MADING THE CONCENTRATION OF GRAPE JUICE USING THE OSMOTIC DISTILLATION

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Abstract

The paper presents the development of a new, novel membrane process for the concentration of aqueous solutions, in particular grape juices, at high temperature. This interest has been due to the potential for the production of concentrates without the destruction of thermally-sensitive components and the substantial losses of volatile flavour/fragrance compounds normally associated with thermal evaporation. This has increased the interest in membrane technology throughout the world.

Keywords: Osmotic distillation (OD); reverse osmosis (RO); membrane distillation (MD); ultra filtration (UF); hydrophobic membranes; microporous membranes; juice permeate; juice retentate; concentration polarization; flux

INTRODUCTION

OD is a novel membrane process used to concentrate aqueous solutions such as fruit and vegetable juices by transport of water vapor across a hydrophobic membrane. The driving force is a vapor pressure gradient across the membrane, produced by the use of concentrated solution of an osmotic agent (stripper solution) on the downstream side. The osmotic agent is usually an inorganic salt. The advantage of this process is that evaporation occurs at ambient temperatures or below, eliminating the effect of heat degradation and reducing the loss of volatile organic flavour/fragrance components. [6] In the case of grape juice, higher concentrations than are achievable by conventional methods can be obtained due to the elimination of "burn on" that is, severe degradation of the viscous juice adhering to the evaporator heat transfer surfaces. [2]

The stripper solution, also referred to as brine, is the solution responsible for the stripping of water from the feed. The solute in the stripper needs to have certain qualities for it to be of use commercially. The main requirements are that it is non-wetting of the membrane, non-volatile and is capable of reducing the vapor pressure below that of the feed. Other factors to consider in choosing a stripper are: solubility; thermal stability; toxicity; corrosivity; and cost. The feed and brine are pumped over opposite faces of the membrane in counter-current flow. The driving force is the vapour pressure gradient across the membrane due to the difference in the water activity of the two liquids. The process is operated at ambient temperature. The vapor pressure, solute concentration and temperature profiles are illustrated in Figure 1.

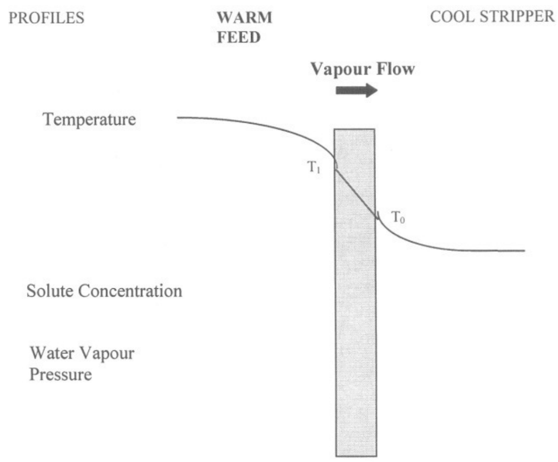

Figure 1. Vapor pressure and concentration profiles of OD

The feed and stripper are recirculated over the membrane back into their respective reservoirs. During this process, the feed is partially concentrated and the stripper is diluted. In this case, the volume of the stripper is not important so long as the dilution is insignificant or the salt concentration is maintained at a constant level by some other means. The volume of the stripper is ideally sufficiently large for both the concentration and temperature to be more easily controlled. Industrial OD plants typically reconcéntrate the brine by pumping to a small evaporator in a closed loop with the brine tank and then cool the returning brine sufficiently to maintain the temperature of the brine supply at an appropriate level. [1]

OSMOTIC DISTILLATION METHOD

Osmotic Distillation (OD), also known as Isothermal Membrane Distillation, was derived from MD and is based on similar principles. The main difference is that OD operates at ambient temperature and the water vapor pressure difference across the membrane is provided by the use of concentrated brine on the downstream side of the membrane. Although MD is operated at temperatures below that of evaporative techniques and has many other advantages over conventional methods, the elevated temperatures can still pose a problem with feeds such as fruit juices and pharmaceutical products that contain thermally labile components. OD offers a viable alternative to the concentration of such sensitive products.

