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On the direct osmotic concentration of liquid foods. Part I: Impact of process parameters on process performance

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Abstract

In a series of two articles, this paper (part I) deals with the impact of process parameters on process performance, while part II deals with process modelling.

In this study, a pilot scale, direct osmotic (membrane) concentration unit was developed and its performance was studied. The unit was built around a flat geometry membrane module with a contacting area of 0.09 m^2 (1 $ft.^2$). Flat, square, reverse osmosis-type membrane sheets with varying characteristics were used with the osmotic module (cell). Model fluids, such as deionised water and low-concentration sucrose or glucose solutions, were utilized as feed fluids to study (and model) the impact of experimental parameters on process performance. Solutions of sodium chloride (NaCl) were used as osmotic media.

Membrane characteristics, feed and osmotic medium concentrations, feed and osmotic medium flow rates, all had a significant impact on the performance of the osmotic module, as measured by the water permeation flux. Experimental data gave a clear indication that at high flow rates compaction of the supporting membrane fabric can in fact cancel the positive effect of improved contacting (larger film coefficients) on water flux.

Keywords: Direct osmosis; Osmotic concentration; Membrane concentration; Liquid concentration; Juice concentration

1. Introduction

Liquid concentration is a crucial processing step for the liquid food processing industry. Main reasons for liquid food concentration include:

- weight and volume reduction, resulting in packaging, transport, handling and storage costs,
- water activity reduction, so as to enhance product stability,
- product preparation for a final (complete) drying treatment.

So far, the techniques mostly used in liquid food concentration include thermal concentration processes, such as evaporation and freeze concentration, plus membrane concentration processes, such as reverse osmosis with or without ultra-filtration as a preliminary processing step.

Most liquid food concentrates are made using evaporators. Heat is the cause of quality degradation problems due to loss of volatile (aroma) compounds and nutrients. Volatiles are lost with the first fractions of evaporated water, since volatile agents have much higher relative volatilities than water. Some of the volatiles can be recovered by vapor distillation, which is only partially successful, as these components are often mixed with non-condensable gases (Chardon, Quemarais, Schwartzberg, Iakovidis, & Lazarides, 1990; Lazarides, Iakovidis, & Schwartzberg, 1990). Besides, aroma recovery adds substantial cost to the overall concentration process.

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Product quality improvement and energy savings have lead to the development of alternative concentration techniques. ''Minimal processing'' techniques have always aimed at protection of fresh product characteristics from an organoleptic and nutritional point of view, with parallel reduction of processing (energy) costs. In fact, osmotic preconcentration of solid foods (i.e. fruits and vegetables) has appeared to be a rather attractive alternative to conventional air-dehydration schemes, stimulating extensive research interest (Lazarides, Fito, Chiralt, Gekas, & Lenart, 1999).

Efforts to solve quality degradation problems associated with evaporative concentration of liquid foods, have focused on so called ''cold concentration techniques''. Such techniques include: freeze concentration, reverse osmosis (RO) and osmotic concentration (OC).

Freeze concentration can produce a higher quality product, but it is faced with the following limitations (Petrotos, 1999):

- only clarified liquids can be concentrated,
- process productivity remains quite low,
- product losses are substantial,
- the process is energy intensive, as it involves a phase change,
- capital costs of a commercial—scale freeze—concentration plant are much higher than the cost of an evaporation plant for a similar throughput. Accordingly, the operating costs for freeze concentration are rather prohibitive.

A technical drawback of RO is concentration polarization and membrane fouling, especially under high pressures, which are necessary to succeed high product concentrations. Under such high pressures, membrane compaction is another major limitation.

A potential solution to the above problems and an attractive alternative to evaporative concentration techniques is offered by purely osmotic, low-pressure membrane techniques, namely *membrane distillation, osmotic distillation* and *direct osmosis*.

A preliminary evaluation of direct osmosis, membrane distillation and osmotic distillation, revealed certain advantages of direct osmosis over the rest two osmotic techniques. Such advantages include substantially cheaper membranes and a longer life cycle of direct osmosis membranes compared to hydrophobic membranes of osmotic or membrane distillation (Petrotos & Lazarides, 2001).

Direct osmosis is not a new processing technique. It has been traditionally used by East European farmers (Cussler, 1984). They used it to concentrate freshly squeezed fruit juices by introducing them in a semi-permeable cloth bag, which was then immersed in a concentrated brine (salt solution).

Based on this traditional process, a commercial process was introduced, where a bag containing juice was immersed into a bath of osmotic medium at elevated temperature, to improve water fluxes (Scott, 1975).

