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Georges Daufin[a]; Jean-François Radenac[a]; Geneviève Gésan[a]; François-Louis Kerhervé[a]; Olivier Le Berre[a]; Françoise Michel[a]; Uzi Merin[b]
[a] LABORATOIRE DE RECHERCHES DE TECHNOLOGIE LAITIÉRE, INSTITUT NATIONAL DE LA RECHERCHE AGRONOMIQUE, RENNES, CÉDEX, FRANCE [b] THE VOLCANI CENTER, AGRICULTURAL RESEARCH ORGANISATION, BET DAGAN, ISRAEL

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A Novel Rig Design for Ultra- and Microfiltration Experiments

GEORGES DAUFIN, JEAN-FRANÇOIS RADENAC, GENEVIÈVE GÉSAN, FRANÇOIS-LOUIS KERHERVÉ, OLIVIER LE BERRE, and FRANÇOISE MICHEL
INSTITUT NATIONAL DE LA RECHERCHE AGRONOMIQUE
LABORATOIRE DE RECHERCHES DE TECHNOLOGIE LAITIÈRE
65, RUE DE SAINT BRIEUC, 35042 RENNES CÉDEX, FRANCE

UZI MERIN
AGRICULTURAL RESEARCH ORGANISATION
THE VOLCANI CENTER
P.O. BOX 6, BET DAGAN, ISRAEL

ABSTRACT

The versatile rig for crossflow ultrafiltration and microfiltration experiments described in this paper can be operated at various concentrations of the feed stream by using a feed-and-bleed mode, either at controlled permeation flux or at controlled transmembrane pressure. Transmembrane pressure can be set as a static counterpressure through a bleed valve, or as a dynamic counterpressure achieved by circulating the permeate cocurrent to the retentate, to maintain an equal transmembrane pressure profile along the filtration path. The rig is equipped with extra independent controls (retentate and permeate temperature, retentate tangential flow velocity, retentate pressure) to enable to master filtration procedures by setting variables to the desired values through any operational pathway. It allows real time data monitoring and storing by a computer through a multichannel analyzer.

INTRODUCTION

The interest in membrane filtration has intensified since its appearance in the early 1960s. Most research in this field was carried out on commercial test rigs available from membrane manufacturing companies. They

range from small test cells to several square meters of membrane area. Some test rigs were built by researchers using different membranes and modules. In order to obtain reliable information on membrane processing, as many variables as possible should be maintained constant (1, 2). Therefore, different operating variables, such as pressure or filtration flux, flow velocity, temperature, etc., should be controllable, and the acquisition of data should be accurate and reproducible. These conditions required a sophisticated filtration rig such as that planned, built, and operated at the Laboratoire de Recherches de Technologie Laitière, Institut National de la Recherche Agronomique, Rennes, for separation, clarification, and fractionation as well as fouling and cleaning experiments using whey, milk, and its products.

