ChE *laboratory*

ULTRAFILTRATION OF DAIRY PRODUCTS AS A CHE LABORATORY EXPERIMENT

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Membrane process experiments involving ultrafiltration (UF) of dairy products have been developed and classroom tested. These experiments provide an effective method for integrating membrane separation processes into the curriculum while at the same time offering learning experiences of broad-based interest to chemical engineering students through familiar applications such as cheese production and environmental cleanup. UF experiments can be used in conjunction with the required unit operations laboratory and mass transfer/separation processes courses, or as a component of elective courses in membrane process technology or biochemical engineering.

Although there has been considerable discussion about revamping the chemical engineering curriculum, topics covered in required courses remain fairly uniform throughout academe. Required separations or mass transfer courses focus heavily on traditional equilibrium-staged operations, including distillation, absorption, and extraction.[1] These unit operations are very important in the chemical and petroleum industries, but chemical engineering students also need to study separation processes that have become increasingly important in other areas, such as food processing, biotechnology, and environmental engineering. A 1995 undergraduate survey of chemical engineering programs indicates that there has been little change in topics covered in mass transfer courses other than slight increases in time spent on adsorption (2.5%) and membranes (2.6%).[1] The importance of including instruction on membrane process technology in the chemical engineering curriculum has been addressed in recent articles.[2-8]

Membrane processes are mass transfer unit operations used for liquid- or gas-stream separations. The family of processes covers a number of operations, including reverse osmosis (RO), nanofiltration, ultrafiltration, microfiltration (MF), dialysis, electrodialysis, gas permeation (GP), and pervaporation (PV).[9] These processes are not really new, but are often unfamiliar to the typical chemical engineer due to lack of exposure during his or her education.

RO is in its fourth decade, having been developed in the early 1960s for industrial operations such as production of potable water from seawater. UF has been used on a com-

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mercial scale since the early 1970s, with dairy processing representing one of the largest applications of membrane technology in the world.[10] The earliest commercial application of UF was the concentration of proteins from whey, a milk by-product generated during the traditional coagulation process for cheese production.[10] Similarly, UF is used to remove lactose and milk proteins from whey wastewater streams prior to disposal, *i.e.,* for environmental treatment. In newer processes for manufacture of soft cheeses, yogurt, ice cream, and other dairy products, UF is used to preconcentrate whole or skim milk prior to entering the production chain.[10-15]

This paper discusses laboratory experiments on ultrafiltration of dairy products that may be used to increase coverage of membrane processes in the chemical engineering curriculum and gives typical results from experiments run by students at Clemson University (CU) and Manhattan College (MC).

At CU, a spiral-wound UF system manufactured by Koch Membrane Systems, Inc., has been used to concentrate various dairy products in the experimental component of an elective course on membrane separation processes and in the senior unit operations laboratory.[6] The CU experiments focus on application of the UF process for environmental cleanup of rinse waters from dairy operations, *e.g.,* a whole-milk pasteurization system, an ice-cream freezing unit, and a chocolate-milk production system.[16] At MC, a Millipore Model TCF10 thin-channel UF system (formerly manufactured by Amicon) is used in the unit operations laboratory course for an experiment on milk concentration.

Based on the authors' experiences, the milk experiments have an intrinsic appeal to all of the chemical engineering students and instructors, not just the biochemical engineers and membrane specialists. Interest is heightened by employing a typical local application; for example, UF is one of the steps used in cheese production at Kraft Foods Inc. in Tarrytown, New York (an industrial site near MC).

The UF experiments discussed in this paper are but one component of an array of membrane process experiments that can be employed to enhance the chemical engineering curriculum. RO, GP, MF, and PV experiments are also included in the courses at CU and MC. [5,6,17-19] Several membrane process experiments will be integrated into the required mass transfer and separation processes courses at Rowan University. In addition, a hand-held RO system is used in the freshman engineering course at Rowan University.[20,21]

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The authors have previously outlined UF experiments for separation and concentration of protein and enzyme mixtures, experiments that are used to supplement an elective course in biochemical engineering at MC.[5,22,23] Other professors have reported on UF experiments for concentration and separation of polyethylene glycol and of protein mixtures, adapted to the unit operations laboratory and a biotechnology laboratory, respectively.[24,25]

