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On the direct osmotic concentration of liquid foods: Part II. Development of a generalized model

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Abstract

The performance of a pilot scale, direct osmotic (membrane) concentration unit was modeled in physical terms. Based on the fundamental equation for cross-flow mass transfer diffusion through membranes and an adequate set of experimental measurements, a generalized mathematical model for direct osmotic concentrators was successfully developed. The required number of independent equations was more than three times the number of the unknown parameters (constants) of the proposed model, in order to allow for modern non-linear problem solvers to converge to a satisfactory solution.

A Non-Linear Regression software package was used to determine optimal values for coefficients and exponents appearing in the proposed model. The validity of the emerging physical model was properly verified. The defined model provided an insight on the relative importance of several experimental parameters and membrane compaction in controlling direct osmotic flux values.

The proposed model and the described modeling approach could be used to develop physically meaningful models for other membrane processes.

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

1. Introduction

With respect to modeling of membrane processes, several attempts were made to obtain mathematical relationships that accurately correlate flux values with critical process parameters (Agashichev, 1998, 2001; Rouvet, Fiaty, Laurent, & Liou, 1998; Van Gauwbergen & Baeyens, 2001).

Most of the work has been focused in the area of reverse osmosis (RO) and ultra-filtration (UF) applications. In the area of DOC applications, a basic formula was suggested to describe the dependence of osmotic flux on the driving force and flow resistances (Beaudry & Lampi, 1990). Besides membrane resistance, total resistance included boundary layer resistances and fouling layer resistance. Non-fouling tests showed that fouling layer resistance is negligible. A similar finding was published by other workers (Wrolstad et al., 1993).

All in all, although DOC seems to be a promising process alternative for liquid food concentration, there has not been a systematic effort for detailed modeling of the process in physical terms.

The main objective of this work was to study and model 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 accurately model this membrane process in terms of physical parameters.

2. Materials and methods

2.1. Experimental set-up and procedures

The experimental rig, the membrane module, experimental materials, membrane cleaning—maintenance and

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experimental procedures were all described in detail in part I of this 2-article series.

2.2. Membrane specifications

A reverse osmosis type, thin film composite, aromatic polyamide membrane (DS-3-SG) with 98.2% rejection (as NaCl) was used in all direct osmosis experiments (Osmonics Inc., Minnetonka, MN, USA). Water permeation coefficient was $A = 2.9\ \mathrm{l/m^2\ atm\ h}$. The membranes were supplied in square sheets, 1 ft × 1 ft.

2.3. Experimental treatments

Detailed conditions of experimental treatments are presented in Table 1. The number of treatments was chosen to give a number of independent equations more than three times the number of constants to be determined from the proposed model. This was because the form of the model is clearly non-linear; a fact that introduces certain difficulties in obtaining a valid mathematical solution (i.e., determining coefficients and exponents of the model). Even known, effective algorithms are not always capable of solving certain non-linear problems, as they often fail to converge to a valid solution due to lack of adequate, reliable experimental data.

2.4. Modeling approach

Mathematical modeling is a useful tool to predict and quantify the behaviour of a unit operation. An adequate mathematical model contributes to a better understanding of the specific process and to a more effective design of equipment involved in the process. Furthermore, through the quantitative information incorporated in the model, there is a possibility to improve process performance.

Development of the model was based on the fundamental mass transfer equation, which correlates the direct osmotic flux to the driving force (i.e., osmotic pressure difference across the membrane) and the mass transfer resistances. The osmotic pressure difference ($\Delta\Pi$) was furthermore corrected by deducting the hydraulic pressure difference (ΔP) across the membrane (despite the fact that ΔP is negligible compared to $\Delta\Pi$).

Accordingly, the basic form of the model became

$$\text{Osmotic flux,}\ F = (\Delta\Pi - \Delta P)/R_{\text{total}} \tag{1}$$

where $\Delta\Pi$, osmotic pressure difference across the membrane; ΔP, hydraulic pressure difference across the membrane and R_{total}, total mass transfer resistance.