DESCRIPTION OF MASS TRANSFER IN OD

Mass transfer in OD is a three step process as seen in Figure 2**.**

- Evaporation of water at the feed-membrane interface
- Diffusion of water vapor through the pores of the microporous, hydrophobic membrane
- Absorption of water vapor into the stripper solution

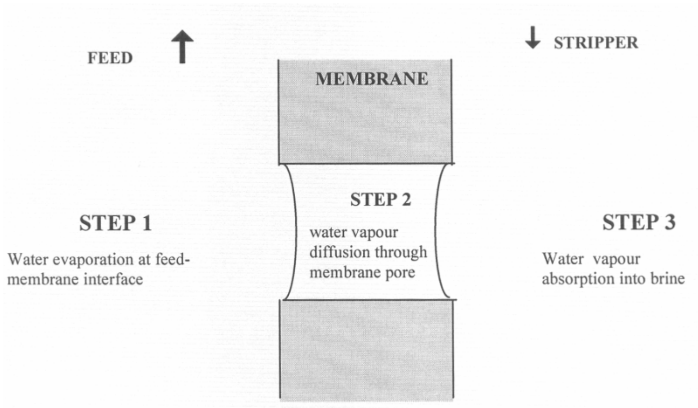

Figure 2. Principles of the osmotic distillation (OD) process

There is a resistance associated with each step in the process. These are the feed side, membrane and stripper side mass transfer resistances.
As in the case of electrical resistors in series, the overall resistance to mass transfer in OD is given by

$$R = Rf + R_m + R_s \quad (1.1)$$

Where R, Rf, R_m, and R_s are the overall, feed side, membrane and stripper side resistances respectively. Since the mass transfer coefficient is analogous to
conductance, which is the reciprocal of resistance, the overall mass transfer coefficient is related to the individual mass transfer coefficients as follows.

$$\frac{1}{k} = \frac{1}{k_f} + \frac{1}{k_m} + \frac{1}{k_s} \quad (1.2)$$

where K = overall mass transfer coefficient kf, k_m, k_s = mass transfer coefficients of feed side, membrane and stripper side respectively**.**

This project was designed to study each of these three steps, with the aim of minimizing the overall resistance and thereby increasing the overall mass transfer coefficient.

The overall mass transfer coefficient is the flux per unit driving force.

$$K = \frac{J}{\Delta P} \qquad (1.3)$$

where J is the flux of water vapor across the membrane and AP is the difference in vapour pressure between the bulk feed and stripper solutions, that is, the driving force that can be written as (Pf- P_s).

$$J = \frac{V_2 - V_1}{(t_2 - t_1)A_m} \qquad (1.4)$$

where V_2 is the volume of water lost from the reservoir at time t_2, V_1 is the volume loss at time t_1 and A_m is the active surface area of the membrane.

REZULTS AND DISCUTIONS

MASS TRANSFER IN LIQUIDS

Solutes diffuse through stagnant liquids according to normal Fickian diffusion, otherwise known as molecular diffusion. In the absence of bulk motion, solutes move through the liquid in order to produce a uniform environment. This is also true for liquids under laminar flow perpendicular to the direction of net solute transport. During OD, the flow of liquid across the face of the membrane may be laminar or turbulent, depending on viscosity, density, linear velocity, and the size of the feed channel. Also, in some circumstances it is necessary to support the membrane by a spacer, leading to an increase in turbulent flow. As in other membrane processes where turbulent flow occurs, there is always a laminar sub-layer (boundary layer) adjacent to the membrane wall[3]

CONCENTRATION POLARISATION BOUNDARY LAYER

During OD, an aqueous feed solution is contacted with the membrane surface and a portion of the water is removed by evaporation into the membrane pores. This results in a higher local concentration of the solute at the membrane surface as compared with the bulk solution. The solute concentration profile is shown schematically in Figure 3