Other workers substituted the bag with a membrane (Popper, Camirand, Nury, & Stanley, 1966). They used reverse osmosis-type membranes made of cellulose acetate, to concentrate grape juice from 16 to 60 °Brix at a water removal rate of 2.5 l/m^2 h. Stirring improved water removal rate by 67%.

Beaudry and Lampi (1990a) suggested that direct osmotic concentration (DOC) is more effective, when a close (indirect) contact of a juice and an osmotic medium (OM) is established on opposite sides of a thin, semi-permeable membrane. The same workers proved that the DOC process is capable of concentrating pulpy and cloudy juices at reasonable rates (4.0 l/m^2 h) without pre-filtering (Beaudry & Lampi, 1990b).

A flat configuration was patented under the patent title: ''*Osmotic Concentration apparatus and method for direct osmotic concentration of fruit juices*'' (Herron, Beaudry, Jochums, & Medina, 1994). Besides, a tubular membrane configuration was used to study DOC of tomato juice (Petrotos, Quantick, & Petropakis, 1998).

A common characteristic of all previous work in DOC is working with real food systems (fruit or vegetable juices). Although such work is very useful in assessing process feasibility, it does not allow for a thorough and accurate study of process and product parameters, as the system is too complicated to manage clear isolation of experimental parameters. Besides, modelling of flux responses is quite difficult, due to feed variation induced process instabilities. According to fundamentals of dehydration system design, feed properties and final product specifications impose crucial process limitations, which define proper process conditions (Lazarides, 2003).

The main objective of this work was to study the impact of crucial experimental parameters on performance of a flat geometry, direct osmotic concentration rig. The idea was to use simple (model) fluids in order to readily explore this membrane process in terms of physical parameters.

2. Materials and methods

2.1. Experimental rig

The experimental rig consisted of the following parts (Fig. 1):

1. Flat membrane module along with its support pocket.
2. Cylindrical, jacketed, feed (''juice'') tank, constructed from stainless steel (SS 316) and having an internal diameter of 0.19 m and a height of 0.31 m. This tank was externally equipped with a jacket for circulating heating liquid or coolant to control the product temperature. In addition, a stirrer was adjusted on the top of the tank.
3. Cylindrical, osmotic medium tank, constructed from stainless steel (SS 316) and having internal diameter 0.295 m and height 0.295 m. A round flat piece of

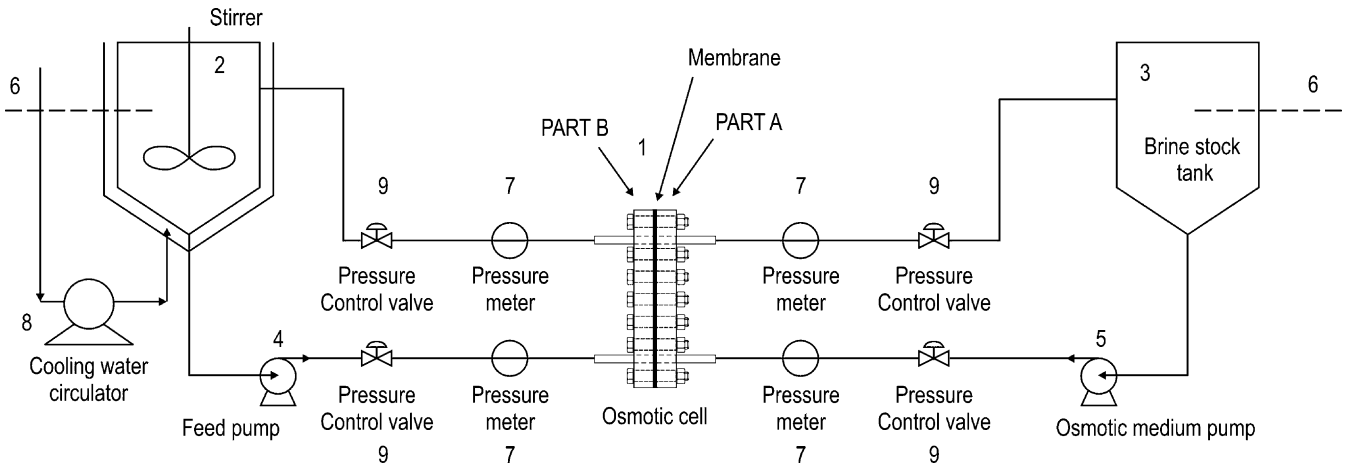

Fig. 1. Schematic diagram of experimental set up for direct osmosis.

SS 316 (diameter 0.26 m; height 0.02 m) was placed at the bottom of the tank to prevent swirling, which could cause pump feeding problems and unstable flow rates.