Total resistance can be analyzed in three terms, namely: membrane resistance (R_{m}), resistance of the osmotic medium boundary layer (R_{om}) and resistance of the boundary layer on the side of the feed liquid (R_{cl}). Thus,

$$R_{\text{total}} = R_{\text{om}} + R_{\text{m}} + R_{\text{cl}} \tag{2}$$

Table 1
Detailed experimental conditions for all treatments

Treatment code no.	Feed liquid (molality)	Feed flow rate, F_2 (m^3/h)	Feed temperature, T_f (°C)	Feed viscosity, μ_2 (Pas) × 10^3	Osmotic medium (molality)	Osmotic medium flow rate, F_1 (m^3/h)	Osmotic medium temperature, T_{om} (°C)	Osmotic medium viscosity, μ_1 (Pas) × 10^3	$\Delta\Pi$ (atm)	ΔP (atm)	Net pressure difference, ΔP_{net}
1	Water	0.680	27.0	0.85	NaCl (4.922)	0.138	33.4	1.16	247.8	1.1	246.7
2	Water	0.666	25.6	0.88	NaCl (4.868)	0.131	32.2	1.19	244.3	1.1	243.2
3	Water	0.713	26.7	0.87	NaCl (3.074)	0.119	33.1	0.91	142.4	1.1	141.3
4	Water	0.698	24.9	0.89	NaCl (4.235)	0.115	32.0	1.19	208.6	1.0	207.6
5	Sucrose (0.087)	0.605	23.3	0.97	NaCl (5.111)	0.132	31.0	1.21	280.8	1.1	279.7
6	Sucrose (0.186)	0.583	25.2	1.01	NaCl (5.100)	0.115	32.4	1.18	281.4	1.1	280.3
7	Sucrose (0.602)	0.601	25.0	1.53	NaCl (4.692)	0.130	33.1	1.17	273.2	1.1	272.1
8	Sucrose (0.622)	0.644	23.5	1.60	NaCl (1.990)	0.122	30.7	0.95	80.1	1.05	79.05
9	Glucose (0.351)	0.637	23.0	1.10	NaCl (4.863)	0.112	30.3	1.23	263.2	1.05	262.15
10	Glucose (0.311)	0.644	23.3	1.07	NaCl (1.939)	0.120	29.7	0.97	85.1	1.05	84.05
11	Glucose (1.021)	0.673	19.8	1.60	NaCl (1.929)	0.119	26.6	1.03	69.3	1.05	68.25
12	Water	0.558	21.5	0.97	NaCl (5.221)	0.402	30.4	1.23	282.9	0.94	281.96
13	Water	0.576	20.0	1.03	NaCl (5.213)	0.245	27.5	1.30	279.3	0.90	278.4
14	Water	0.580	20.5	1.00	NaCl (5.036)	0.126	26.9	1.31	251.5	1.1	250.4
15	Glucose (0.276)	0.792	19.7	1.15	NaCl (5.192)	0.131	26.7	1.32	287.7	0.67	287.03
16	Glucose (0.281)	0.526	19.6	1.16	NaCl (4.942)	0.130	27.1	1.31	268.8	0.90	267.9
17	Glucose (0.281)	0.227	19.5	1.16	NaCl (5.079)	0.131	29.9	1.21	279.5	0.77	278.73

According to Petrotos (1999), membrane resistance (R_m) consists of two components: First, is the resistance of the top, ultra-thin, selective layer that is the reciprocal of osmotic coefficient (A). This resistance can be determined by a simple reverse osmosis test. Second, is the resistance of the backing material (R_{md}) (fabric plus Ultra-filtration structure, if any). This gives

$$R_m = \frac{1}{A} + R_{md} \tag{3}$$

The same worker suggested that R_{md} depends on porosity (ε), tortuosity (τ) and thickness (λ) of the ultra-thin supporting layer structure and on diffusivity (D) of water in the osmotic medium solution. In fact, when tortuosity (τ) and thickness (λ) increase, the resistance (R_{md}) increases. On the opposite, R_{md} decreases, as diffusivity (D) and porosity (ε) increase.