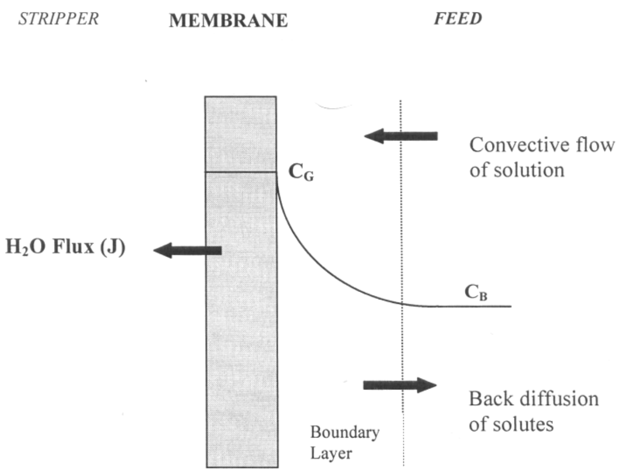

Figure 3. Schematic of concentration polarization during OD

EFFECT OF FEED VELOCITY ON MASS TRANSFER

The initial set of experiments entailed the study of the variation of K, the overall mass transfer coefficient, with flow rate..Also shown are the K values for pure water feed at each feed velocity. The latter data were collected to ensure that the changes in K with feed velocity, and hence with pumping pressure, could not be attributed to leakage through pinholes or wetted-out areas.

The results show that feed side resistance to mass transfer plays a significant part in the transfer of H_2O in the Osmotic cell, particularly at low velocities. This result was critical in the development of OD since it showed that industrial modules used for the production of grape juice concentrate required high feed flow rates and/or turbulence enhancing designs. It is noteworthy that the juice K value approached the maximum attainable (that corresponding to pure water feed) at the highest flowrate used. The difference is of the order of 2 % and this can be attributed to the effects of the unavoidable phenomenon of concentration polarization.

Of interest was the leveling off of the juice curve in the 6-8 $x10^2$ m s^{-1} region followed by a steady increase as the velocity increased to 12 x10 m s^{-1} at a juice velocity of ca. 9 cm s^{-1}. This was often observed using the Osmotic unit and is attributed to a critical change in fluid dynamics. The presence of spacer material on both sides of the membrane precluded the ready calculation of the Reynolds number and hence the point at which the transition between laminar and turbulent flow occurred.

The effects of concentration polarization in the concentration of raw grape juice using hollow fibre module can be seen in figures 4.a and 4.b. As expected, over a period of time, the feed concentration increased to a level of 70 °bx. During this process it was noted that OD flux decreased over time which corresponded with the feed concentration and the extent of concentration

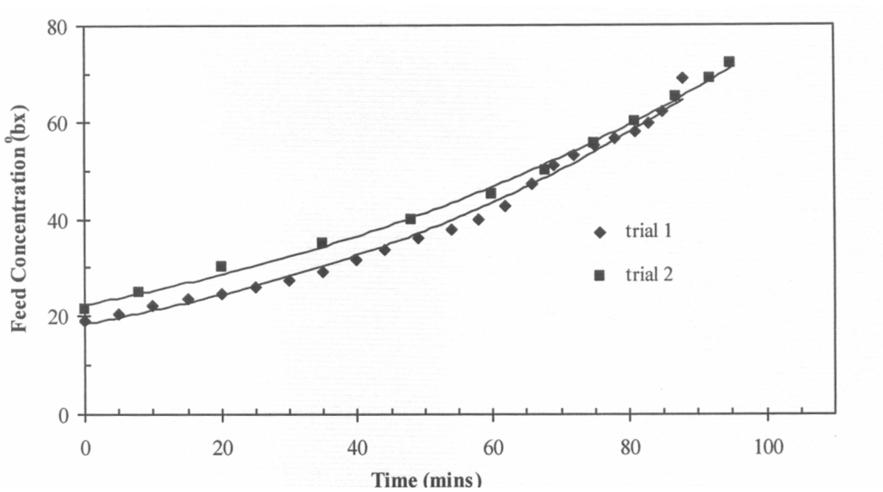

Figure 4.a. Concentration of grape juce over time during OD hollow fibre membrane module