SEPARATION PRINCIPLES

Ultrafiltration is one of a group of membrane filtration processes that depends on pressure as the driving force for separation. The average pore size in UF membranes varies from 300 to 300,000 Daltons (ranges vary depending on source), which will retain macromolecules such as milk proteins while passing inorganic salts and small organic molecules such as lactose ($C_{12}H_{22}O_{11}$) through the membrane. The key proteins in milk are caseins, which form large micelles (10 to 300 nm), α-lactalbumin and β-lactoglobulin. The α-lactalbumin has a molecular weight of 18,300, while β-lactoglobulin exists as a dimer with a molecular weight of 36,000; therefore, a membrane with a molecular weight cutoff (MWCO) of 10,000 is needed to insure complete retention of milk proteins.[10] A polysulfone membrane with a MWCO of 20,000 is generally used for milk ultrafiltration, with an elevated operating temperature (52°-54°) and transmembrane pressures of 170-310 kPa.[11] The membrane originally used for this application was cellulose acetate.[14]

The molecular weight cutoff of a membrane may be defined as the molecular weight that is 90% rejected by the membrane (some manufacturers use a different percent, *e.g.,* 95%), which indicates that a 10,000 MWCO membrane will reject 90% of solutes having a MW more than 10,000. Rejection is actually a function of the size, shape, and surface-binding characteristics of the hydrated molecule, as well as the pore-size distribution of the membrane; therefore, molecular weight cutoff values can be used only as a rough guide for membrane selection.

System performance is usually defined in terms of permeate flux, J, with dimensions of (volume/area-time), *i.e.,* typical units are (L/m^2-h). Flux can be determined by measuring each incremental volume of permeate, ΔV, collected in time

period, Δt, and dividing by the effective surface area of the membrane or the transfer area.

$$J = \frac{\Delta V / \Delta t}{\text{transfer area}} \quad (1)$$

Effective surface area for the TCF10 thin-channel system is less than the total membrane surface area because portions of the membrane are blocked by ridges along edges of the spiral channel. In the spiral-wound membrane module in the Koch system, the total membrane area is available for transfer.

Theoretically, flux of a pure solvent (J_{solv}) through a porous membrane is directly proportional to the pressure gradient across the membrane and inversely proportional to the membrane thickness, t_m, as follows:[9]

$$J_{solv} = \frac{K_{solv}}{t_m}(\Delta P - \Delta\pi) \approx \frac{\Delta P}{R_m} \quad (2)$$

where ΔP is the pressure drop across the membrane (transmembrane pressure) and $\Delta\pi$ is the difference in osmotic pressure across the membrane. Osmotic pressure is relatively low for macromolecular solutions (the type separated by UF processes), so the $\Delta\pi$ term can be generally neglected in Eq. 2.[9] Osmotic pressures for whey proteins become more significant at higher concentrations.[10] The permeability constant, K_{solv}, accounts for factors such as membrane porosity, pore-size distribution, and viscosity of the solvent at a given temperature. The membrane thickness, t_m, is not readily measured for an asymmetric membrane; however, the resistance to flow through the membrane, R_m, may be used in place of the quantity (t_m/K_{solv}). The value of R_m can be determined at a given temperature by running water-flux experiments at various operating pressures.

Concentration polarization, gel formation, and fouling are important factors that need to be considered in UF separations. As shown schematically in Figure 1, a concentration gradient or boundary layer of increased solute concentration forms near the membrane surface during UF. This gradient, which is called concentration polarization, results from counteracting effects of convective flow of solute towards the membrane and diffusion of solute back toward the bulk fluid. Concentration polarization is regarded as a reversible boundary-layer phenomenon that causes a rapid initial drop in flux to a steady-state value, whereas fouling is categorized as an irreversible phenomenon that leads to a long-term flux decline.[10] Concentration polarization may occur with or without gelling (Figure 1 depicts gel formation), and gel formation may be reversible or irreversible. If the gel is difficult to remove from the membrane (irreversible), the membrane is said to be fouled.[9-10]

In UF processes, many of the solutions being filtered form gels, cakes, or slimes at the wall because convective transport toward the membrane is relatively high compared to diffusivities of macromolecules.[9] Membrane fouling is reported to be common during milk ultrafiltration.[10] A gel layer was formed in the milk experiments reported in this paper, verified visually at the end of the TCF10 experiments as a pale yellow spiral of solids deposited on the white membrane surface. Gel formation was deemed to be reversible (non-fouling) since a long-term decline in solvent flux was not observed and the gel came off the membrane quite easily when rinsed with distilled water.

