changes in terms of time for UF5kDa membrane is shown in Fig. 2. In this figure, flux is decreasing because of membrane fouling and concentration polarization. Fig. 2 shows that the slope of the curves approximately becomes zero after 25 min. As a matter of fact, at this time the curves approach steady state.

It is noticeable that if transmembrane pressure was 4-ε (ε < 1), flux will be stopped completely after some minutes. Because of this reason, it can be assumed that the pressure of 4 bar is a turning point for good performance of this membrane in terms of the different transmembrane pressures.

With regard to Fig. 3, the results of UF10kDa membrane show that in the same condition the flux has the maximum value at the beginning of the experiment. At the same transmembrane pressure, the flux decreases till it becomes steady. Steady state time is gotten approximately at 30-45 min.

With respect to Fig. 4, which is related to N30F membrane, it can be seen that at the pressure 10 bar, flux reaches steady state at 30 min, but for the other pressures the steady time is approximately 35 min. At the beginning of the experiment, similar to the ultra membranes, the flux has maximum value and after some minutes, the flux decreases while reaches constant value. The other similarity of these two types of membrane i.e. nano and ultra membranes is that the flux increases by increasing transmembrane pressure.

A comparison of steady flux for UF10kDa, UF5kDa and N30F membranes in different transmembrane pressures is observed in Fig. 5. Considering this figure, it is obviously clear that an increase in the transmembrane pressure yields an increase in permeate flux.

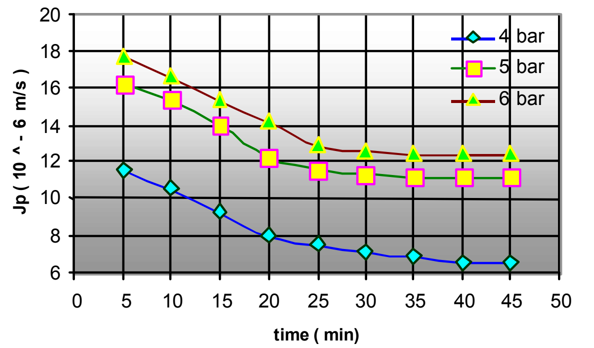

Fig. 2: Dependence of permeate flux on time in different transmembrane pressures for UF5kDa membrane

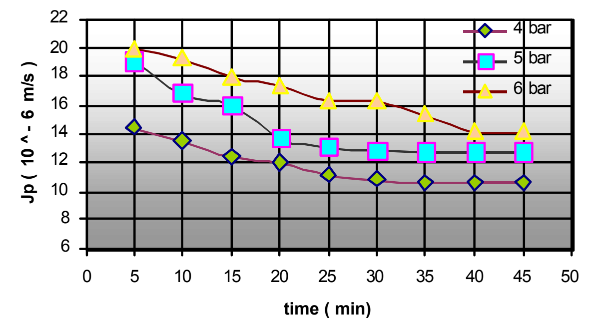

Fig. 3: Dependence of permeate flux on time in different transmembrane pressures for UF10kDa membrane

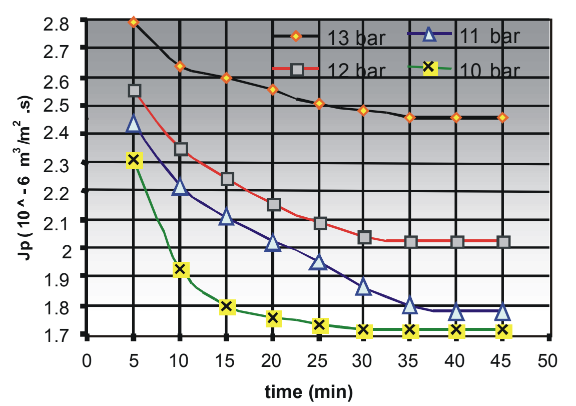

Fig. 4: Dependence of permeate flux on time in different transmembrane pressures for N30F membrane