4. A centrifugal, feed pump (EBARA model CDM 70/7 Italy) driven by a 0.55 kW motor and delivering 20–80 l (water)/min.
5. An osmotic medium pump (same as No. 4).
6. Two Pt-100 thermocouples with digital output, each one connected in the feed (product) and osmotic medium tank, respectively.
7. Four analog pressure gauges to measure the inlet and outlet fluid pressure before and after the osmotic module.
8. A centrifugal type, cooling water circulator to circulate cooling water in the product tank jacket (RS 25/50r WILO—Germany).
9. Four flow- and pressure-control ball-type valves; two for controlling pressure at feed inlet/outlet and two for the osmotic medium line.
10. Plastic reinforced pipes with internal diameter 8 mm for the interconnections of the parts of the above mentioned experimental rig.

A photo of the experimental set up is shown in Fig. 2.

2.2. Membrane module

A flat shaped direct osmotic membrane module was constructed in order to accommodate the membrane used as a barrier between the two fluids involved in the process and to allow two flow-paths to be formed across the membrane.

The module consisted of two (34.3×34.3 cm) square pieces of synthetic material (Plexiglas) with thickness 1.4 cm. One of these two pieces (part A) formed the circulation chamber for the osmotic medium. It had a 28.4×28.4 cm recess, 1 mm in depth and two fluid distribution manifolds serving as fluid inlets and outlets. The second piece (part B) formed the chamber for the circulation of the feed fluid. It had a 28.4×28.4 cm recess, 3 mm in depth, covered by orthogonal shaped, parallel projections with 3 cm length and 0.5 cm height, thus forming channels 1 cm in width (distance between two parallel projections was 1 cm). Two fluid distribution manifolds (with four pipes each) were used for multiple point inlet and outlet of the feed.

The detailed morphology of the two pieces forming the direct osmotic module is presented in Fig. 3.

In order to install the membrane in the module the following procedure was followed: a piece of plastic net, 1 mm in thickness, was put in the recess of part A of the module. Consequently the flat membrane sheet was put in between two identical square gaskets and this sandwich was sited on part A. Finally, part B was put on top. The whole formation was held between steel reinforced frames by a set of 20 peripheral bolts.

In all experiments, the active membrane layer was put so as to face the feed fluid, while the membrane backing material was always facing the plastic net, which formed the flow micro-channels for the osmotic medium.

2.3. Membrane specifications

A reverse osmosis type, thin film composite, aromatic polyamide membrane was used in all direct osmosis experiments (Osmonics Inc., Minnetonka, MN, USA). Such a membrane consists of several layers (thin films), including a top-selective (active) layer, which selectively allows water transfer, and a bottom, backing layer. Detailed specifications of the four types of membranes which were used in this study can be found in Table 1. The membranes were supplied in square sheets, 1 ft. $\times$ 1 ft. The net contact area within the membrane module was 0.08 m^2.

2.4. Membrane cleaning and maintenance

Upon completion of each experimental run, the membrane was cleaned in place by using the P3 Ultrasil 10 alkaline detergent (Henkel-EcoLab Co-Greece).

Fig. 2. Experimental direct osmotic rig.

The cleaning regime involved three steps:

1. Initial rinsing for about 20 min with soft water.
2. Cleaning for 30 min by circulation of 1% detergent solution at a temperature 50–55 °C.
3. Final rinse of the system with soft water for at least 20 min.

All cleaning was performed under low pressure (<3 bar) and maximum flow rate.

Each time that the experimental work was interrupted for more than a day, the cleaned module was dismantled and the membrane sheet was properly preserved. The preservation process consisted of putting the clean membrane sheet in a container filled with 0.5% sodium bisulphate solution, with the active membrane layer facing upwards and keeping this container at 4 °C.

2.5. Experimental materials

In an effort to minimize unnecessary, feed induced complications, simple (model) liquids were used as feed materials in this study. Model feed liquids included deionized water and simple sugar (i.e. glucose or sucrose) solutions.

Deionized water was used for preparation of the osmotic media and experimental (model) feed fluids (sucrose or glucose solutions).

Pure (99%), highly concentrated (75 °Brix) glucose solutions were supplied by AMYLUM Hellas SA (Thessaloniki, Greece).

