University of Rhode Island

DigitalCommons@URI

Past Departments Faculty Publications (CHS)

College of Health Sciences

1999

Ultrafiltration/Reverse Osmosis Concentration of Lobster Extract

C. N. Jayarajah

University of Rhode Island

C. M. Lee

University of Rhode Island

Follow this and additional works at: https://digitalcommons.uri.edu/chs_past_depts_facpubs

Terms of Use

All rights reserved under copyright.

Citation/Publisher Attribution

Jayarajah, C. N. and Lee, C. M. (1999), Ultrafiltration/Reverse Osmosis Concentration of Lobster Extract. *Journal of Food Science*, 64: 93-98. doi: 10.1111/j.1365-2621.1999.tb09868.x Available at: <http://dx.doi.org/10.1111/j.1365-2621.1999.tb09868.x>

This Article is brought to you for free and open access by the College of Health Sciences at DigitalCommons@URI. It has been accepted for inclusion in Past Departments Faculty Publications (CHS) by an authorized administrator of DigitalCommons@URI. For more information, please contact digitalcommons-group@uri.edu.

Ultrafiltration/Reverse Osmosis Concentration of Lobster Extract

C. N. Jayarajah and C. M. Lee

ABSTRACT

A membrane concentration system consisting of tubular polysulphone ultrafiltration (UF) and polyamide reverse osmosis (RO) was evaluated for concentrating key water soluble flavor compounds from lobster extracts. Major flavor-giving compounds in the extract were glutamic acid, glycine, arginine, uridine 5'-monophosphate (UMP), succinic acid and glucose. Factors affecting performance of the UF/RO systems, such as flow rate, feed solid level, temperature and pressure, on permeate flux and solids rejection were measured. The optimum UF conditions were 1.5% feed solid level, 15 L/min feed flow rate, 50°C feed temperature and 1 MPa log mean transmembrane pressure. The RO system retained all dissolved flavor components and its ideal operating conditions were 40°C, 2.8 MPa log mean transmembrane pressure and a flow rate of 15 L/min.

Key Words: UF/RO, membrane concentration, lobster extract, flavor

INTRODUCTION

KEY FLAVOR CONSTITUENTS IN SEAFOOD are reported to be primarily water soluble including low molecular weight free amino acids, nucleotides, organic acids and sugars (Hayashi et al., 1978, 1979, 1981; Konosu et al., 1978). A synthetic mixture may be formulated based on a flavor profile, but sensory tests indicate that a formulated extract had mild and weaker "body" compared to a boiled crab extract (Konosu and Yamaguchi, 1986). Thus, a full bodied flavor extract may meet consumer demands if the flavor-giving compounds come from crabmeat. Major production concerns are reliability of raw material, recovery of volatile and nonvolatile components during extraction and potential loss of heat-labile flavor constituents during concentration.

Recovery processes may involve evaporation, freeze concentration or membrane separation. Evaporation may destroy thermally labile compounds and volatiles may escape (Koseoglu et al. 1990). Freeze concentration has been used to separate lobster flavor compounds without heat-induced flavor loss, but results in low yield (Jayarajah and Lee, 1987; 1988). Membrane processes using thin film tubular membranes with high solute retention have been commercially exploited in fruit juice concentration (Pepper et al., 1989). Alvarez et al. (1997) evaluated process variables in concentrating apple juice by RO using a polyamide tubular membrane. It is commercially

viable to separate protein from surimi waste streams by a thin film tubular composite UF membrane (Mohr et al., 1989). The RO membrane concentration is the most energy efficient of those processes and it should be economical to remove 2/3 of the water by RO and 1/3 by evaporation (Kessler, 1986).

Our major objectives were to evaluate the performance of a UF/RO tubular membrane system for lobster flavor recovery on the basis of permeate flux and solids rejection and to analyze flavor constituents in raw extracts.

MATERIALS & METHODS

Extraction procedure for feed solutions

The extraction process (Fig. 1) involved thawing lobster bodies in water to facilitate removal of carapace and sand, followed by washing in a jet stream of water to discard unwanted entrails. The clean lobster bodies were then ground at moderate speed using a meat grinder (Model A-120, Hobart Manufacturing Co., Troy, OH) with neither blade nor perforated plate, but with a screw installed. Minced product was combined with an amount of water half the weight of the mince and steam-cooked 20 min to extract water soluble flavor components and inactivate spoilage causing enzymes and microorganisms. The cooked diluted mince was filtered through cheese cloth to produce a filtrate (the first extract). The retentate was pressed using a Carver laboratory press (Carver Inc., Menomonee Falls, WI.) to a maximum pressure of 14 MPa to express mince juice. The combined first extract and pressed juice was used as feed stock for the UF/RO operation. Part of the extract was centrifuged at 2420g in a Sorvall refrigerated centrifuge (RC2-B, New-

town, CT) for 10 min to prepare supernatant and sediment for analyses of free amino acids, nucleotides and organic acids.