DESCRIPTION OF THE RIG AND ITS CHARACTERISTICS

Field of Utilization

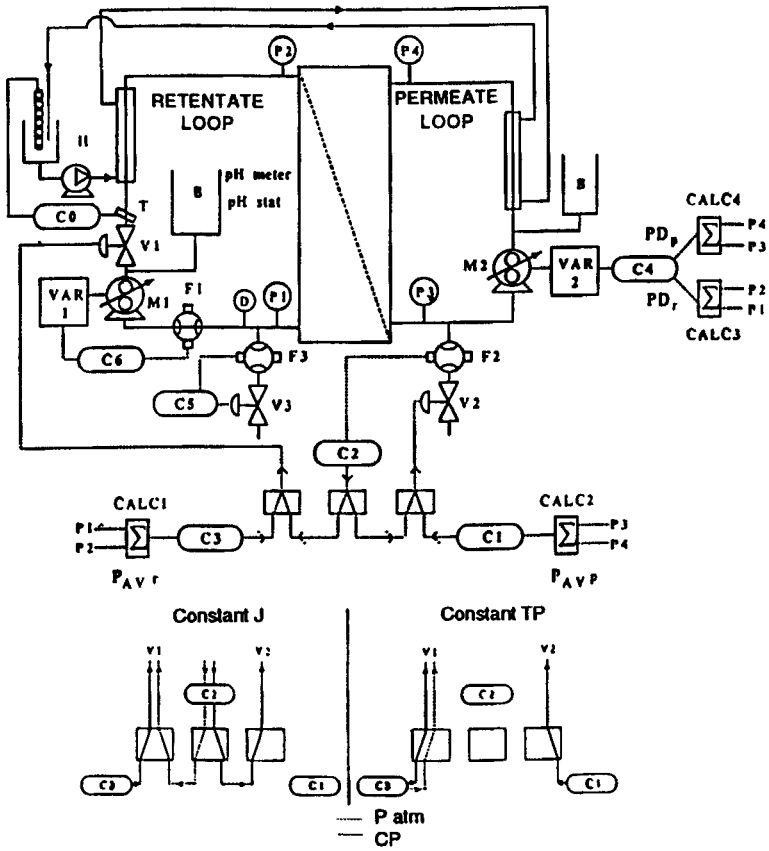
The objective of the rig was to perform ultrafiltration and microfiltration experiments using various modules of organic and inorganic membranes. It was designed to enable constant concentration experiments through a feed-and-bleed mode and to operate either at constant transmembrane pressure (difference across the membrane) or at constant flux by switching to different electrical circuits. The differences in operating regimes between ultrafiltration and microfiltration require control of the permeate pressure. Permeate could be withdrawn either at atmospheric or at any given counterpressure, either static or dynamic (3, 4) (see explanation in text). The final cleaning cycle is also monitored, and its operating variables are recorded to assess its efficiency (5, 6).

Rig Design

The rig is modularly designed by using clamps which enable versatility in mounting different components. It can use membrane modules like plate-and-frame for organic membranes, up to 0.1 m^2; and tubular for inorganic membranes of 2–15 mm i.d., 0.20 to 1.20 m long, up to 5×10^{-2} m^2. Temperature is thermostatically controlled by heat exchangers through Pt-100 ohm sensors.

The rig is equipped with several programmable controllers. Each controller receives a signal from various sensing units such as pressure, flow rate, temperature, etc., and controls its operation by maintaining its preset value.

Figure 1 shows the basic rig setup. It consists of a 120–1,200 $L \cdot h^{-1}$, 0–10 bar feed and recirculating screw pump, M1 (dead volume 450 mL,



	- toggle switch
B	- feed tank
C0-6	- regulator
CALC1-4	- controller
CP	- permeate under counter pressure
D	- safety pressure sensor
F1-3	- electromagnetic flow meter
H	- thermostatically controlled heating system
M1-2	- volumetric pump
P_{atm}	- permeate at atmospheric pressure
P1-4	- pressure gauge
Pr, Pp	- average pressure in the retentate, permeate compartment
PDr, PDp	- pressure drop in the retentate, permetate compartment
T	- temperature sensor
V1-3	- valve
VAR1-2	- frequency variator

FIG. 1 Schematic drawing of the rig set-up.

1.7 I 10 type PCM, Vanves, France). The feed is monitored by a flow meter, F1 (ND 25 Krohne AltoFlux-1000, Romans, France), connected to a controller, C6 (Microcor IIIP, Coreci, Lyon, France) which changes the pump speed through a frequency controller, VAR1 (FMV 1003, Leroy Somer, Angoulême, France). A controller, C3, is connected to an air valve, V1 (CV 1.2 Varipak Masoneilan, Condé sur Noireau, France), that sets the retentate pressure, Pr, which is fed back by the two retentate, inlet and outlet piezoelectric pressure gauges P1 and P2 (0.02 bar accuracy) (Haenni, Jegenstorf, Switzerland) to a calculator CALC1 (Sfere, CVP400C, Chassieu, France). A safety pressure sensor, D (Danfoss, Trappes, France) protects the rig from an accidental pressure increase. Controller C5 monitors the retentate extraction flow rate through F3 (MG 711/F-D ND4, Mareg, Vincennes, France) by activating an air valve, V3 (CV 0.01, same make as V1). It is optional to withdraw concentrate at a constant rate through V3 and thus to operate at a constant volume concentration ratio. The feed tank (B) is situated close to the pump entrance as a venturic tube in order to operate on the minimum retentate dead volume it permits (1045 mL when equipped with a 1.2 m, 6 mm i.d. membrane).