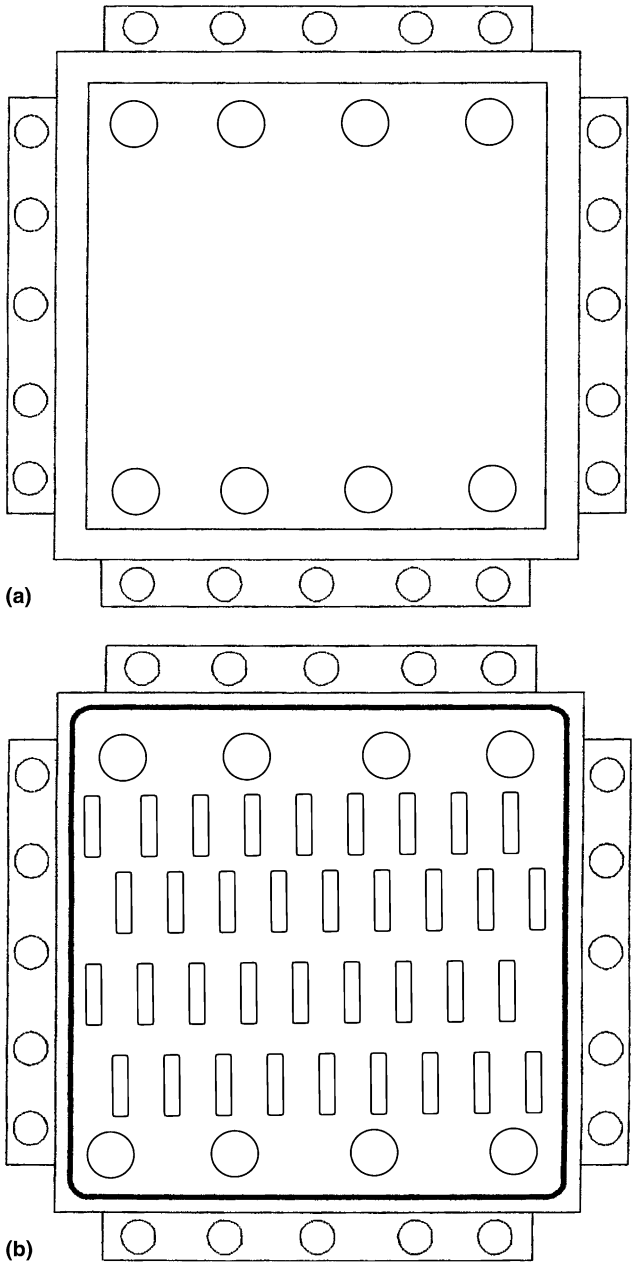

Fig. 3. Morphology of two parts of the direct osmosis module.

Pure (99.9%), crystallic sucrose was used to prepare thin, product (feed) solutions (Hellenic Sugar Industries SA, Thessaloniki, Greece).

Pure (99.9%) sodium chloride was used to prepare the osmotic medium solution (Kallas SA, Greece). Due to the need for refractive index readings, it was important to obtain clear (not cloudy) solutions.

2.6. Experimental procedures

The experimental procedure consisted of the following steps:

1. A certain quantity (6 kg) of feed solution was placed in the product tank and the agitator and cooling system were turned on.
2. A certain quantity (5 kg) of sodium chloride solution was placed in the osmotic medium tank and the centrifugal pump was put in operation for approximately 30 min to achieve solution homogeneity through re-circulation.
3. For a normal experimental operation, the product pump was first put in operation. When the first quantity of product (feed) returned to the feed tank, the back pressure valve was set at about 2 bar and immediately the osmotic medium pump was put in operation. Care was taken to maintain a positive pressure difference between the product side and osmotic medium side, so as to stabilize the membrane position on the plastic net and avoid contact (of membrane) with the sharp projections of the product chamber.
4. The normal run lasted 4 h. Throughout this time course both the osmotic medium and the product were sampled in regular time intervals (i.e. every 15 min for the first 90 min; every 30 min thereafter).
5. The refractive index of collected samples was measured by a high precision electronic refractometer (Stanley and Bellingham, UK model RFM340). A standard correlation between concentration and refractive index was used to determine concentration of sodium chloride solutions (Petrotos, 1999).
6. The drop in concentration of the osmotic medium during the 4 h run was used to calculate (by integration) the average permeation flux in the course of the run. This was considered to represent the mean concentration of the osmotic medium and product for the run.

2.7. Experimental treatments

Clear-cut experiments were designed to evaluate the impact of crucial experimental parameters on process performance (water flux). Detailed conditions of experimental treatments are presented in Tables 2–4.

Table 1
Membrane specifications

Membrane type	Thickness of top-selective layer (μm)	Thickness of backing material (μm)	Rejection coefficient (% NaCl)	Water permeation coefficient, A-value (l/m^2 bar h)
DS-3-SG	81.2	92.3	98.2	2.9
DS-3-SE	77.0	98.7	98.9	1.4
DS-3-SC	78.7	97.8	99.0	1.1
DS-11-AG	53.5	88.2	99.5	4.3

Each type of membrane was tested with the same feed (deionized water) at three different brine concentrations (5%, 15% and 23%) using the same experimental conditions (feed and medium temperatures/flow rates, in/out pressures, etc.).