Proximate analysis

Proximate analysis was carried out according to AOAC (1975). The total protein nitrogen content of the fractions was determined by the micro-Kjeldhal method. The non-protein nitrogen content was determined after precipitating the protein with 10% trichloroacetic acid.

HPLC analysis of free amino acids

Free amino acids were analyzed by a Perkin-Elmer HPLC Series 4 Solvent Delivery System (Perkin-Elmer Corp, Norwalk, CT) equipped with a cation exchange column (AA 911, Interaction Chemicals, Mountain view, CA) and a Rheodyne injector (Cadet, CA) with a 6 μ L loop. Total protein nitrogen was determined on 6N HCl hydrolyzed samples. After evaporating to dryness, samples were dissolved in 5 mL 0.1N HCl and filtered through 0.22 μ m cellulose nitrate filters (Whatman, Clifton, NJ) prior to injection onto the HPLC column. Analysis was done in triplicate. For HPLC analysis, the mobile phase consisted of two sodium citrate buffers of increasing pH (3.15 and 7.40) and was pumped at 0.5 mL/min. A step-wise gradient elution was followed for 60 min. The HPLC grade sodium citrate buffers (Pierce, Rockford, IL) were filtered through 0.45 μ m Nylon-66 filters (Ranin Instrument Co., Woburn, MA) and degassed using helium before analysis. Free amino acids were identified from retention time using amino acid standards in 0.1N HCl. The separated amino acids from the column were derivatized on a post-column reaction system which consisted of two Perkin-Elmer Series 10 reagent pumps for OPA and NaOCl solutions. The reaction was temperature controlled at 52°C. The isoindole derivatives formed were detected by a LC-10 Perkin-Elmer filter fluorescence detector with excitation at 350 nm and emission at 418-700 nm. Data collection and calculation based on areas were performed with Chrom2 Software on a Perkin-Elmer 3600 Data station connected to a Perkin-Elmer 660 printer.

HPLC analysis of nucleotides

The nucleotide analysis was carried out following the method developed by McKeag and Brown (1978) using a Perkin-Elmer Series 3B liquid chromatograph fitted with a

Authors Jayarajah and Lee are affiliated with the Dept. of Food Science & Nutrition, Univ. of Rhode Island, Kingston, RI 02881. Address inquiries to Dr. C. M. Lee.

Rheodyne injector which had a 6 μ L loop. The system consisted of a strong anion exchange, Partisil 5 SAX RACII column (4.6 mm $\times$ 10 cm) (Whatman, Clifton, NJ). The ion-exchange moiety was a quaternary nitrogen which was Si-O-Si bonded to partisil. The column was protected by a guard column (5 cm $\times$ 4.6 mm) filled with pellicular C₁₈ packing material (Whatman). Doubly-deionized water was used to make all buffers. The pH of low concentration aqueous buffer consisting of 0.007M KH₂PO₄ and 0.007M KCl was adjusted to 4.0 with dilute phosphoric acid. The high concentration eluent was made with 0.25M KH₂PO₄ and 0.50M KCl and its pH was adjusted to 5.0 with dilute KOH. All solvents were filtered through a 0.45 μ m Nylon-66 filter and degassed with helium prior

to use. Initially an isocratic elution with a low concentration mobile phase was carried out for 5 min followed by a 35 min linear gradient (0–100%) elution and finally an isocratic elution with a high concentration buffer. Each run lasted for 65 min including 15 min of reequilibration. The separation of compounds was conducted at 50°C and a flow rate of 2.0 mL/min. The column was flushed with doubly-deionized water and stored in HPLC quality 100% methanol when not in use. Sample preparation was similar to that reported by Khym (1975). The protein was precipitated by addition of 2 parts of trichloroacetic acid (6% by weight) and centrifuged. After the pH of the supernatant was adjusted to neutral, the supernatant was filtered through 0.22 mm Nylon-66 filters (Ranin Instrument Co,

Woburn, MA) and injected onto the HPLC column in triplicate. The nucleotide standards obtained from Sigma Chemicals (St Louis, MO) were diluted to 0.10 mM with doubly-deionized water. The standards were stored at –20°C. The eluted nucleotides were detected at 254 nm and 0.10 AUFS sensitivity using a variable length Perkin-Elmer LC-95 UV/Visible Spectrophotometer, and identified from retention times. Data collection and calculation were done in the same setup used for amino acid analysis.