Dimensional analysis was used to define resistances (R_{om}) and (R_{cl}), of the two liquid boundary layers across the membrane as follows:

$$R_{om} = k_1 \times \mu_1^{v1}/F_1^{s_1} \text{ and } R_{cl} = k_2 \times \mu_2^{v2}/F_2^{s_2} \tag{4}$$

However, preliminary experimental data (Figs. 1 and 2) indicated that osmotic flux is inversely proportional to the flow rate on both sides of the membrane. In turn this means that the two boundary layer resistances (R_{om}) and (R_{cl}) tend to increase, as the flow rate increases. At first sight this finding contradicts the classical mass transfer theory, as it is expressed in Eq. (4).

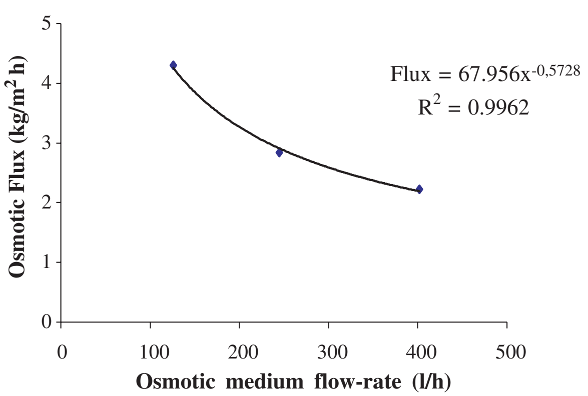

Fig. 1. Osmotic flux dependence on osmosis medium flow rate.

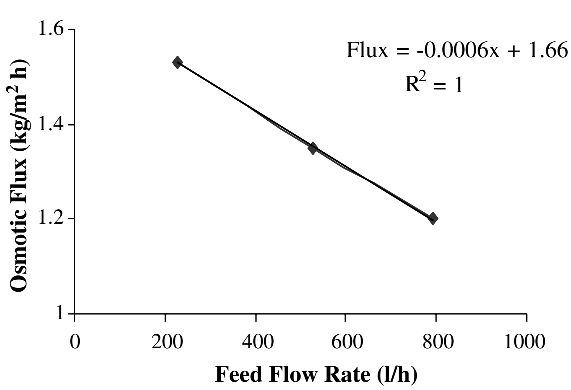

Fig. 2. Dependence of osmotic flux on feed flow rate.

Apparently a new hypothesis has to be made. The increased fluid velocities or (equivalently) flow rates of the two liquids across the membrane seem to exert mechanical stress that affects porosity and tortuosity of the compressible membrane backing material. Compression of this material results in reduced mass (water) transfer rates, strongly counteracting the positive effect of increased flow rates on flux, via decreasing polarization resistance. In turn, this means that the major part of mass transfer resistance is due to diffusion through the membrane backing material (and not due to resistance of the boundary layers).

In order to account for these mechanically induced effects, the resistance of backing material (R_{md}) was analyzed as follows:

$$R_{md} = k_3 \times \mu_1^{v3} \times F_1^{s_3} + k_4 \times \mu_2^{v4} \times F_2^{s_4} \tag{5}$$

This is because the mechanical stress developed on each side of the membrane largely depends on flow velocity (or flow rate, F) and viscosity (μ) of the liquid on that side of the membrane, as these parameters largely control the flow induced mechanical stress.

By using Eqs. (4) and (5), total resistance to mass transfer can be written as follows:

$$R_{total} = R_{om} + \frac{1}{A} + R_{md} + R_{cl}$$

$$R_{total} = k_1 \times \mu_1^{v1}/F_1^{s_1} + \frac{1}{A} + k_3 \times \mu_1^{v3} \times F_1^{s_3} + k_4 \times \mu_2^{v4} \times F_2^{s_4} + k_2 \times \mu_2^{v2}/F_2^{s_2}$$

$$R_{total} = (k_1 \times \mu_1^{v1}/F_1^{s_1} + k_3 \times \mu_1^{v3} \times F_1^{s_3}) + \frac{1}{A} + (k_4 \times \mu_2^{v4} \times F_2^{s_4} + k_2 \times \mu_2^{v2}/F_2^{s_2})$$