Each experimental treatment was run in duplicate experiments.

2.8. Mass transfer calculations

Mean water flux values were calculated for each experimental run by averaging total water transfer (from feed to brine) over the time length of the experiment, taking into account the active membrane area. Flux values were, therefore, expressed in kg (water)/m^2 h.

Table 2
Experimental treatments[a] for studying the impact of feed (sucrose solution) concentration on permeate (water) flux

Brine		Feed (sucrose)		Osmotic pressure difference, ΔΠ (bar)
Concentration, % w/w (molality)	Viscosity (mPa s)	Concentration, % w/w (molality)	Viscosity (mPa s)	
10.0 (1.90)	1.191	3.0 (0.09)	1.08	90
10.0 (1.90)	1.191	6.0 (0.19)	1.18	89
10.0 (1.90)	1.191	18.0 (0.64)	1.79	75
23.0 (5.11)	1.745	3.0 (0.09)	1.08	306
23.0 (5.11)	1.745	6.0 (0.19)	1.18	303
23.0 (5.11)	1.745	18.0 (0.64)	1.79	291

[a] The following experimental conditions were established in the above experiments:
Feed temperature (°C) 22 ± 2
Brine temperature (°C) 31 ± 2
Feed flow rate (l/h) 630 ± 20
Brine flow rate (l/h) 115 ± 15
Feed pressure in/out (bar) 1.9/1.4
Brine pressure in/out (bar) 1.2/0.1
Membrane area (m^2) 0.08
Process duration (h) 4
Membrane type DS-3-SG.

Table 3
Experimental treatments[a] for studying the impact of feed (glucose solution) concentration on permeate (water) flux

Brine		Feed (glucose)		Osmotic pressure difference, ΔΠ (bar)
Concentration, % w/w (molality)	Viscosity (mPa s)	Concentration, % w/w (molality)	Viscosity (mPa s)	
10.0 (1.90)	1.191	3.0 (0.17)	1.083	88
10.0 (1.90)	1.191	6.0 (0.35)	1.179	83
10.0 (1.90)	1.191	18.0 (1.22)	1.757	60
23.0 (5.11)	1.745	3.0 (0.17)	1.083	304
23.0 (5.11)	1.745	6.0 (0.35)	1.179	299
23.0 (5.11)	1.745	18.0 (1.22)	1.757	276

[a] The rest experimental conditions were the same as in Table 2.

Table 4
Experimental treatments[a] for studying the effect of brine concentration on flux

Brine		Feed		Osmotic pressure difference, ΔΠ (bar)
Concentration, % w/w (molality)	Viscosity (mPa s)	Liquid	Viscosity (mPa s)	
5.0 (0.90)	1.083	Deionized water	0.890	41
10.0 (1.90)	1.191	Deionized water	0.890	92
15.0 (3.02)	1.352	Deionized water	0.890	158
20.0 (4.28)	1.541	Deionized water	0.890	243
23.0 (5.11)	1.745	Deionized water	0.890	308

[a] The rest experimental conditions were the same as in Table 2.

Based on the measured flux values, calculation of the *overall mass transfer coefficient* (U) was carried out by using the mass transfer equation

$$U = \frac{\text{Flux}}{\Delta\Pi - \Delta P}$$

where:

Flux is in kg/m^2 h.
ΔΠ is the osmotic pressure difference across the membrane (in bars).
ΔP is the hydraulic pressure differential across the membrane (in bars).

3. Results and discussion

3.1. Impact of membrane characteristics on direct osmosis flux

The impact of membrane characteristics on water permeation rates can be seen in Fig. 4. A first reading of this figure suggested that the membrane material could play a crucial role in process performance. Membrane type SC gave by far the lowest osmotic flux, more or less independent of the driving force (osmotic medium concentration). Membranes SG and SE gave different flux values at low brine concentration and practically identical performances at higher (brine) concentrations. At all brine

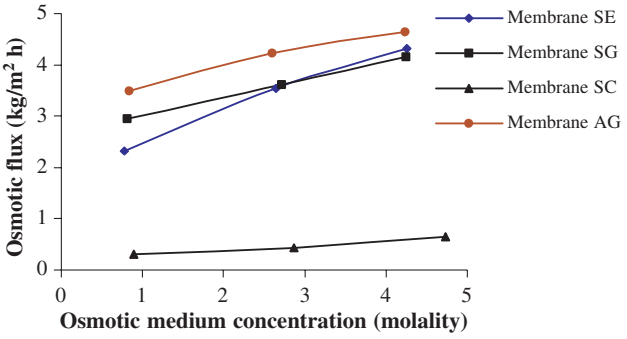

Fig. 4. Impact of membrane type (characteristics) and osmotic medium concentration on osmotic flux.

