Experimental Strategy for the Optimal Design of Ultrafiltration Units Used to Recover Some Food Biopolymers

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This PhD thesis research project is aimed at setting up a batch or total recycle experimental procedure at both bench-top and pilot scales to identify the most appropriate mathematical model to simulate accurately the recovery of selected food biopolymers via tubular or hollow-fibre ultrafiltration modules and provide a basis for their optimal design in an industrial scale.

Strategia sperimentale per la progettazione ottimale di unità di ultrafiltrazione per il recupero di biopolimeri di interesse alimentare

Questo progetto di tesi di dottorato mira a mettere a punto un procedimento sperimentale, in batch o a riciclo totale, prima in impianto da banco e poi in impianto pilota, atto ad individuare il modello matematico in grado di simulare il processo di recupero di selezionati biopolimeri di interesse alimentare mediante moduli a membrana di ultrafiltrazione tubolari o a fibre cave, consentendone il dimensionamento ottimale in scala industriale.

1. State-of-the-Art

Since the early 60s membrane separation processes have presented quite a limited diffusion and have just more recently begun to be recognised as efficient, economical and reliable separation processes. Depending on membrane pore size, feed cross flow velocity, transmembrane pressure difference applied (ΔP) and permeation flux, membrane filtration processes can be classified as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), the configuration of their modules being *tubular* (T), *hollow-fibre* (HF), *spiral-wound* (SW) or *plate-and-frame* (PF) (Cheryan, 1998; Daufin *et al*., 1998; Ho and Sirkar, 1992).
Table 1 lists the main applications of UF membrane processing in the food and beverage sector together with specific membrane type and configuration, range of solvent permeation flux (J_{Wv}) and solute true rejection (r_t) .
A great number of problems limit UF membrane sale growth like membrane resistance to solvent, fouling problems, design considerations for the incomplete comprehension of mass transfer mechanisms in membrane systems, cleanability, investment and membrane replacement costs, and competing technologies. Formation of a gel-polarised layer onto membrane surface, as well blocking of membrane surface pores or fouling of support materials, results in a more or less pronounced permeation flux decay. Such a decay is still difficult to quantify at the design stage by resorting to any of the numerous transport models available in the literature, that is *non porous or homogeneous membrane models* (*i.e.* solution-diffusion, extended solution-diffusion, and solution-diffusion-imperfection models), *pore-based models* (preferential sorption-capillary flow, finely porous, and surface force-pore flow models), and *irreversible thermodynamics phenomenological models* (such as Kedem-Katchalsky and Spiegler-Kedem models).

Table 1 Main applications of UF membrane processes in the food sector.

Application	**Example**	**Membrane module characteristics**				
		Type	**Material**	**Cut-off** (kDa)	**J_{Wv}** ($dm^3\ m^{-2}\ h^{-1}$)	**r_t** (%)
Fractionation	Milk or whey (protein from lactose and minerals)	PF, SW, T	C, PS, PES	10-100	5-100	70-97
	Oil fractions from oil-in-water emulsions	SW	TFC	8	10-13	70-90
Clarification	Alcoholic juices and beverages	HF, SW, T	PAN, PS, PES	10-100	5-100	70-97
	Removal of colloids, pigments, low MW compounds	HF, T	PAN, PES	10-50	5-50	70-83
Concentration	Albumin and proteins	PF, SW, T	C, PS, PES	10-100	5-30	70-83
	Polysaccharides (karragineen, xanthan)	HF	PS	500	5-10	-

C: Ceramic; PS: Polysulfone; PES: Polyethersulfone; PAN: Polyacrylonitrile; TFC: Thin Film Composite.

For instance, it is difficult to express the real relationship between J_{Wv} and ΔP and, in particular, the fact that in the UF range J_{Wv} is controlled by pressure for $\Delta P<6$ Pa and by mass transfer for $\Delta P>6\text{-}10$ Pa, this effect being counteracted by increasing feed flow rate (Q_F) or process temperature (T), as well as decreasing feed solute concentration (C_B). Moreover, while in the RO range J_{Wv}-logC_B plot linearly decreases with C_B increasing and vanishes for C_B as such that the feed osmotic pressure equals feed input pressure, in the UF range such a plot does not tend to zero, but it may reach a minimum value (definitively different from zero), which remains practically constant or increases up to a maximum value before finally decreasing as solute concentration increases (Pritchard *et al.*, 1995).

Almost all the biopolymers recovered via UF processes exhibit a non-Newtonian behaviour of the pseudoplastic type. This is generally described via the Ostwäld-de Waele model, that allows a quite accurate reconstruction of the liquid apparent viscosity (η_a) in the intermediate shear rate region only, but at very low shear rates overestimates η_a, thus leading to mass transfer coefficients extremely underestimated. Thus, this PhD thesis project will be directed to select which mathematical model with the minimum number of statistically independent parameters allows the best reconstruction of UF membrane process performances buy resorting to well known experimental design techniques to minimise the experimental trials needed.

2. PhD Thesis Objectives and Milestones

Within the overall objective mentioned above this PhD thesis project can be subdivided into the following activities according to the Gantt diagram given in Table 2:

A1) **Determination of the physical properties of a few selected biopolymers** (sodium alginates and pectinate, and whey proteins) in aqueous solutions to model their density, osmotic pressure (A1.1) and rheological behaviour (A1.3) as functions of C_B.

A2) **Assessment and modelling of the UF processes in a bench-top plant scale** to identify the mathematical model capable of reconstructing their performances as functions of the main operating variables (ΔP; T; Q_F, C_B) and membrane constitution, porosity and configuration, that is a C-T (A2.1) and a PES HF (A2.2) membrane module. An experimental strategy to limit polarisation layer growth will also be established to optimise both UF module performance.

A3) **Scaling-up of the UF processes** in the pilot plant scale to validate the prediction capability of mathematical models identified during activity A2 when using commercial C-T (A3.1) and PES-HF (A3.2) UF membrane modules.

A4) **Optimisation of the UF processes examined** so as to assess their optimal operating conditions (A4.1) and set up a generalised experimental procedure (A4.2) to determine the main engineering parameters necessary to design the UF unit in an industrial scale.

A5) **Writing and Editing** of the PhD thesis, scientific papers and oral and/or poster communications.

Table 2 Gantt diagram for this PhD thesis project.

Activity	Months	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
A1)	***Biopolymer Physical Properties***																								
	1) Density, Osmotic Pressure																								
	2) Rheological Behaviour																								
A2)	***UF Process Modelling***																								
	1) Ceramic Tubolar Module																								
	2) Hollow-fibre Module																								
A3)	***Scaling-up of UF processes***																								
	1) Ceramic Tubolar Module																								
	2) Hollow-fibre Module																								
A4)	***UF Process Optimisation***																								
	1) Optimal UF Processes																								
	2) Generalised Exp.l Procedure																								
A5)	***Thesis and Paper Preparation***																								

3. Selected References

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