HPLC analysis of organic acids, sugars and alcohol

Analyses of organic acids, sugars and alcohol followed the method of McCord et al. (1984) using a Perkin-Elmer Series 4 Solvent Delivery System equipped with Aminex HPX-87H ion exclusion column (Bio-Rad, Richmond, CA). The column was held at 65°C. The column was protected by a Bio-Rad Micro Guard cation H⁺ cartridge. The set-up also had a Rheodyne injector fitted with a 6 mL sample loop. The mobile phase consisted of 0.05N sulfuric acid (Fisher Scientific, Fairlawn, NJ) which was filtered through 0.45 μ m Nylon-66 filters (Ranin Co, Woburn, MA) and degassed with helium. The flow rate was maintained at 0.6 mL/min throughout the isocratic elution. The sample was filtered using a SEP-PAK C₁₈ cartridge (Millipore-Waters Chromatography Division, Milford, MA) and the filtrate was injected in triplicate. The standards were diluted to 1% solution by weight with double-deionized water. Eluted compounds were identified from retention times using a refractive index detector (RF401, Waters Associates). Data collection and handling were carried out as described previously.

Experimental set-up for pilot scale UF/RO system

The type of membrane module used in the pilot scale experiment was B1 (10 cm $\times$ 121 cm; effective area 0.9 m²; channel height 12.5 mm) (Paterson Candy International, Whitchurch, Hampshire, England). The module was supplied by APV Crepaco (Tonawanda, NY). According to Hedges and Pepper (1984) and Hanisch (1986), the high feed velocity in the tubular system provided a self-cleaning action and increased shear stress at the membrane surface, making it less prone to fouling than nontubular designs. Orange juice fouling has been overcome by the PCI tubular unit at a minimum velocity of 2.5 m/s (Pepper et al. 1977).

The module resembled somewhat a tube-in-shell heat exchanger. The module consisted of 18 tubes connected in series and mounted inside stainless steel support tubes. The tubes were porous to allow fluid to permeate. The tubes were installed inside a stainless steel shroud with end-caps attached on either side of the terminal end. The module was flushed thoroughly with Monarch RO 115 detergent

Fig. 1—Flow chart of lobster juice extraction process.

(H.B. Fuller Company, Minneapolis, MN) for 4h, followed by tap water and deionized water prior to installation of membrane. The membrane used in the ultrafiltration system was PU 120 made with polysulphone with the specifications of pH range 2–12, 15 bar maximum pressure, 70°C maximum temperature tolerance, and 20,000 M.W. cutoff. The membrane used for RO was AFC 99 and was made of polyamide with specifications of pH range 3–11, 70 bar maximum pressure, 70°C maximum temperature tolerance, and 99% NaCl rejection. These membranes were manufactured by Paterson Candy International and were supplied by APV Crepaco.

The feed was circulated by a Tonkaflo Multistage Centrifugal Series 1800 pump (Osmonics, Minnetonka, MN) from a 100L capacity tank (Fig. 2). This high pressure pump consisted of all stainless steel and plastic wetted parts. The other major parts of the system were the module inlet and outlet pressure gauges (0–7 MPa range), and retentate flow meter. All high pressure side tubings, valves and fittings were made of 316 stainless steel. Tygon tubing was used in the low pressure side. The feed was maintained at the desired temperature by running retentate through a 18m stainless steel tubing (1.27 cm OD) coil which was immersed inside a tank filled with running tap water.

The feed was pumped through a flow con-

trol valve (by-pass line) and pressure control valve into the module inlet. The flow rate was adjusted by valve settings on the by-pass line and module inlet valve. The operating pressure was controlled independently of the flow rate by settings of module inlet and outlet (back pressure) valves. The pressure inside the module was read from gauges on the inlet and outlet lines. The flow rate was read from the flow meter attached to the retentate line. A volumetric cylinder was used to measure permeate flux over 1 min. The temperature of the feed was kept with minor fluctuations of $\pm 2^\circ\text{C}$ by the heating effect of the pump and the cooling effect of water. Flow rate of cooling water was adjusted to achieve a desired feed temperature.