As mentioned above, concentration polarization with gel formation is observed for many UF separations. The gel layer frequently has far more resistance to flow than the membrane and thus controls solvent flux.[9] Equation (2) can be modified to include the effect of the gel layer by adding a term for resistance to flow through the gel,

$$J = \frac{\Delta P}{\left(t_m / K_{solv} + t_g / K_g\right)} = \frac{\Delta P}{\left(R_m + R_g\right)} \quad (3)$$

In Equation (3), t_g is the gel thickness and K_g is the gel permeability constant, which are generally not known, and R_g is the resistance to flow through the gel, which can be measured experimentally. The value of R_g varies with pressure, bulk concentration, and cross-flow velocity at lower transmembrane pressures, but tends to become pressure independent at higher transmembrane pressures.[10]

Another approach to solving this mass-transfer problem

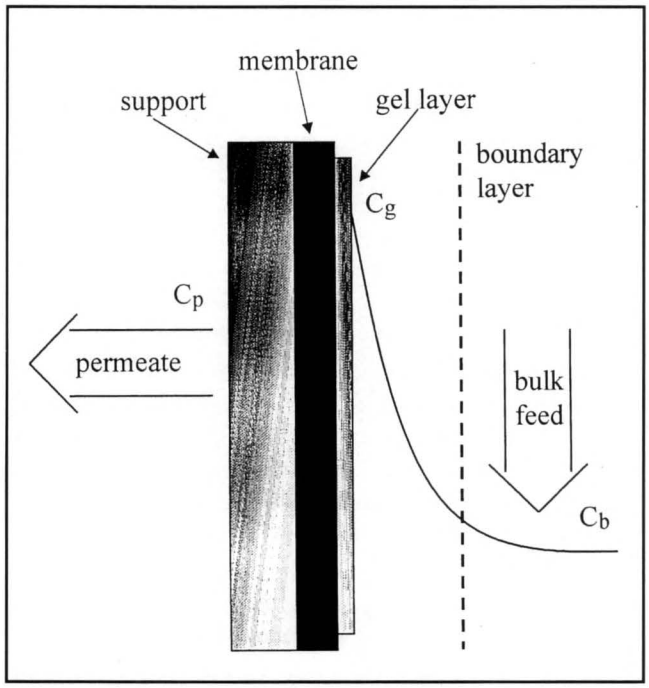

Figure 1. *Schematic illustrating flux of permeate through an asymmetric ultrafiltration membrane (membrane plus support) in the presence of concentration polarization with gel layer formation. Symbols C_b, C_g, and C_p represent bulk, gel, and permeate concentrations, respectively.*

starts with the equation of continuity and assumes a stagnant film of thickness δ. The derivation, as detailed by Zeman and Zydney,[10] results in

$$J_{solv} = \frac{D}{\delta} \ell n \left(\frac{C_w - C_p}{C_b - C_p} \right) \quad (4)$$

where k is the mass-transfer coefficient, C_w is the solute concentration at the wall, C_p is the solute concentration in the permeate, C_b is the bulk concentration, and D is the solute diffusivity.

In cases where the gel layer controls mass transfer and C_p=0, the wall concentration in Eq. (4) can be replaced by the gel concentration, C_g, which results in a simplified equation referred to as the gel-polarization model[9-10]

$$J_{solv} = k \ell n \frac{C_g}{C_b} \qquad \text{where } k = \frac{D}{\delta} \quad (5)$$

The mass-transfer coefficient, k, increases as cross-flow velocity increases; the gel concentration, C_g, however, is generally regarded as a constant even though different values are reported depending on the type of equipment used.[10] Also, diffusivity changes with temperature and viscosity. Thus, at constant temperature and cross-flow velocity, experimental data can be measured for flux as a function of bulk concentration, then graphed to determine values for k and C_g. This method can be used to estimate an experimental value for k from the data collected in milk experiments run with 10,000 to 18,000 MWCO membranes since $C_p \approx 0$ with respect to milk proteins. When the permeate is not a pure solvent (C_p>0), which occurs with a 30,000 MWCO membrane, equations for solvent flux must account for C_p and are therefore somewhat more complicated.[9]