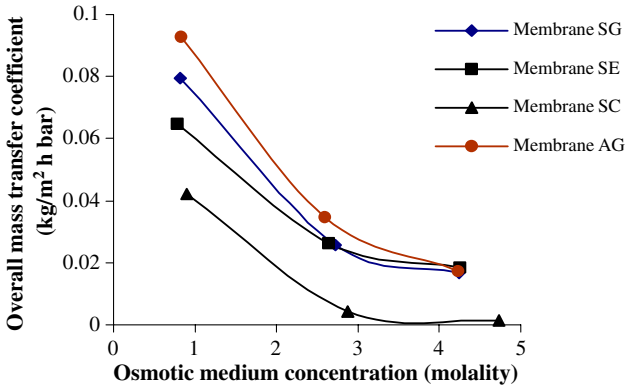

Fig. 5. Impact of membrane type (characteristics) and osmotic medium concentration on overall mass transfer coefficient (U).

concentrations the above two membranes were not as efficient in terms of water flux, as the AG-type.

With a closer look at membrane performance, it appeared that the three membrane types (AG, SE, SG) gave different performances at low brine concentrations, but they yielded practically identical results (in terms of transfer coefficients) at higher brine concentrations (Fig. 5). Again, membrane SC gave by far the lowest performance all along the tested brine concentration range.

The above results indicate that, at low brine concentrations membrane characteristics play a key role in mass transfer resistance. At higher brine concentrations, however, boundary layer resistances could play an equally important role, thus counter-acting the advantage (benefit) of a more permeable membrane.

According to Petrotos (1999), the membrane resistance can be analysed in two components. First comes the resistance of the top, ultra-thin selective layer, which is given by the reciprocal of osmotic coefficient (A). Second is the resistance of the backing material, which largely depends on its thickness (λ), porosity (ε) and tortuosity (τ). In fact, membrane type AG, that gave the best results, had the largest A-value (4.3 l/m^2 bar h) and the lowest thickness of backing material (88.2 μm), that is the lowest total membrane resistance, compared to all other types (Table 1).

3.2. Impact of osmotic pressure differential on direct osmosis flux

Osmotic flux increased linearly with the osmotic medium concentration (Fig. 6), but the total flux increase (by ca. 30%) lagged far behind the brine concentration (and $\Delta\Pi$) increase (by ca. 360%). This is a clear indication of a large mass transfer resistance.

In fact the mass transfer coefficient decreased by ca. 75% with increased osmotic medium concentration (Fig. 7). These results indicate the need for research to reduce mass transfer resistance on the brine side (the specific experiments were using water as feed liquid). Such an effort would require a two-side approach. One would be to improve the transport properties of the backing material

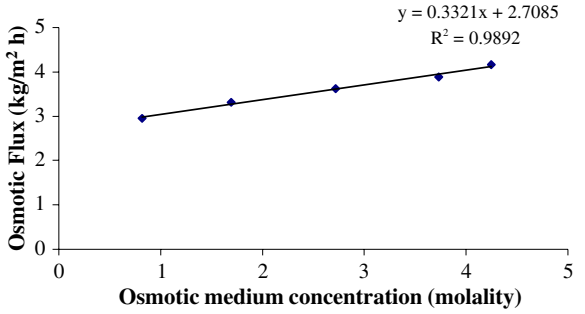

Fig. 6. The effect of osmotic medium concentration on the direct osmotic flux.

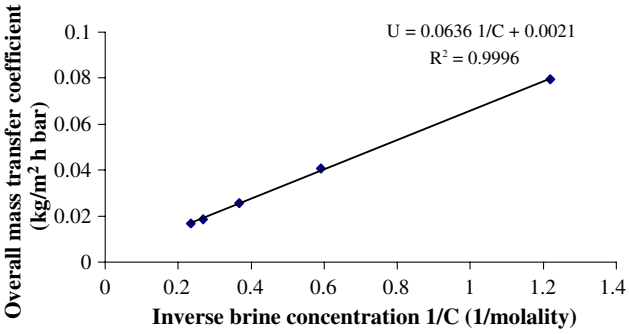

Fig. 7. Impact of osmotic medium concentration on overall mass transfer coefficient.

(probably by making it thinner). Second would be to improve contact conditions leading to improved film coefficients. Before this is achieved, a higher driving force cannot be efficiently utilized.

3.3. Impact of feed concentration on direct osmosis flux

A dramatic decrease in water flux took place, as feed concentration was increased from 3% to 18% (a molality increase from 0.1 to 0.6) (Figs. 8 and 9). The flux decrease appeared in a similar manner in both brine concentrations (10% and 23%).