Preliminary experimental procedure

Membrane compaction is defined as a permanent change in membrane which causes a steady decline in water flux with time (McCutchan, 1977). Water flux of membrane was taken as a measure of initial membrane performance and any membrane compaction. Immediately after installation of the system was flushed with deionized water to remove packaging preservative (Proxel ICI and Glycerol). Then water flux was measured as a function of pressure. For RO, deionized water was used while tap water was used for the UF system. The flux was measured at 10, 15 and

20 L/min flow rates. The log mean transmembrane pressures across the UF tested were 0.34, 0.51, 0.68, 0.86, 1.03, 1.20 and 1.37 MPa. All operating flow rates and pressures were carried out independently at 30, 40 and 50°C. The initial conditions employed before changing variables were, flow rate = 15 L/min, log mean transmembrane pressure = 0.86 MPa, feed temperature = 30°C and the operation time = 60 min. Pressure was increased every 5 min and water flux was measured at the end of each pressure level. Rudie et al. (1985) stated that significant flux reduction occurred within the first 10h of operation. Hence membrane flux was measured every hour for about 27h as a measure of membrane compaction. The constant levels of parameters maintained during 27h were log mean transmembrane pressure = 1.03 MPa, flow rate = 15 L/min and feed temperature = 30°C. Following the 27h operation, the water flux as a function of pressure experiment was repeated. The same procedure was followed for the RO system except for the following operating pressures. The log mean transmembrane pressures held were 0.68, 1.38, 2.07, 2.76, 3.45 and 4.14 MPa. During the constant 27h operation, the log mean transmembrane pressure was maintained at 2.41 MPa.

It was also essential to determine the time required for the UF/RO system to reach initial steady-state operation. Permeate flux and solids rejection characteristics of the membrane were taken as criteria for determining steady-state over 1h. Permeate flux was measured every 10 min during the 1h of operation. Samples were collected at these intervals for total solid determination. The constant levels of log mean transmembrane pressures in the UF were held at 0.68, 1.03 and 1.37 MPa. However, RO was operated at 1.38, 2.06 and 2.75 MPa. The flow rates were held at 10, 15, 20 L/min for both systems. The feed solids used in the UF were 0.5, 1.5 and 3.0% and those of RO were 0.5, 2.0 and 3.5%. The temperature of the feed was maintained at 30°C throughout all runs. The membrane system was allowed to run at a log mean transmembrane pressure of 1.03 MPa for the UF and at 2.06 MPa for the RO, both at 15 L/min flow rate for 1h prior to changing variables.

In order to evaluate possible membrane fouling, permeate flux and solids rejection were monitored every hour over a 6h period under steady-state operating conditions at 15 L/min and 30°C. The log mean transmembrane pressures applied for the UF were 0.68 and 1.03 MPa and that of RO was 2.41 MPa.

Final experimental design

In the final UF/RO experimental design, permeate flux was measured as a function of pressure within fixed levels of feed solids, flow rate and feed temperature. The levels of log mean transmembrane pressures used in the UF were 0.68, 0.85, 1.03, 1.20 and 1.37 MPa. The feed solids tested were 0.5, 1.5 and 2.5%. The pressure was increased every 10

Fig. 2—Schematic of flow loop for a pilot scale UF/RO system. (P1 = inlet pressure gauge; P2 = outlet pressure gauge.)

Table 1—Proximate composition of raw material and feed solution (%)

Material	Moisture	Protein	NPN ^a	Fat	Ash
Meat	82.73	13.56	5.33	1.43	2.16
Feed	97.28	1.65	1.06	0.09	0.90

^aNPN=nonproteinaceous nitrogen.

min and permeate flux, permeate solids and retentate solids were determined after each pressure increase. The permeate flux was measured by collecting permeate over 1 min. The flow rates studied were 10, 15 and 20 L/min at 30, 40 and 50°C feed temperatures. The flow rate and log mean transmembrane pressure were maintained at 15 L/min and 1.03 MPa, respectively, for 1 h prior to changing variables. Identical procedure was followed for the RO with the following changes. The feed solids evaluated were 0.5, 1.5 and 3.0%. The log mean transmembrane was held at 2.06 MPa during the steady period. The log mean transmembrane pressures tested in the final design were 1.37, 1.72, 2.06, 2.41 and 2.75 MPa. To ensure consistent performance of the UF/RO pilot scale operation, the start-up and shut-down procedures were strictly followed and the efficiency of the membrane was monitored by measuring water flux after each cleaning.