Term 1 of the above equation, also referred as resistance R_1, represents the boundary layer resistance on the osmotic medium side (R_{om}) plus the impact of the osmotic medium side on compression of the membrane backing material. Thus,

$$R_1 = k_1 \times \mu_1^{v1}/F_1^{s_1} + k_3 \times \mu_1^{v3} \times F_1^{s_3}$$

R_1 can be further simplified by multiplying and dividing each term by (F_1^{θ}) or ($F_1^{s_1+\theta}$), where (θ) takes a positive value such as $\theta \gg (s_1 + s_3)$ and $s > \theta$. Then

$$R_1 = k_1 \times \mu_1^{v1} \times F_1^{\theta}/F_1^{s_1+\theta} + k_3 \times \mu_1^{v3} \times F_1^{s_3+s_1+\theta}/F_1^{s_1+\theta}$$

or

$$R_1 = (k_1 \times \mu_1^{v1} \times F_1^{\theta} + k_3 \times \mu_1^{v3} \times F_1^{s_3+s_1+\theta})/F_1^{s_1+\theta} \tag{6}$$

The sum in the numerator of Eq. (5) can also be substituted by a mathematical formula of the type ($k \times \mu_1^{v} \times F_1^{s}$), where k, v, s take suitable positive values. Thus,

$$R_1 = k \times \mu_1^{v} \times F_1^{s}/F_1^{1+\theta} = k \times \mu_1^{v} \times F_1^{s-s_1-\theta} = C_1 \times \mu_1^{N_1} \times F_1^{s_1}$$

where C_1, N_1 and s_1 take positive values.

Similarly, term 3 of same equation, also referred as resistance R_2, represents the boundary layer resistance on the feed side (R_{cl}) plus the impact of the feed side on compression of the membrane backing material. In a similar manner, this term can be simplified to a similar form:

$$R_2 = k_4 \times \mu_2^{v4} \times F_2^{s4} + k_2 \times \mu_2^{v2}/F_2^{s2} = C_2 \times \mu_2^{N_2} \times F_2^{s_2}$$

Finally, R_{total} takes the form

$$R_{total} = C_1 \times \mu_1^{N_1} \times F_1^{s_1} + \frac{1}{A} + C_2 \times \mu_2^{N_2} \times F_2^{s_2} \quad (7)$$

Then, the final model for osmotic flux (F) becomes

$$F = (\Delta\Pi - \Delta P)/(C_1 \times \mu_1^{N_1} \times F_1^{s_1} + \frac{1}{A} + C_2 \times \mu_2^{N_2} \times F_2^{s_2}) \quad (8)$$

where $\Delta\Pi$, osmotic pressure difference across the membrane; ΔP, hydraulic pressure differential across the membrane; μ_1, μ_2, viscosities of osmotic medium and feed liquid, respectively; F_1, F_2, mass flow rates of osmotic medium and feed liquid, respectively and C_1, C_2, N_1, N_2, s_1, s_2 are positive constants.

2.5. Modeling software

Development of a mathematical model poses three general requirements:

1. a comprehensive set of accurate experimental data,
2. a general mathematical formula, which describes the process,
3. an effective software package to determine optimum values of coefficients, constants and exponents of the suggested mathematical formula based on accumulated experimental data.

The software package could be either a non-linear regression, best curve fitting programme or a combination of an Algebraic Modeling Computer Language, like GAMS (General Algebraic Modeling System). In the latter case, the program codes ("writes") the problem, while an effective solver applies an optimization algorithm to solve the problem. The objective is minimization of the sum of squared deviations of the model function, under certain (equality or inequality) constraints.

Determination of constants and exponents in the suggested model was achieved by use of the non-linear regression software package and algorithm (NLREG—www.NLREG.com). The package is capable of handling any kind of non-linear function, because it solves the problem without going though a linearization step of the non-linear function.

Confirmation and validation of the obtained solution were made by (additionally) describing the problem in GAMS language (General Algebraic Modeling System—www.GAMS.com), using the CONOPT optimization algorithm and solver (ARKI Consulting and Development A/S, Bagsvaerd, Denmark), in combination with a least-square objective function.