The mass-transfer coefficient can also be calculated from empirical equations.[9,10] Fully developed turbulent flow in UF devices appears to occur at Reynolds numbers around 2,000. In the TCF10 system, fluid flows through a spiral channel of width, w, height, 2h, and length, L; thus, the Reynolds Number (Re) is calculated using the equivalent diameter, d_{eq}, of the channel:

$$d_{eq} = 4 \frac{\text{cross - sectional area}}{\text{wetted perimeter}} = 4 \frac{2\,hw}{2\,w + 4\,h} \quad (6)$$

Similar equations are used to determine the equivalent diameter in the spiral-wound Koch system.

In the equations that follow, u_b is the average linear velocity through the channel, D is diffusivity, μ is viscosity, and ρ is density. Physical properties are based on the fluid on the retentate/feed side of the membrane, taken at the average concentration from the beginning to the end of each run. Viscosities of milk solutions as a function of concentration and temperature are available in the literature.[13] For turbulent flow through a channel, the following equation can be used:[9]

$$k = 0.023 \frac{D}{d_{eq}} Re^{0.83} Sc^{1/3} \quad (7)$$

$$\text{where} \quad Re = \frac{d_{eq} u_b \rho}{\mu} \quad \text{and} \quad Sc = \frac{\mu}{D\rho}$$

where Sc is the Schmidt number, Re is the Reynolds number, and the other terms are previously defined. Additional empirical correlations are given in the literature to determine mass transfer coefficients for UF systems under both turbulent and laminar flow conditions.[9,10]

Experimental Methods

Manhattan College System

A Millipore TCF10 thin-channel ultrafiltration system (formerly manufactured by Amicon) was used at Manhattan College for UF experiments with skim milk solutions. Photographs of this system and schematics of the thin channel are given in the literature.[10,12,22] The TCF10 is a 0.6-L bench-scale system that is designed to use 0.090-m diameter flat membranes. Pressure is supplied to the top of the feed chamber from a nitrogen cylinder. A peristaltic pump that is provided with the UF system generates high-velocity flow across the membrane by pumping a feed solution parallel to the membrane surface through a thin spiral channel. Cross flow is designed to minimize concentration polarization and gel formation and subsequently to increase permeate flux through the membrane. According to the manufacturer, the spiral channel has dimensions of 0.0095 m in width (w), 0.00038 m in height (2h), and 0.414 m in length (L). The effective membrane surface area or transfer area is reported to be 4.0×10^{-3} m^2. Experiments were run at room temperature (22-23°C).

The TCF10 unit was operated in a batch mode where the retentate leaving the spiral channel was recycled back to the feed chamber, while the permeate was separated from the feed solution and collected in a separate container. This mode of operation causes the bulk concentration, C_b, to increase with time. All experiments used 0.4 L of feed solution and limited permeate collection to about 10% of the initial feed solution in order to limit changes in bulk concentration. Skim milk was prepared by dissolving powdered milk in distilled water following instructions on the package (10.54% solids). Different milk-water feed solutions were prepared in ratios varying from 1:0 to 1:8 by diluting skim milk with distilled water. Although higher milk concentrations would occur in the industrial process, diluted solutions can be used to demonstrate concentration effects in a reasonable period of time. The most time-consuming step in the milk experiments occurs between runs when the students

have to open the system, clean the membrane, and reassemble the unit.

Millipore markets four membranes that can be used for the milk experiments; with water fluxes in decreasing order, these are PM30>YM30>PM10>YM10. The symbols are PM for polysulfone membrane, YM for cellulose acetate membrane, 30 for 30,000 MWCO, and 10 for 10,000 MWCO. The YM membrane is treated so as to be hydrophilic with low protein binding properties, while the PM membrane has the advantage of high throughput. These membranes have an asymmetric or anisotropic structure, *i.e.,* a very thin polymeric skin with an extremely fine, controlled, pore structure supported by a much thicker (and stronger), highly porous substrate. A YM30 membrane was selected for the experiments run at Manhattan College in 1996 and 1997 because this membrane permits a number of runs to be executed at different concentrations, cross-flow velocities, and transmembrane pressures during a single laboratory period. The PM10 and YM10 membranes were also used in 1998 for comparison with the YM30 membrane.