It was interesting to find that at the highest sucrose concentration the two brine treatments gave practically identical water flux (Fig. 8), despite the tremendous difference in osmotic pressure differential ($\Delta\Pi = 75$ versus 291 bar for the two feed/brine combinations, see Table 2).

At higher feed concentrations there are two causes for decreased flux values; first, is the higher boundary layer resistance (on the feed side) due to increased viscosity and concentration polarization phenomena; second, is the decreased osmotic pressure differential. At higher brine concentrations (23% vs. 10%), a third mass transfer resistance becomes critical; the boundary layer resistance on the brine side. This resistance appears to be large enough to counter-act the positive impact of a four times higher osmotic pressure differential, which is associated with the 18% sucrose/23% NaCl combination, compared to 18% sucrose/10% NaCl combination.

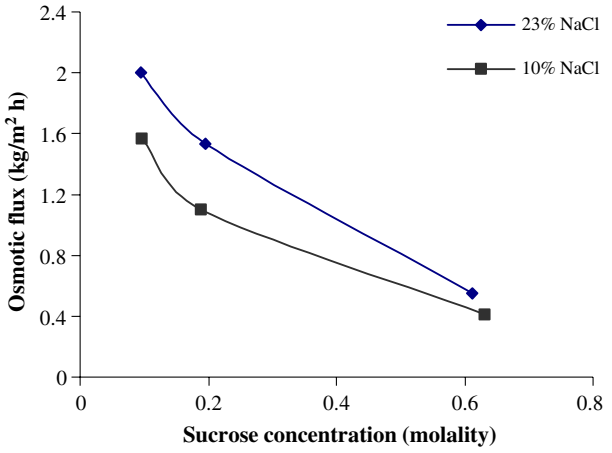

Fig. 8. Impact of feed (sucrose) concentration on osmotic flux.

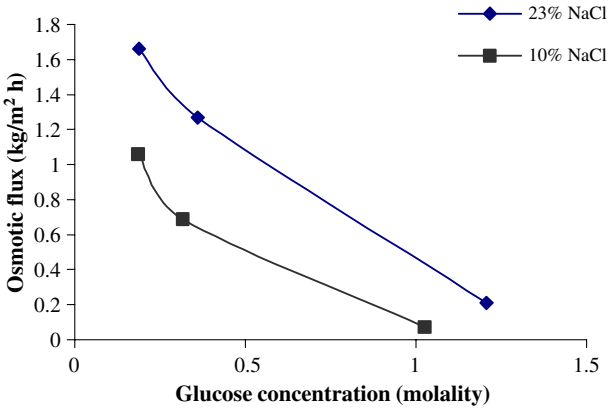

Fig. 9. Impact of feed (glucose) concentration on osmotic flux.

With the higher brine concentration (23%), at any molality level the osmotic flux of glucose solutions was always higher than the flux of sucrose solution. The differences were smaller for the lower brine concentration (Figs. 8 and 9). Such differences could be explained on the basis of different viscosities of the two feed solutions.

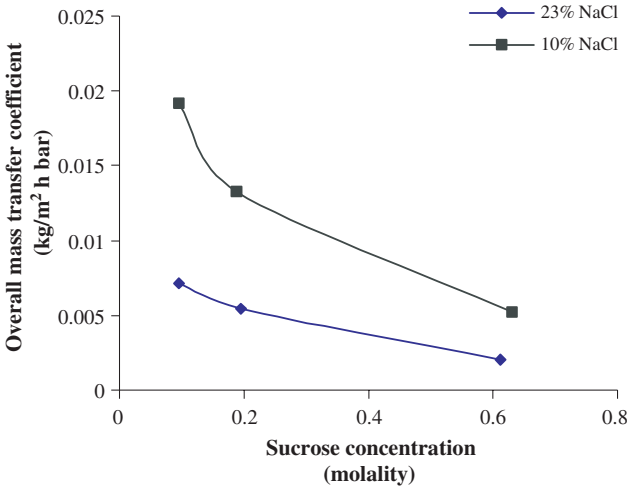

Fig. 10. Impact of feed (sucrose) concentration on overall mass transfer coefficient.