By focusing on the curves of ultrafiltration membranes in Fig. 5, it is observed that the slope of curves between 4 and 5 bar is greater than the slope of same curves between 5 and 6 bar. By this comparison it can be concluded that increasing the pressure, although results in the increase of permeate flux, but the fouling of membrane and concentration polarization, which have a direct relation with transmembrane pressure, cause the decrease of permeate flux. The curve of UF5kDa

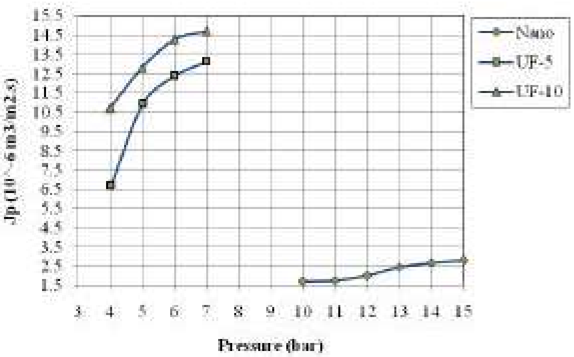

Fig. 5: Dependence of permeate final flux on transmembrane pressure for UF10kDa, UF5kDa and N30F membranes

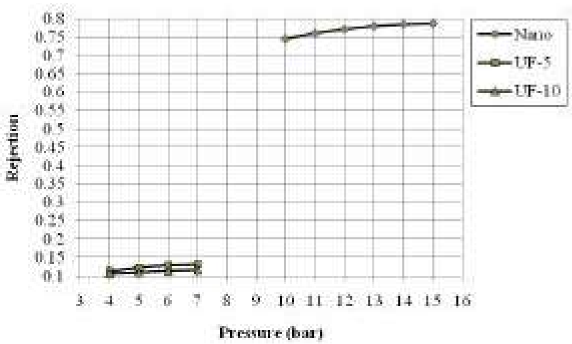

Fig. 6: Dependence of rejection on transmembrane pressure for UF10kDa, UF5kDa and N30F membranes

membrane in Fig. 5 shows that by increasing the pressure from 4 to 5 bar, the permeate flux value increases to 4.51 units, but if transmembrane pressure is increased from 5 to 6 bar, the permeate flux increases to 0.89 units only. This value is around 20% of first change of permeate flux. This indicates that by increasing transmembrane pressure, one point is gotten which after that point, there isn't significant increase in permeate flux.

According to the curve of N30F membrane in Fig. 5, it can be concluded that the slope of curve increases with the increase of the pressure.

It is necessary to mention that at first the feed concentration value was 140,000 ppm when the experiments were carried out by ultra membranes. But this concentration of feed wasn't appropriate for N30F membrane, because after few times permeate flux got zero value; so by diluting, the concentration of solution was decreased to 70,000 ppm and accomplishing the experiments became possible by this membrane.

Rejection Value: According to Fig. 6, the increase of transmembrane pressure results in the rejection increase. Because by increasing transmembrane pressure, permeate flux increases and the solvent and solute pass across the membrane more; but the increase of solute flux is lower than solvent flux. Thus rejection increases because permeate concentration is decreased.

As it is expectable, if UF5kDa membrane is replaced by UF10kDa membrane, it causes the pore size of membranes becomes smaller so sugar molecules are decreased in permeate, then permeate concentration becomes small and rejection will be increased. Comparing the rejection for two membranes in Fig. 6, it can be seen that UF5kDa membrane has higher rejection than UF10kDa membrane. So UF5kDa membrane is more appropriate but rejection value which is achieved isn't close to ideal value 1 and it isn't good for this separation. But the results that will be presented subsequently indicate that both membranes are appropriate in decolorization of feed solution.

As it can be seen from the curve of N30F membrane in Fig. 6, the rejection increases with increasing the transmembrane pressure and its value reaches around 0.8 for N30F membrane. This value is pretty well. It also indicates that N30F membrane is more appropriate one than ultra membranes for molasses filtration.