CLEMSON UNIVERSITY SYSTEM

A Proto-Sep IV portable spiral-wound UF system manufactured by Koch Membrane Systems was used at Clemson University for UF experiments. The Koch bench-scale system includes an HFM 180 (polyvinylidene fluoride) Abcor spiral-wound UF membrane with an 18,000 MWCO and a nominal surface area of 0.28 m^2. Experiments were run in a batch mode with the retentate returned to a 72.5-L stainless steel feed tank. Transmembrane pressure and tangential flow were both supplied by a Wilden MI-Champ air-operated, double-diaphragm pump. Dairy solutions (whole milk, milk-water solutions, and dairy wastewater streams) were analyzed for total solids content, pH, chemical oxygen demand (COD), total carbohydrates, fat content, and protein content as described by Steinbeck.[16] Experiments were run at 30 to 50°C.

The Koch Proto-SEP IV system is actually better suited for qualitative feasibility studies than for obtaining quantitative data for scale-up. The standard system contains a single-pressure gauge and flow-control valve located in the concentrate line. The air-actuated diaphragm feed pump is rather noisy, and it delivers a pulsatile flow rate that is dependent on the air pressure. The concentrate pressure and flow rate cannot be set in a completely independent fashion, and the concentrate pressure is not the same as the average transmembrane pressure that is more commonly used as a characteristic operating variable. Despite these limitations, the Koch unit can be used to demonstrate basic UF operating principles and certain relationships with a variety of feed streams. For example, runs can be made to determine the effect of pressure on flux at constant composition by returning the permeate and concentrate to the feed tank. With the perme-

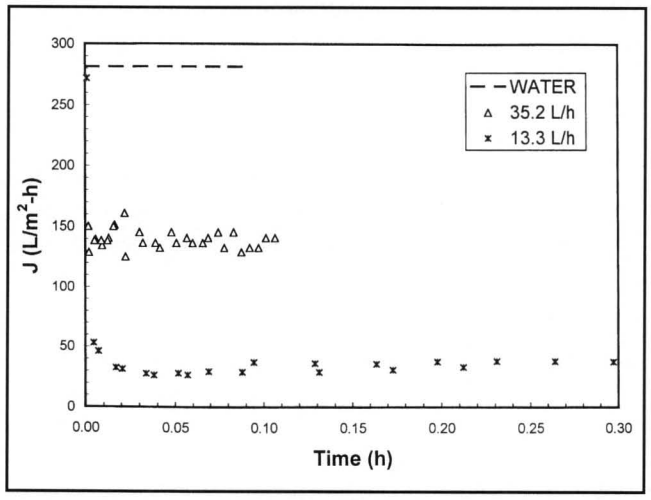

Figure 2. *Effect of volumetric flow rate through the thin channel of a TCF10 system on flux. Data taken with a 1:8 (skim milk to water ratio) solution at 207 kPa transmembrane pressure and room temperature, using a YM30 membrane.*