Certain rational constraints were imposed, in order to minimize run time and avoid unacceptable (though mathematically correct) solutions. Constraints were incorporated to the system of equations derived from mathematical data processing and served as boundary conditions for the problem.

3. Results and discussion

Measured flux values from all experimental together with the respective experimental conditions and the imposed constraints were fed into the NLREG software package and the model constants were determined (Table 2).

A high correlation coefficient ($R^2 = 0.9918$) indicated that experimental data are adequately described by the proposed model. This finding was also confirmed by the results of one-way analysis of variance, yielding an F-value = 200.58 ($P < 0.00001$) (Table 3). Finally, validity of the proposed model was further supported by the normal probability plot of residuals (Fig. 3).

Fig. 4 points to a very good agreement between experimental (actual) and (model) predicted flux values ($R^2 = 0.992$).

After incorporation of the calculated parameter values, the proposed model takes the following form:

$$F = (\Delta\Pi - \Delta P)/(3.14 \times 10^5 \times \mu_1^{1.07} \times F_1^{0.70} + 0.35 + 3.79 \times 10^9 \times \mu_2^{2.48} \times F_2^{0.32}) \quad (9)$$

Another approach to determine the constants of the model was attempted by using GAMS language for computer coding, in connection with the CONOPT optimization solver. The same constraints were applied and the least-square algorithm was used as the optimization

Table 2
Calculated[a] values for model constants (see Eq. (7), $A = 2.9$)

Parameter	Calculated value
C_1	3.1401663×10^5
N_1	1.06869415
s_1	0.69922718
C_2	3.7880692×10^9
N_2	2.48053176
s_2	0.317359888

[a] $R^2 = 0.9918$.

Table 3
One-way analysis of variance

Source	DF	Sum of squares	Mean square	F-value	Prob (F)
Regression	6	33.31816	5.553026	200.58	0.00001
Error	10	0.0276854	0.0276854		
Total	16	33.59501			

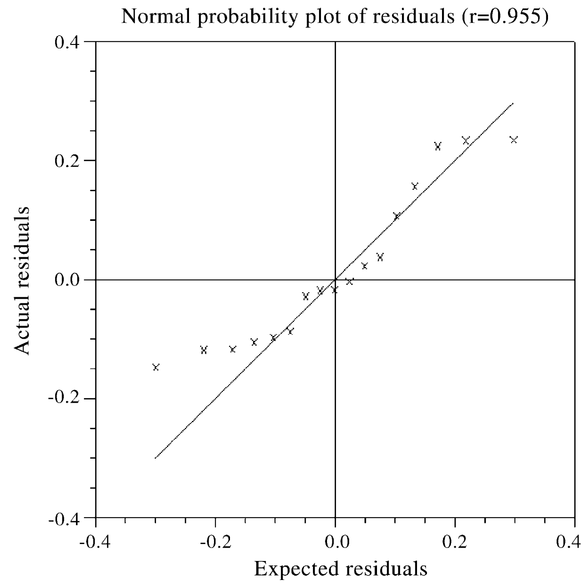

Fig. 3. Normal probability plot of residuals.

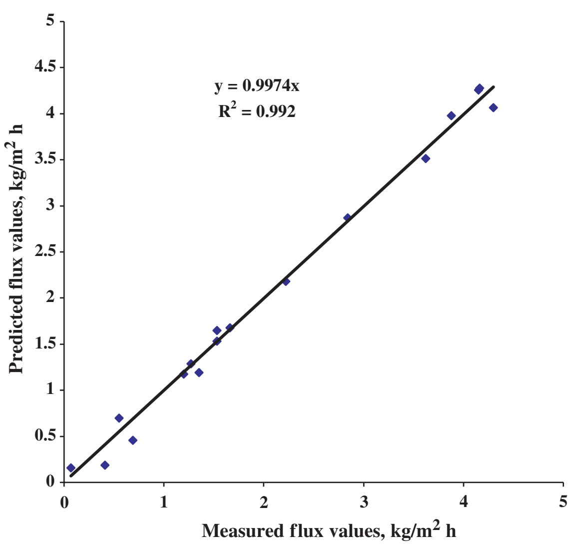

Fig. 4. Correlation between measured and predicted flux values.

principle (objective function). A comparison of GAMS/CONOPT results with NLREG (Table 4) shows that values of calculated constants are practically identical.