Permeate Characteristics: Tables 1 and 2 exhibit the density and Brix for the feed and permeate respectively. As it can be seen from Table 1, the permeate of nano membrane has lower density than it of two ultra membranes. This means that N30F membrane could prevent from passing large molecules of feed.

Table 1: Solution density for three different membranes (g/cm^3)

Solution	UF5kDa	UF10kDa	N30F
Feed	1.073	1.073	1.073
Permeate	1.061	1.065	1.043

Table 2: Solution Brix for three different membranes

Solution	UF5kDa	UF10kDa	N30F
Feed	16.7	7.16	16.7
Permeate	14.3	14.9	6.2

Table 3: Comparison of permeate characteristics for ultrafiltration and nanofiltration membranes

Property	UF10kDa	UF5kDa	N30F
Color	Dark yellowish brown	Light brown	Yellow
Purity	89.82	90.54	91.6
Adsorption(420n)	1.03	0.9	0.81
Adsorption(720n)	0.0091	0.0085	0.002

The results of Table 2 indicate that the Brix of permeate was close to the Brix of water for N30F membrane. This is expectable because sugar materials and suspended particles of feed can not pass through the N30F membrane.

Color, average purity and permeate adsorption for ultra and nano membranes are presented in Table 3. The results indicate that the permeate of nano membrane has higher average purity than it of ultra membranes and this permeate has more clarified color than it of other membranes. With comparison of ultra membranes, it is proved that UF5kDa membrane has more clarified permeate than UF10kDa membrane.

CONCLUSION

After fulfilling the experiments by ultra and nano membranes, it can be resulted that the pore size of two ultra membranes UF5kDa and UF10kDa isn't appropriate for desugarization of molasses because the rejection value which is achieved has significant deviation from 1. It means these two membranes can not prevent from passing sucrose molecules but decolorization of them is the important item that can not be ignored. On the other hand because of the lower hydraulic resistance of ultra membranes, the permeate flux of these membranes is greater than it of nano membrane. In N30F membrane, sucrose molecules have been filtered and the amount of sugar would be very few in permeate and the rejection value obtained around 0.8. In addition, Brix, viscosity and sucrose concentration were decreased in permeate. The permeate color of the nano membrane was clarifier and lighter than the permeate color of the ultra membranes and feed color. These mean that the nano membrane was more successful than the ultra membranes in repelling and decolorization.

REFERENCES

1. Djuric, M., J. Gyura, Z. Zavargo, Z. Seres and M. Tekic, 2003. Modeling of Ultrafiltration of Non-sucrose Compounds in Sugar Beet Processing. Journal of Food Engineering, 65: 73-82.
2. Shahidi, M. and M. Razavi, 2006. Improving Thin Sugar Beet Juice Quality through Ultrafiltration. Desalination, 200: 518-519.
3. Luccio, M.Di., B.D. Smith, T. Kida, C.P. Borges and T.L.M. Alves, 2000. Separation of Fructose from Mixture of Sugars Using Supported Liquid Membranes. Journal of Membrane Sci., 174: 217-224.
4. Gyura, J., Z. Seres, G. Vatai and E. Bekassy Monlar, 2002. Separation of Non-sucrose Compounds from the Syrup of Sugar Beet Processing by Ultra-and Nanofiltration Using Polymer Membranes. Desalination, 148: 49-56.
5. Gyura, J., Z. Seres and M. Eszterle, 2004. Influence of Operating on Separation of Green Syrup Colored Matter from Sugar Beet by Ultra-and Nanofiltration. Journal of Food Engineering, 66: 89-96.
6. Hinkova, A., Z. Bubnik, P. Kadlec and J. Pridal, 2002. Potentials of Separation Membranes in Sugar Industry. Separation and Purification Technol., 26(1): 101-110.
7. Parvaneh, V., 2002. Quality Control and Chemical Experiments of Food Materials. Tehran University Publications.