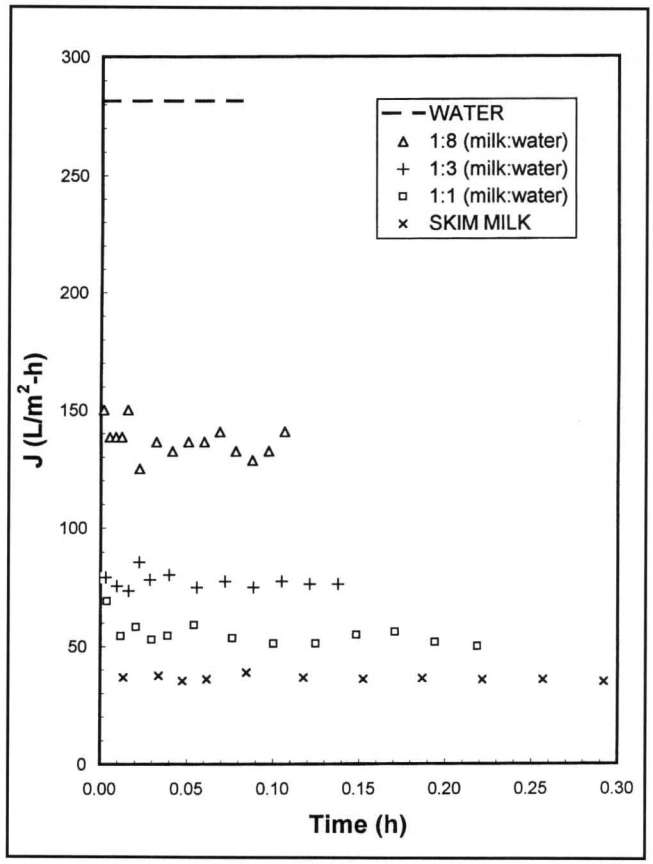

Figure 3. *Effect of solids concentration on flux in a TCF10 system with a YM30 membrane. Data recorded at 207 kPa, 35.2 L/h, and room temperature.*

ate diverted to a different collector, the effect of feed concentration on flux can be observed. The feed tank can be heated or cooled to demonstrate the effects of temperature.

TYPICAL EXPERIMENTAL RESULTS

The first step that needs to be performed in UF, MF, or RO membrane experiments is measurement of the flux versus pressure behavior of the membrane when filtering pure water. This measurement may be used to determine whether the equipment is set up properly by comparing water fluxes with expected values and to calculate the membrane resistance (R_m) from Eq. (2). Water fluxes should also be calculated between runs (after removing, rinsing, and reinstalling the membrane) in order to make sure that the membrane is clean enough for reuse. If water flux is lower than expected, the asymmetric membrane may be upside down or it may need additional cleaning. If water flux is higher than expected, a tear or other defect in the membrane is possible. The slope of the water flux/pressure data for the YM30 membrane in the TCF10 system is about 1.4 L/(m^2-h-kPa).

The effect of cross-flow velocity on flux in the TCF10 system is graphed in Figure 2. As shown, flux of pure water is relatively high and constant with time. Prior experiments demonstrate that the characteristics of the flux curves are quite different for the YM30 and PM30 membranes.[23] Under the same operating conditions, the flux of the polysulfone membrane (PM30) drops gradually throughout the experiment, while the flux of the cellulostic membrane (YM30) drops immediately to a steady-state value and remains relatively constant thereafter.[23] This observation can be attributed to the fact that the YM30 membrane is treated to minimize protein adsorption and thus minimize fouling. At the lower cross-flow velocity (Figure 2), a few high-flux points are observed in the first few seconds of the run as the flux drops due to concentration polarization and gel formation, but the flux remains stable and constant thereafter, indicating that the gel, once formed, controls flux. At the higher cross-flow velocity (Figure 2), the transition period is very rapid, so that the flux appears to drop immediately to the steady-state value. As expected, a higher cross-flow velocity significantly increases solvent flux by improving mass-transfer conditions at the membrane surface. Since R_m was previously calculated from the water-flux data, values of R_g for both the high and low cross-flow cases can be determined using Eq. (3).