The permeate side is equipped with a pump, M2, 50-600 $L \cdot h^{-1}$ (450 mL dead volume, same make as M1), a heat exchanger, and two pressure gauges, P3 and P4 (0.02 bar accuracy, same make as P1). The permeate extraction flow rate is controlled by the controller C2 (constant flux) or C1 (constant pressure) and is withdrawn through a flowmeter, F2 (ND 2.5, same make as F1), and a permeate pressure valve, V2 (CV 0.05, same make as V1). The dead volume of the permeate side is 940 mL (when equipped with a 1.2 m, 10 mm o.d. membrane), which makes it necessary to calculate the true permeate concentration from the concentration at the outlet of the permeate compartment, which is considered to be a perfectly stirred reactor.

All the data (temperature, pressure, flow velocity, permeate and retentate extraction flow, pH, etc.) are collected through an Analog to Digital Convertor Multi Channel Analyzer (μMac 4000, Analog Devices, Norwood, Massachusetts, USA), which is capable of registering data every $\frac{1}{3}$ s/12 channels. A line of data that contains the summation of the averaged readings taken every fraction of a second (depending on the number of channels in use) is printed at the operator's request. All data acquired by the Multi Channel Analyzer is registered by an IBM PS/2 computer.

Range of Operating Parameters

The domain of operating parameters of the rig is: tangential flow rate, 0–15 $m \cdot s^{-1}$; transmembrane pressure, 0–9 bar; flux, 10–7500 $L \cdot h^{-1} \cdot m^{-2}$;

retentate extraction, 0.2–50 $L \cdot h^{-1}$; volume concentration ratio, 0–100; temperature, 2–90°C. The ratio between membrane area and retentate dead-volume is around 20 m^2/m^3 for a tubular inorganic membrane. This ratio is 3 to 10 times lower than that of an industrial plant. Consequently, the residence time in the loop and the time to get to a desired volume concentration ratio are longer. The rig was operated according to needs, usually from 10 minutes to over 15 hours. The minimal volume of feed to ensure complete water clearing is about 3 L.

MODES OF OPERATION

Ultrafiltration with Permeate at Atmospheric Pressure, P_{atm}

At Constant Transmembrane Pressure (7)

For operation in this mode, the feed pump M1 is set to the desired flow velocity and the valve V1 to the desired pressure. Controller C3 maintains constant pressure through the calculator CALC1, which averages the signals obtained by the two retentate pressure gauges and operates as programmed.

At Constant Flux (8, 9)

Upon starting the run at constant flux, the flow velocity of the feed is programmed to the desired level, and when that has been achieved the retentate valve V1 is activated through controller C2 with valve V2 fully open. During a run, as soon as the permeate flux is lowered due to fouling, flowmeter F2 informs controller C2, which signals valve V1 to close, and then the pressure is increased in order to maintain the preset flux.

Filtration with Permeate under Static Counterpressure

With microfiltration, the tangential flow rate must be kept high, resulting in a high-pressure drop on the retentate side and consequently in high pressure. To work within a desirable range, (not exceeding 1 bar) in order to limit fouling, the permeate side must operate under pressure. This is achieved by closing permeate valve V2. When the desired retentate flow velocity is obtained (usually 6–9 $m \cdot s^{-1}$), several bars of pressure may be generated according to the module in use. Retentate pressure Pr, as $(Pr_{in} + Pr_{out})/2$, is set to the maximum pressure needed for the experiment (ultrafiltration, 6–9 bar; microfiltration, 2–4 bar).

At constant flux (10) the preset flux needed is achieved and then maintained by gradually opening valve V2. *At constant transmembrane pres-*

sure, permeate pressure (Pp), as $(Pp_{in} + Pp_{out})/2$, is preset to a value at which Pr − Pp equals the desired transmembrane pressure. Pp is set constant and controlled through C1 by opening valve V2.