Using the model of Eq. (9), the magnitude of the two fluid resistances, namely the one on the side of osmotic medium ($R_1 = C_1 \times \mu_1^{N_1} \times F_1^{s_1}$) and the one on the side of the feed liquid ($R_2 = C_2 \times \mu_2^{N_2} \times F_2^{s_2}$) can be determined for all sucrose and glucose treatments (Table 5). A detailed comparison of the two resistances indicates that resistance (R_2) is significantly greater than the respective resistance of the osmotic medium (R_1).

Furthermore, considering the fact that the two exponents (s_1 and s_2) of F_1 and F_2 were found to be less than unit, the contribution of these exponential terms ($F_1^{s_1}, F_2^{s_2}$) is quite small. On the opposite, the exponents N_1 and N_2 are greater than unit; thus the exponential terms for fluid viscosities ($\mu_1^{N_1}$ and $\mu_2^{N_2}$) play an important role in determining the magnitude of total resistance (R_{total}).

The significantly greater size of flow resistance on the feed liquid side demonstrated that reduction of water diffusion was rather due to compaction of the UF layer than the compaction of backing fabric. This particular finding appears to challenge the well-established opinion that in purely UF applications, the broken curves in pressure–flux diagrams are exclusively due to the formation of a macromolecular polarization gel on the membrane surface (Chiang & Cheryan, 1986; Coulson & Richardson, 1991;

Table 4
Comparison of model constants calculated by two different optimization packages; namely, NLREG and GAMS/CONOPT

Model constant	NLREG calculated value	GAMS/CONOPT calculated value
C_1	314,016.63	313,992.76
N_1	1.069	1.069
s_1	0.699	0.699
C_2	3.788×10^9	3.759×10^9
N_2	2.481	2.479
s_2	0.317	0.329

Table 5
Calculation and comparison of mass transfer resistances (R_1, R_2) and relative contributions of exponential terms for 10 different treatments[a] with sucrose or glucose solutions as feed

Treatment code no.	Flow rates (F_1, F_2 (kg/s)) and viscosities (μ_1, μ_2 (Pas)) for osmotic medium (1) and feed (2)				Resistance		Resistance ratio	Values of exponential terms			
	F_1	$\mu_1 \times 10^3$	F_2	$\mu_2 \times 10^3$	R_1	R_2	R_2/R_1	$F_1^{s_1}$	$F_2^{s_2}$	$\mu_1^{N_1}$	$\mu_2^{N_2}$
5	0.13	1.21	0.61	0.97	58.13	108.33	1.86	0.243	0.85	1.23	0.93
6	0.12	1.18	0.58	1.01	51.39	118.35	2.30	0.220	0.84	1.19	1.02
7	0.13	1.17	0.60	1.53	55.49	334.79	6.03	0.240	0.85	1.18	2.87
8	0.12	0.95	0.64	1.60	42.48	382.38	9.00	0.230	0.87	0.95	3.21
9	0.11	1.23	0.64	1.10	52.74	150.43	2.85	0.216	0.87	1.25	1.27
10	0.12	0.97	0.64	1.07	42.94	140.95	3.28	0.227	0.87	0.97	1.18
11	0.12	1.03	0.67	1.60	45.52	387.76	8.52	0.226	0.88	1.03	3.21
15	0.13	1.32	0.79	1.15	63.46	179.99	2.84	0.241	0.93	1.35	1.41
16	0.13	1.31	0.53	1.16	62.61	161.50	2.58	0.240	0.82	1.33	1.45
17	0.13	1.21	0.23	1.16	57.82	123.69	2.14	0.241	0.62	1.23	1.45

[a] For detailed experimental conditions, see Table 1.

Lee & Howell, 1984; Renner & Abd El-Salam, 1991). According to the findings of this work, these curves could appear because of the compaction of the top (UF) membrane layer.