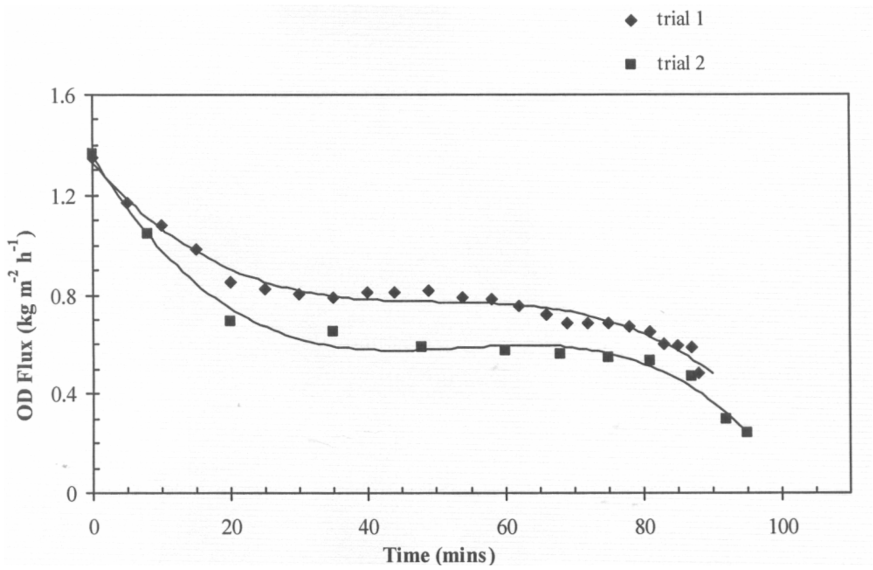

Figure 4.b. Comparison of OD flux over time during OD on grape juce using hollow fibre membrane

EFFECT OF PRELIMINARY FEED ULTRA FILTRATION ON OD FLUX.

The objective of this work was to determine the possibility for an improvement in flux during the concentration of grape juice, using a UF pretreatment step. The flux results of the initial experiments using UF membranes with values of 50 000 and 1 000 000 Daltons were compared with those obtained using untreated juice and are shown in Figure 4.1.2. The curve shows the variation of K with juice concentration.

It is shown that although UF pretreatment using the 50 000 membrane resulted in an insignificant increase in flux over that of the 1 000 000, both were considerably higher than that of the non treated 20 °bx juice at all juice concentrations. This may be attributed to an increase in water vapor pressure after UF due to improved transfer through the boundary layer after UF. It is also noted that the flux decreased with increasing feed concentration. As observed in the work on feed side mass transfer, the flux decreased more rapidly above 50 °bx, presumably due largely to a rapid increase in viscosity. [2]

WATER VAPOR PRESSURE

The transfer of water vapor across the membrane is driven by a vapor pressure gradient across the membrane. Ideally, the larger the difference in vapor pressure between the feed and stripper side, the higher fluxes that will be achieved. The results that UF has on the water vapor pressure of grape juice over the concentration range are shown in Figure 5. The vapor pressure of a UF permeate produced using the membrane was measured and compared with the vapor pressure of non treated grape juice. The results show that, although not a large difference, the vapor pressure of the UF juice is higher than that of the non treated juice. It is noteworthy that the vapor pressures for ultra filtered grape juice are similar over a common concentration range (3.2 - 2.7 kPa at 20 - 40 °Bx) whereas that for the untreated grape juice is considerably greater.. The hydrophobic components being oils/waxes should be removed by UF. The UF membranes are made dominantly hydrophobic, so little adherence on the membrane is expected.[3]

A noteworthy observation showing the effect of feed vapor pressure on OD flux for UF and non UF treated grape juice. The vapor pressure of both juices at various concentrations was plotted against the flux observed at that concentration. The figure shows that for any given feed vapor pressure, for the same stripper conditions, it resulted in a similar OD flux whether the juice was UF pretreated or not. This confirms the idea that the vapor pressure difference across the membrane is a limiting factor in the OD process. The fact that the vapor pressure of UF pretreated juice reaches higher levels than that of non