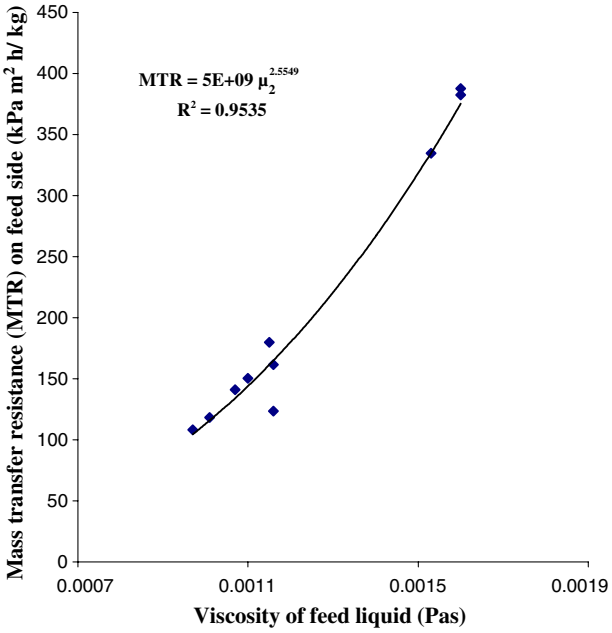

Fig. 11. Impact of feed viscosity on boundary layer resistance (R_2).

Another view of the above phenomena can be seen through comparison of the two overall mass transfer coefficient (U) curves (Fig. 10).

At low feed concentrations, the two brine concentrations gave highly different transfer coefficients. In fact the lower (10%) brine treatment gave more than a double coefficient value, compared to the higher (23%) brine treatment. Thus, the benefit of a drastically larger driving force (higher $\Delta\Pi$) was largely cancelled by a larger boundary resistance on the brine side. As the feed concentration was increased, the increasing feed-boundary layer resistance resulted in decreased transfer coefficients.

3.4. Impact of feed viscosity on mass transfer resistance on the feed side

The impact of feed viscosity on feed-mass transfer resistance is shown in Fig. 8. Although the specific module did not allow the use of a larger range of viscosities (due to large pressure drops), the strong impact of viscosity was quite clear. A doubling in viscosity caused a four-fold increase in mass transfer resistance on the feed side (see Fig. 11).

3.5. Impact of feed and medium flow rates

Increased flow rates are normally expected to result in increased flux values, since they have a positive impact on film (transfer) coefficients. In this study an opposite response was encountered; that is, increased flow rates gave decreased water flux values (Figs. 12 and 13). This unexpected behavior could only be explained by considering

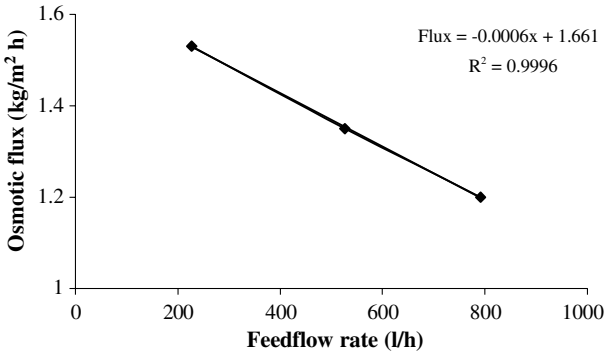

Fig. 12. Impact of osmotic feed flow rate on osmotic flux.

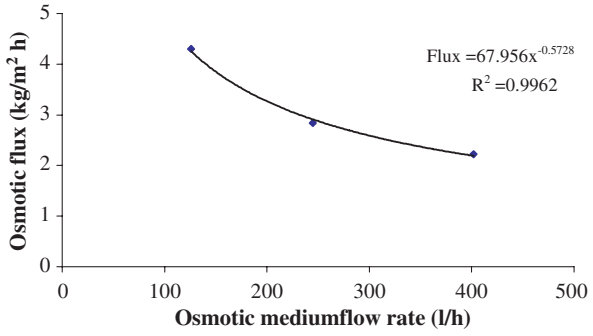

Fig. 13. Impact of osmotic medium flow rate on osmotic flux.

effects that could possibly counter-act improved contacting conditions. Such an effect is possible compaction of the membrane backing material at higher flow rates. In fact, mathematical analysis of the resistance components encountered in this experimental set up (part II) clearly supported the truth of this hypothesis, pointing to the need for improved membrane materials (more suitable for direct osmosis applications).

4. Conclusions

1. Membrane characteristics are very important at low osmotic medium (brine) concentrations, but they only play a minor role at higher concentrations, where boundary layer resistance and poor contacting conditions could result in a dramatic drop of overall mass transfer coefficients, thus counter-acting the benefit of a larger driving force.
2. Large osmotic pressure differentials can only be exploited with suitable membranes, when they are used under satisfactory contact conditions, leading to small boundary layer resistance and large overall transfer coefficients. Poor contacting conditions on the brine side can easily cancel the benefit of a larger driving force.
3. Large feed and (secondly) brine viscosities could pose serious limitations in obtaining satisfactory osmotic fluxes, especially under poor contacting conditions.
4. Further research should be focused on development of process specific membrane materials (i.e. membranes with thin, porous backing layer) and membrane holding modules that allow for excellent contacting conditions, especially at higher brine concentrations.

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