As a result, the main parameter controlling mass (water) transfer resistance is the viscosity of the feed liquid. As shown in Fig. 5, feed viscosity exerts a hyperbolic effect ($N_2 = 2.48$) on feed boundary layer resistance (R_2). At the same time, osmotic medium viscosity exerts a linear effect on its boundary layer resistance (Fig. 6). This finding is in line with claims reported in previous publications with respect to importance of fluid viscosities on the osmotic flux (Petrotos, 1999; Petrotos, Quantick, & Petropakis, 1998).

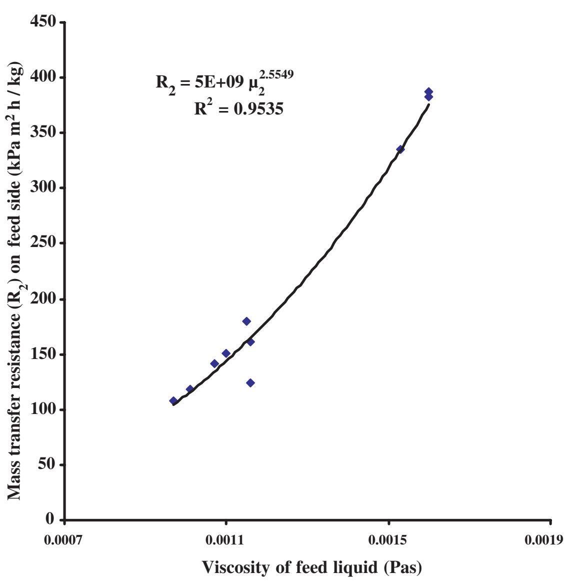

Fig. 5. Impact of feed viscosity on resistance (R_2).

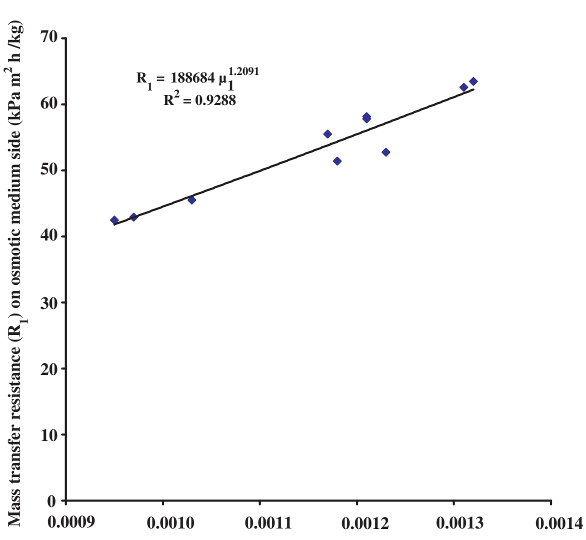

Fig. 6. Impact of osmotic medium viscosity on resistance (R_1).

4. Conclusions

1. Based on the fundamental mass transfer equation for osmotic diffusion and an adequate set of experimental measurements, a generalized mathematical model for direct osmotic concentrators was successfully developed. The number of independent equations was more than three times the number of the unknown parameters (constants) of the proposed model in order to allow for modern non-linear problem solvers to converge to a satisfactory solution.
2. Either pure non-linear regression software (like NLREG) or equivalent non-linear Algebraic Modeling software (i.e., GAMS or AMPL and the like) in combination with suitable solvers (i.e., CONOPT and the like) can be successfully used to obtain optimum values for model constants (i.e., exponents and coefficients).
3. The application of this general methodology yielded a quantitative model for a flat geometry direct osmotic concentrator. The defined model provided an insight to the effect of several process parameters on the magnitude of osmotic flux and revealed the important role of liquid viscosities in controlling direct osmotic flux values. Elaboration on these quantitative findings, revealed the important effect of membrane compaction on direct osmotic flux.
4. Finally, addressing the important issue of diffusion inhibition due to UF layer compaction calls for further research in using materials less susceptible to mechanical stress for manufacturing UF supports for the top selective layer of direct osmosis membranes.

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