Determination of log mean transmembrane pressure and solids rejection

The inlet and outlet pressures were read from gauges fitted on the respective ports. The log mean transmembrane pressure was calculated using equation.

$$p = [(P_1 - P_2)/\ln(p_1/p_2)]$$

where p_1 is the inlet pressure and p_2 is the outlet pressure.

The rejected solids on the surface of membrane was calculated using equation:

$$R = [(C_b - C_p)/C_b] \times 100$$

where C_p is the permeate concentration while C_b is the average bulk concentration. The average bulk was calculated by taking the average of retentate solids content and feed solids content.

RESULTS & DISCUSSION

Lobster extract

The cleaned lobster bodies without the carapace constituted 58% by weight of the total raw material, and gave a final supernatant with a solids content of 2.72% (Table 1). The nonprotein nitrogen fraction (NPN) of the supernatant after centrifugation which contributes key flavor components was 1.06% by weight. Essentially, the major portion (66% by weight) of the NPN fraction consisted of free amino acids. Among those present, glutamic acid, glycine and arginine were high-

Table 2—Amino acid composition at different stages of lobster juice extraction process (HPLC data, mg/100 mL)

Amino acids	Meat		Extract		Sediment		Supernatant	
	Total	Free	Total	Free	Total	Free	Total	Free
Tau	179.69	164.78	87.73	86.78	83.79	80.08	84.67	82.40
Asp	1259.8	59.50	96.55	28.83	1156.3	31.46	85.46	25.60
Thr	432.95	28.90	33.29	12.25	346.33	9.52	34.10	12.10
Ser	276.65	20.54	24.14	9.37	172.04	9.04	29.01	10.70
Glu	1858.1	116.40	166.47	66.29	1421.6	54.63	153.50	61.10
Pro	679.91	105.95	71.58	53.84	472.02	46.05	69.78	51.50
Gly	659.59	260.65	126.18	106.47	522.30	88.09	117.01	100
Ala	725.23	129.35	94.39	64.58	558.60	52.62	87.62	60.50
Val	597.07	29.00	42.80	15.37	594.91	10.50	37.63	14.60
Met	276.65	22.23	17.98	NDT	206.68	4.15	13.17	14.60
Ile	622.07	18.92	33.13	9.26	530.67	7.49	29.99	18.60
Leu	997.19	38.40	64.92	20.39	838.00	14.70	56.64	27.40
Tyr	373.56	13.13	17.50	NDT	413.36	5.23	15.60	NDT
Phe	522.04	26.62	21.31	NDT	558.60	6.78	17.52	NDT
Lys	1051.9	84.18	75.58	32.74	810.00	24.77	15.66	27.40
His	275.09	18.75	26.14	6.49	385.43	6.72	81.34	7.15
Arg	1034.7	178.10	103.54	63.08	709.42	61.78	98.78	61.10

^aNDT=not detected.

est in quantity (Table 2). The nucleotide fraction was comparatively very low and uridine 5'-monophosphate (UMP) was the most predominant, while succinic acid followed by acetic acid and glucose were the other principal flavor components identified (Table 3). The distribution of inosine 5'-monophosphate (IMP) and guanosine 5'-monophosphate (GMP) at a ratio of 1:7 in the extract (both known as umami taste potentiators) was the same as that (0.39 to 2.80 mg/100g) in the lobster body meat hydrolysate (Yang, 1998).

Steady-state and fouling of pilot scale UF operation

In general, within constant flow rates, higher pressures always produced increased permeate flux because of the upsurge in driving force. Essentially, the permeate flux remained relatively constant at all 10 min intervals. Therefore, the initial 10 min waiting period with constant operating variables was reasonably adequate to reach steady operation. Thereafter the filtration would occur with gel polarized flux as proposed by Cheryan (1986). Results of the study on long term fouling effects of UF with different solids contents showed that the permeate flux remained nearly constant over a period of 6h for all solids levels except the 1.5% feed which gave a small variation in permeate flux.