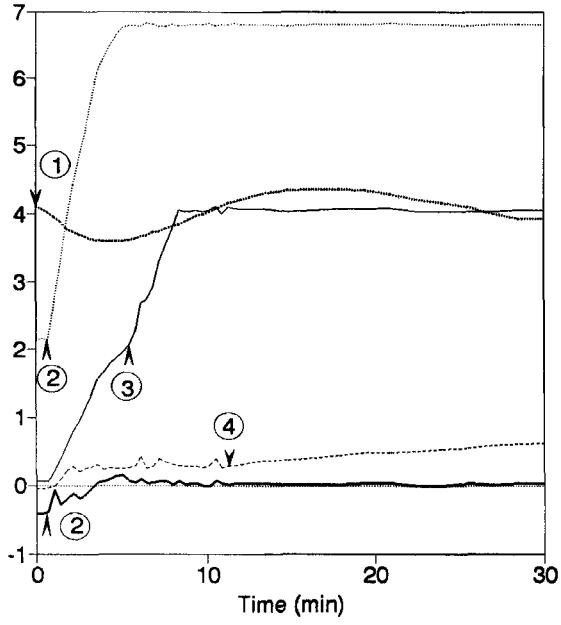
Filtration with Permeate under Dynamic Counterpressure

In order to operate at an equal transmembrane pressure along the filtration module, the permeate is circulated through a bed of polypropylene balls (2–3 mm diameter) by pump M2, cocurrent to the retentate, and equal pressure profiles are achieved on both sides of the membrane (3). The set pressure drop (PD) is calculated by calculators CALC3 and 4 (PDr and PDp) and is sent to controller C4. Controller C4 compares the two signals and maintains PDp = PDr by controlling frequency controller VAR 2, and thus the speed of pump M2. To ensure a high quality cocurrent system control, correction of the pressure drop by location of the pressure gauges relative to the inlet and outlet of the filtration tube should be performed. This way of calculating pressure drop makes it possible to perform whey microfiltration with a transmembrane pressure gradient lower than 0.02 bar instead of at 0.2 bar without correction (10). With this application of the rig to whey microfiltration, the transmembrane pressure gradient in the static mode was over 0.4 bar, and it increases as the retentate concentration increases.

To start filtration, valve V2 will be ordered to open:

1. *At constant transmembrane pressure*, controller C1 is given a permeate pressure value and it signals valve V2 to maintain it.
2. *At constant flux*, controller C2 is given a permeate flux value and signals valve V2 to maintain it. The set flux is now obtained and maintained by a decrease of the permeate pressure through controller C2, which signals valve V2 to open.

The loop is then in a fully automatic mode, with or without retentate bleeding. There is a separate control for either constant retentate extraction flow rate or constant volume concentration ratio. A scheme of the operational flow-path for stationary operating conditions is given in Fig. 2 where the recorded values demonstrate the good stability of the platform at stationary conditions (10). From numerous runs of ultrafiltration or microfiltration of various dairy products (milk, whey, caseinates, etc.), it can be stated that the standard variation coefficient of the controlled values are (in %): flux, ≤ 3; transmembrane pressure, ≤ 3; retentate pressure, ≤ 3; flow velocity, ≤ 0.2; transmembrane pressure gradient, ≤ 50 (with controlled values in the range of ± 0.03 bar); temperature, ≤ 2.5; volume concentration ratio ≤ 2.



			Variation coefficient (%)
1.	Temperature (°C)	4.0	2.5
2.	Retentate flow velocity ($m.s^{-1}$)	6.8	0.1
	Difference of pressure drop between retentate and permeate (bar)	0.02	50
3.	Retentate Pressure (bar)	4.0	0.3
4.	Transmembrane pressure (bar)	increases as a function of time	

FIG. 2 Example of operational flow-path to stationary working conditions for microfiltration of a 1% caseinate solution (M6 Carbosep membrane, 0.08 μm). In setting up the operating parameters, successively follow the order 1, 2, 3, and 4.

CONCLUSION

The rig provides a host platform for almost every experimental filtration module intended for either microfiltration or ultrafiltration. Recent improvements in the dynamic counterpressure system are promising tools for improving microfiltration and ultrafiltration performance. It remains

to be seen whether it is acceptable economically. However, from a scientific point of view, it provides a technical device for studying, understanding, and modeling crossflow filtration processes because filtration conditions of flux and transmembrane pressure can be maintained and controlled along the hydraulic path.

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