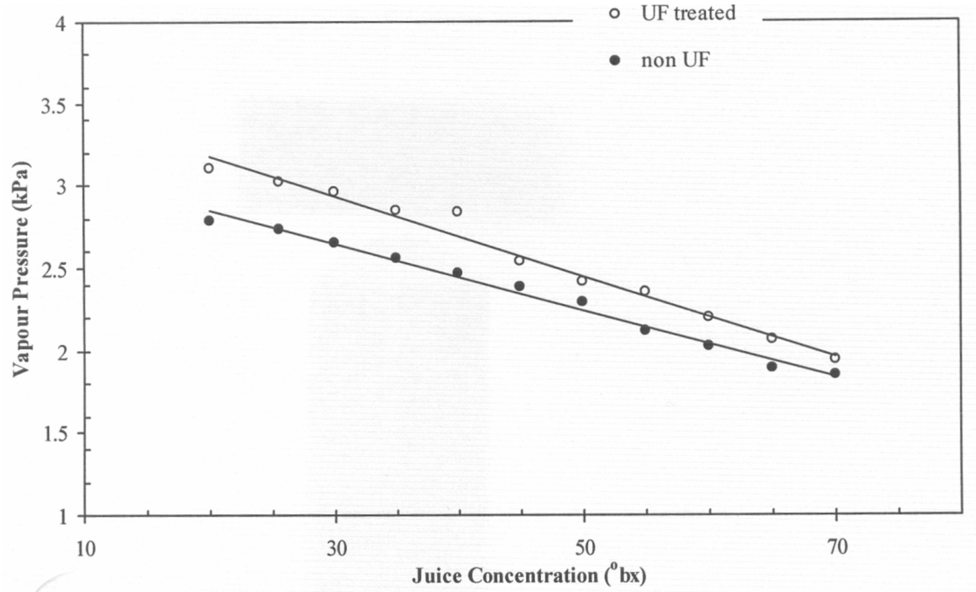

Figure 5. Effect of ultra filtration on vapor pressure of grape juice using UF membrane

CONCLUSION

Feed side resistance to mass transfer plays a significant part in the transfer of water vapour through the membrane particularly at low velocities. OD flux plateaus out at high velocities almost reaching the maximum equivalent to that of pure water, as a result the benefits of increasing feed velocity is limiting. Concentration polarization is minimized at high feed velocities

As feed concentration increases throughout the OD process, the rate of water flux (and in turn, overall mass transfer), decreases. Ultra filtration using membranes with pore diameters of 0.1 μ,m or less has been shown to result in increased flux during concentration of the permeate by OD. This has been attributed to the significant decrease the feed viscosity which causes a decrease in shear stress decreasing the pressure on feed velocity and in turn on the whole operating system. In addition to the higher OD flux, it was shown that the fermentable sugars content of untreated juice at 68 °bx could be achieved at a significantly lower Brix value of the ultra filtrated juice avoiding the problems of highly viscous feeds.[3]

It has also been shown that pretreating juices by UF, increases the vapor pressure of the juice which in turn increases the vapor pressure difference with the stripper. This has a positive effect on the OD system as the driving force of OD is the vapor pressure difference across the membrane. Although surface tension of the juice remained on average constant throughout the concentration process, there was an overall increase when the juice was pretreated with ultra filtration. This ensures the chance of wet-out is decreased prolonging the hydrophobicity quality and the life of the membranes. Ultra filtration has a significant and positive effect on the mass transfer across the membrane.

REFERENCES

1 . Attila Rekto Gyula Vatai and Erika Békássy-Molnár 2005 Multi-step membrane processes for the concentration of grape juice, Presented at the International Congress on Membranes and Membrane Processes (ICOM), Seoul, Korea

2 . A.M. Barbe, P.A.Hogan, R.A Johnson, A.F.G. Bailey, J. Sheng, 1999 The effect of ultra filtration on the subsequent concentration of grape juice by osmotic distillation, Journal of Membrane Science pp 1-10.

3 . R.A. Johnson, A.F.G. Bailey, 1994 Concentration of aqueous solutions by isothermal membrane distillation, Proceedings of the Workshop on Latest advances in Membrane Technology, Sydney, t, p.p 70.

4 . C. H. Shin, R.A Johnson, Determination of an appropriate osmotic agent formulation for use in OD, Journal of Membrane Science (in press).

5. R.S. Ramteke, N.I. Singh, M.N. Rekha, W.E. Eipeson 1993 Method for concentration of fruit juice:n: a critical evaluation, J.Food Sci. Technol., Vol 30, no 6, pp 391-402

6. A.M. Barbe, J.P. Bartley, A.L. Jacobs, R.A. Johnson 1998, retention of volatile flavor/fragrance components in the concentration of liquid foods by osmotic distillation, J. Membrane Sci, pp 67-75