Steady-state and fouling of pilot scale RO operation

When changes in permeate flux of feeds at three solids levels were observed with time in the pilot scale RO operation (Fig. 3), all flow rates tested within a pressure level at 0.5% solids gave almost constant permeate flux. The pressure level with a value of 2.75 MPa gave the highest permeate flux of 32 L/m²h followed by 2.06 MPa with 23 L/m²h permeate flux. The 1.38 MPa pressure level produced the least flux representing 14 L/m²h. In reverse osmosis permeation rate is mainly pressure dependent. The effect of osmotic pressure on permeate flux is more pronounced than concentration polarization and fouling

Table 3a—Nucleotides in lobster extract supernatant

Nucleotides	mg/100 mL
5'-CMP	1.46
5'-UMP	69.0
5'-IMP	0.07
5'-GMP	0.50
5'-AMP	0.13
5'-CDP	NDT ^a
5'-UDP	1.25
5'-IDP	NDT
5'-GDP	NDT
5'-ADP	NDT
5'-CTP	NDT
5'-UTP	0.25
5'-ITP	NDT
5'-GTP	0.05
5'-ATP	NDT

^aNDT=not detected.

Table 3b—Organic acids and sugars in lobster extract supernatant

Components	g/100 mL
Glycogen	+
Glucose	15.3
Fructose	NDT ^a
Arabinose	NDT
Ribose	+
Succinic acid	359.30
Lactic acid	+
Acetic acid	26.10
Propionic acid	NDT
Inositol	NDT

^aNDT=not detected.

effects. It took about 10 min to reach gel polarized flux and the flux kept relatively constant over 1h. Again 2.0% solid content produced similar trend. Each pressure level produced 7 to 10 L/m²h reduced flux than that of 0.5% feed solid. The external driving force was not sufficient enough to overcome osmotic pressure exerted by increased solute concentration. The drop in permeate flux due to increased feed solid from 2.0% to 3.5% was 4 to 5 L/m²h. At 3.5% level, concentration polarization and fouling effects were suspected for the drop in permeate flux. The pressure, flow rate levels of 2.06 MPa and 20 L/min provided around 13 L/m²h permeate flux whereas 10 L/min flow rate and 2.06

MPa pressure produced around 8 L/m²h permeate flux.

In the long term fouling test, the gel polarized flux remained unaltered over 7h operation. However, 3.5% feed solids showed a slight decline in flux at the end of 3rd h of operation due probably to concentration polarization. The tight nature of the RO membrane was evident from its 100% solids rejection for all three feed solids contents.

Pilot scale UF performance

Changes in permeate flux of feeds with transmembrane pressure were monitored in the pilot scale UF operation (Fig. 4). For low feed solids, the increased permeate rate at higher temperatures might be due to the corresponding increase in diffusivity as suggested by Cheryan (1986). At 2.5% solids level, however, a reverse occurred presumably from increased gel polarization at higher temperatures. A similar observation was reported for ultrafiltration of sweet whey (Maubois, 1980). Protein gel layer prevented permeation rate of low molecular weight species. An increase in driving force resulted in a sharp increase in thickness of gel. The rise in gel thickness brought down permeate flux to previous levels by providing hydrodynamic resistance to flux. The solids rejection ranged from 20–35% upon applied pressures (0.6–1.4 MPa) for both feed solids levels of 1.5 and 2.5% at all temperatures.

The 15 L/min flow rate gave the highest initial flux of 55 L/m²h and 49 L/m²h at 0.6 MPa at 30°C and 50°C, respectively. At high temperatures, higher flow rate accumulated protein precipitates on the surface of the membrane thus impeding permeation of water. In general, permeate flux followed a logarithmic increase with higher increments in driving force. However, 20 L/min flow rate showed an upward trend only upto 1 MPa. Then it gave a constant permeate flux with subsequent increase in pressure indicating that the onset of gel polarized flux was at 1 MPa. No apparent effect of flow rate on solids rejection was observed under the pressures and temperatures studied.

High feed solids (1.5% and 2.5%) resulted in marked gel polarization at all pressure levels. An elevated flow rate of 15 L/min resulted in increased initial flux for 1.5% and 2.5% feed solids. This was because of rapid removal of gel influencing and fouling material from the surface of the membrane which helped maintained the permeation rate. However, an increase in pressure beyond 1 MPa for 2.5% solids did not produce increased permeate flux. At that high solids level a boosted driving force pushed more solids to the membrane surface and thereby retarded permeation. For 2.5% feed solids, the permeate flux dropped markedly after increasing the flow rate from 15 L/min to 20 L/min. At the higher flow rate, more solutes from the bulk were likely deposited on the membrane sur-

face, resulting in increased gel thickness which slowed permeation.