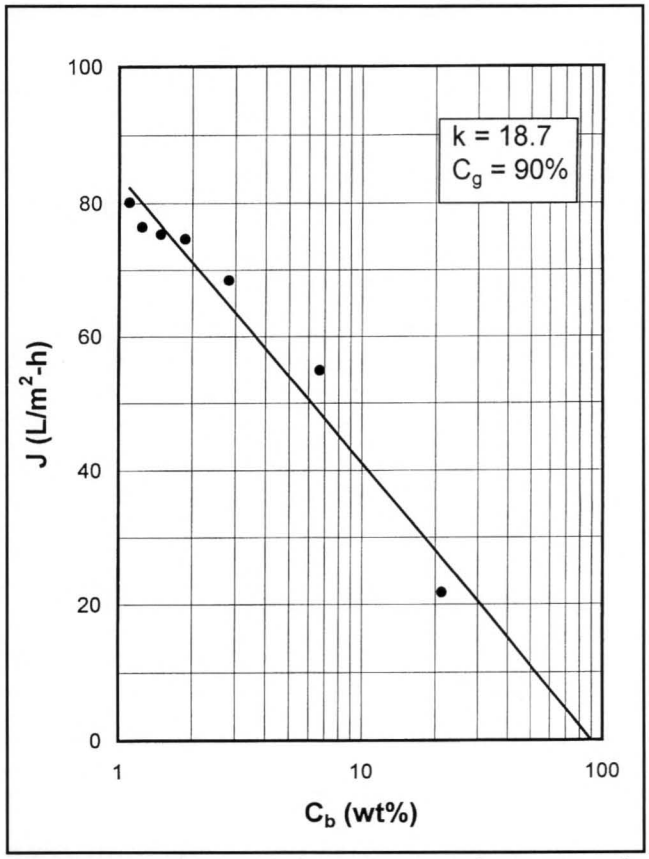

Figure 4. *Experimental determination of mass transfer coefficient, k, and gel concentration, C_g, using a Koch spiral-wound UF system with an Abcor 18,000 MWCO membrane. Data recorded at 50°C, 1400 L/h, and 67 kPa transmembrane pressure by Steinbeck.*[16]

The students were also asked to determine the effect of milk concentration on flux. Typical data are graphed in Figure 3 for a YM30 membrane in the Millipore system. As in the previous figure, milk fluxes (at high cross-flow velocity) drop immediately from pure-water values to steady-state values. Comparing trials with milk-water ratios increasing from 1:8 up to 1:0 (undiluted skim milk), it is evident that flux decreases as concentration increases. The solvent fluxes remain relatively constant at the steady-state values in Figures 2 and 3; specifically, there is no evidence of irreversible fouling and its associated long-term flux decline. Since a 30,000 MWCO membrane was used for the data in Figure 3, the milk concentration in the permeate is greater than zero, which must be taken into account in determining experimental values of k and C_g.

Using data from the Abcor 18,000 MWCO membrane,[16] a meaningful graph of Eq. (5) can be generated as shown in Figure 4, although there is room for speculation on the location of the "best" straight line and the resulting slope and intercept, or k and C_g values. Based on reports that the maximum concentration levels are seven-fold for skim milk and five-fold for whole milk (or about 65% solids),[11] the value of C_g determined in Figure 4 appears to be reasonable. As evidenced in Figure 4, additional data points are needed at higher bulk concentrations (closer to C_g) if more accurate experimental values for k and C_g are desired.

After graphing and analyzing the data from the milk UF experiments, the students were asked to calculate a mass transfer coefficient using Eq. (7) or other appropriate em-

pirical equations. Finally, they were expected to compare the calculated coefficient with the experimental coefficient obtained from the graphical analysis. For a more theoretical approach, the students could be asked to derive Eq. (4) from the equation of continuity. Based on three years of MC student data, milk-flux data generally followed the expected trends, but agreement between experimental and calculated values for the mass-transfer coefficient was highly variable. This variability results from the fact that high milk concentrations were not run, which in turn leads to errors in the experimental values of k and from the approximate nature of the empirical equation. Higher concentrations would require more time for individual runs, but should improve accuracy. It is questionable whether the extra time per experiment is justified to tie down a single number, because UF principles can be demonstrated qualitatively using lower concentrations, thus allowing time to study other operating conditions.

CONCLUSIONS

Experiments involving ultrafiltration of dairy products have been developed at Clemson University and Manhattan College and tested in lecture and laboratory courses. These experiments appeal to the students (like the "got milk?" advertisement) and are an effective method of introducing membrane separation processes into the chemical engineering curriculum. Whether UF experiments are run in an open-ended or structured format, better results are generally obtained when a "resident expert" who is familiar with membrane processes is available to the students for consultation. Based on the authors' experiences, learning is enhanced if the experiments are used to enrich lecture courses where the instructor can close the feedback loop with classroom discussion. On the other hand, respectable results were obtained by students in the required senior laboratory course when neither the instructor nor the teaching assistant claimed any special expertise in membrane separations. The small Millipore TCF10 system seems to be more user friendly than the larger Koch Proto-Sep system; however, both systems have been used effectively to demonstrate membrane separation principles.

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