Pilot scale RO performance

The permeate flux of feeds at varying transmembrane pressures in the RO operation were much lower than those in UF as expected (Fig. 4). The permeate flux at 1.2 MPa pressure for 0.5, 1.5 and 3.0% feed solids content were 10, 5 and 0.9 L/m²h, respectively. The permeate flux increased logarithmically with increase in pressure for 1.5 and 3.0% feed solids tested. Since the osmotic pressure is much lower at a lower solute concentration, the effect of driving force would be higher at lower concentration resulting in an increased permeate flux. At 15 L/min flow rate, 3.0% feed solids produced an initial permeate flux increased by 0.7 L/m²h. This was

because of the removal of polarized solutes from 3% feed solids by the higher flow rate. The increased slope at higher pressures for 1.5% and 3.0% feed solids was probably due to an increased flow rate of 20 L/min which reduced any concentration polarization on the surface of the membrane. A combination of reduced polarization and higher driving force resulted in increased permeation rate.

When changes in the permeate flux of feeds were monitored with varying transmembrane pressure at 1.5% feed solids or 15 L/min flow rate (Fig. 5), at 30°C no notable changes in permeate flux were found by varying flow rates. However, at 40°C and 50°C the effect of flow rate was discernible along with marked increases in permeate flux. Such increased permeate flux at higher temperatures was probably due to reduction in solution viscos-

Fig. 3—Changes in permeate flux of feeds with time in a pilot scale RO system at 30°C feed temperature with varying feed solids, flow rates and transmembrane pressures.

Fig. 4—Changes in permeate flux of feeds with log mean transmembrane pressure in a pilot scale UF and RO systems at varying feed solids and temperatures, and flow rates.

Fig. 5—Changes in permeate flux of feeds with log mean transmembrane pressure in a pilot scale RO system at 1.5% feed solids with varying flow rates and feed temperatures, and at 15 L/min flow rate with varying feed solids and temperatures.

ity which improved mass transfer rate of solutes.

The feed at 50°C showed the highest slope with increased driving force because of less resistance from the low viscosity. The 1.5% feed solids showed signs of increased concentration polarization at 30°C and 40°C. All three temperatures produced polarized flux at 3.0% solids. The RO gave a complete 100% solids rejection at all temperatures, feed solids and flow rates.

CONCLUSIONS

THE OPTIMUM UF CONDITIONS FOR SEPARATING low molecular weight solutes were, 1.5% feed solids, 15 L/min flow rate, 50°C and 1 MPa log mean transmembrane pres-

sure. The RO gave a 100% solids rejection for all feed solids levels, temperatures and flow rates, retaining all dissolved solutes including flavor compounds. The optimum operating conditions for the RO were 40°C, 2.8 MPa log mean transmembrane pressure and a flow rate of 15 L/min. A combination of UF and RO was required for lobster flavor compounds recovery. The UF could be used to separate proteins and large m.w. nonflavor compounds from the extract. The resulting permeate enriched with low molecular weight flavor components could be subsequently concentrated by RO.

REFERENCES

Alvarez, V., Alvarez, S., Riera, F.A., and Alvarez, R. 1997. Permeate flux prediction in apple juice concentrate by

- reverse osmosis. *J. Membrane Sci.* 127: 25-34.
- AOAC. 1975. *Official Methods of Analysis*, 12th ed. Association of Official Analytical Chemists, Washington DC.
- Cheryan, M. 1986. Modeling of ultrafiltration processes. Ch. 4, in *Ultrafiltration Handbook*, p. 127-170. Technomic Publishing Company, Lancaster, PA.
- Hanisch, W. 1986. Cell harvesting. Ch. 3, in *Membrane Separations in Biotechnology*, W. C. McGregor (Ed.), p. 61-88. Marcel Dekker, New York.
- Hayashi, T., Yamaguchi, K., and Konosu, S. 1978. Studies on flavor components in boiled crabs-II. *Bull. Japan Soc. Sci. Fish.* 44: 1357-1362.
- Hayashi, T., Asakawa, A., and Konosu, S. 1979. Studies on flavor components in boiled crabs-III. *Bull. Japan Soc. Sci. Fish.* 45: 1325-1329.
- Hayashi, T., Yamaguchi, K., and Konosu, S. 1981. Sensory analysis of taste active compounds in the extract of boiled snow crab meat. *J. Food Sci.* 46: 479-493.
- Hedges, R.M. and Pepper, D. 1984. Reverse osmosis concentration and ultrafiltration separation in biochemical processes. *Filtration & Separation* 21: 112-114.
- Jayarajah, C.N. and Lee, C.M. 1987. Freeze-thaw concentration of shellfish extract and its flavor profile. Presented at the First Joint Conferences of Tropical & Subtropical Fisheries Technologists and Atlantic Fisheries Technologists of Americas, Orlando, FL, Nov. 22-26.
- Jayarajah, C.N. and Lee, C.M. 1988. Extraction and concentration of flavoring agent from shellfish waste. Presented at the 48th Annual Meeting of Inst. of Food Technologists, New Orleans, LA, June 19-22.
- Kessler, H.G. 1986. Energy aspects of food preconcentration, in *Concentration and Drying of Foods*, D. MacCarthy (Ed.), p. 147-163. Elsevier Applied Science Publishers, New York.
- Khym, J.X. 1975. Evaluation of microparticle chemically bonded reversed-phase packings in the high-pressure liquid chromatographic analysis of nucleosides and their bases. *Clin. Chem.* 21: 1245.
- Konosu, S. and Yamaguchi, K. 1986. Flavor of fish and shellfish with special reference to taste-active compounds, in *International Symposium on Engineered Seafood including Surimi*, R. Martin and R. Collette (Ed.), p. 545-575. National Fisheries Institute, Washington, DC.
- Konosu, S., Yamaguchi, K., and Hayashi, T. 1978. Studies on flavor compounds in boiled crab-I. *Bull. Japan Soc. Sci. Fish.* 44: 505-510.
- Koseoglu, S.S., Lawhon, J.T. and Lusos, E.W. 1990. Use of membranes in citrus juice processing. *Food Technol.* 44 (12): 90-97.
- Maubois, J.L. 1980. Ultrafiltration of whey. *J. Soc. Dairy Technol.* 33: 55.
- McCord, J.D., Trousdale, E., and Ryu, D.D.Y. 1984. An improved sample preparation procedure for the analysis of major organic compounds in grape must and wine by high performance liquid chromatography. *Am. J. Enol. Vitic.* 35: 28-29.
- McCutchan, J.W. 1977. Tubular reverse osmosis plant design, in *Reverse Osmosis and Synthetic Membranes*, S. Sourirajan (Ed.), p. 541-563. National Research Council Canada, Ottawa, Canada.
- McKeag, M. and Brown, P.R. 1978. Modification of high pressure liquid chromatographic nucleotide analysis. *J. Chromatogr.* 152: 253-254.
- Morh, C.M., Engelgau, D.E., Leeper, S.A., and Charbonneau, B.L. 1989. Meat and poultry processing in *Membrane Applications and Research in Food Processing*, p. 40-55. Noyes Data Corp. Park Ridge, NJ.
- Pepper, D., Banfield, D.L., and Hardwick, W.H. 1977. British reverse osmosis practice, in *Reverse Osmosis and Synthetic Membranes*, S. Sourirajan (Ed.), p. 405-415. National Research Council Canada, Ottawa, Canada.
- Pepper, D., Orchard, A.C.J., and Merry, A.J. 1989. Concentration of tomato juice and other fruit juices by reverse osmosis, in *Process Engineering in the Food Industry*, R.W. Field and J.A. Howell (Ed.), p. 281-290. Elsevier Applied Science, New York.
- Rudie, B.J., Torgimson, T.A., and Spatz, D.D. 1985. Reverse osmosis and ultrafiltration membrane compaction and fouling studies using ultrafiltration pretreatment, in *Reverse Osmosis and Ultrafiltration*, S. Sourirajan and T. Matsuura (Ed.), p. 403-414. American Chemical Society, Washington, DC.
- Yang, Y. 1998. Enzyme-assisted lobster flavor production from lobster process by-product. M.S. thesis, Univ. of Rhode Island, Kingston, RI.

Ms received 5/9/98; revised 8/4/98; accepted 8/31/98.

Contribution No. 3607 of the College of Resource Development, Univ. of Rhode Island, with support from the Rhode Island Agricultural Experiment Station. The work was supported in part by the National Fisheries Institute.