



Integration of quality-dependent prices in the optimization strategy for chemicals ultrapurification by reverse osmosis membrane cascades

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ABSTRACT

The present work is focused on the optimization of multistage reverse osmosis membrane cascades applied to the ultrapurification of chemicals for the semiconductor industry (hydrogen peroxide was chosen as case study). This paper is a part of the author's overall work on the subject. The novelty of this paper is the introduction of a price-dependent model for the product quality that can be adjusted between customer and producer. The membrane systems were formulated with product quality-dependent price resulting a non-linear programming problem. The optimal number of stages included in a cascade was strongly dependent of the desired product quality while the formulated quality-dependent price model determined the target purity. Five quality-dependent price fittings were used to illustrate the case study: linear, parabolic, exponential, sigmoidal, and bisigmoidal relationships between product quality and price. For the sigmoidal and bisigmoidal fittings, least operation conditions can afford revenues similar to ones under linear, parabolic, or exponential models, but with lower costs.

Keywords: Reverse osmosis; Membrane cascade; Hydrogen peroxide; Ultrapurification; Optimization; Product quality and price

1. Introduction

The importance of clean substrate surfaces in the fabrication of microelectronic devices has been recognized since the dawn of semiconductor device technology in the 1950s. It is now well known that the device performance, reliability, and product yield of silicon circuits are critically affected by the presence of

chemical contaminants and particulate impurities on the wafer surface [1]. Electronic chemicals, which are the chemicals and materials used to manufacture and package semiconductors and printed circuit boards, require extreme low content of metallic impurities to avoid reliability problems due to loss of oxide integrity or shortening of minority carrier lifetime [2,3]. Ultrapurification processes become necessary to achieve these exigent limits from technical grade

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chemicals. Among all the ultrapurification alternatives (distillation, adsorption, ion exchange, and membrane technologies), reverse osmosis emerges as the most desirable technology according to environmentally friendly criteria, as auxiliary chemicals are not needed and zero-effluent generation can be achieved by commercialization of the retentate streams to non-electronic purposes [4,5].

SEMI, the Semiconductor Equipment and Materials International association, is the entity serving the manufacturing supply chains for the microelectronic, display, and photovoltaic industries. SEMI assists the worldwide development of the most respected technical standards in this technological sector. Among all the topics regulated, some refer to the electronic chemicals characteristics. For the particular case of hydrogen peroxide (chosen as case study because it is one of the most demanded wet electronic chemicals, as it is employed to remove photoresists and to clean silicon wafer surfaces), SEMI C30-1110 Document defines the requirements that the chemical must fulfill to be considered as electronic grade chemical [6]. Table 1 shows the limits imposed by the corresponding SEMI Document, including the maximum metallic concentrations allowed.

Multistage process becomes indispensable to achieve the exigent electronic grades. Integration of the several stages by recirculation of retentate streams to previous stages, configuring a membrane cascade, is considered an effective way to combine a high removal of metallic impurities with a high recovery rate [7–10]. Reverse osmosis membrane cascades have demonstrated their technical and economic viabilities for chemicals ultrapurification [11] and optimization according to economic criteria of the design and operation variables of industrial scale installations have been carried out [12]. The complete optimization of reverse osmosis cascades and other types of networks should include the optimal design of both individual modules and the network configuration. The problem of the design of reverse osmosis networks has been considered from the optimization techniques by the

generation of the configurations and their optimization with mass and energy integration or multi-objective optimization, mainly for desalination units of seawater in order to minimize costs and energy consumption or to maximize permeate production and economic profit [13–20].

Water quality, in terms of both raw water quality and product water specifications, has an impact on desalination process design and optimization. Different feed water qualities, having different corresponding compositions, have been taken into consideration, and the effects over the optimal design and operation conditions of the networks have been investigated [21–26]. However, considerations about the quality of the obtained product have not received so much attention: just a fixed quality constraint is imposed to be fulfilled. The optimization of hydrogen peroxide ultrapurification processes has followed an analogous path and only the achievement of the SEMI Standard requirements were established without further concern about the quality of produced hydrogen peroxide.

The aim of the present work was the analysis of the influence of the quality of the product over the economically optimum membrane systems by means of product quality-dependent prices. A framework with quality-dependent prices implied an opportunity for integration of economic and product quality objectives in a unique economic objective. Five different quality-dependent price fittings were proposed to illustrate a case study based on the production of Grade 1 chemical by a three-stage cascade. These fittings included linear, parabolic, exponential, sigmoidal, and bisigmoidal relationships between product quality and price.

As mentioned above, a review over the demand by the semiconductor industry of the different wet chemicals shows that hydrogen peroxide is among the most consumed one [27,28]. It is present in several mixtures employed in the silicon wafer cleaning sequence to eliminate organic matter, particulate contamination, and metallic impurities. The demand of hydrogen peroxide by the semiconductor sector is expected to

Table 1

Impurity limits and other requirements for electronic grade hydrogen peroxide according to SEMI C30-1110 standard. Assay H₂O₂ 30–32%

SEMI electronic grade	Total oxidizable carbon (TOC) limit (ppm)	Anion limit range	Cation limit range
1	20	2–5 ppm	10–1,000 ppb
2	20	200–400 ppb	5–10 ppb
3	20	200–400 ppb	1 ppb
4	10	30 ppb	100 ppt
5	10	30 ppb	10 ppt

continue growing despite the static importance of this chemical among the wet chemicals due to emerging alternatives [29].

Therefore, the ultrapurification of hydrogen peroxide was selected as case study. As illustrating examples, industrial-scale systems were designed to treat an annual target of 9,000 tons of technical grade peroxide in order to produce each electronic SEMI Grades (from 1 to 5) by membrane cascades. The reverse osmosis membrane selected for these processes systems was the BE model from Woongjin Chemical, which is a polyamide membrane that has demonstrated good performance for hydrogen peroxide ultrapurification [12]. The full characterization of the membrane transport properties and the corresponding model parameters can be found in a previous work [30].

2. Process modeling and objective formulation

The mathematical model for integrated countercurrent membrane cascades is based on overall and solute (metallic impurities) material balances and the Kedem–Katchalsky transport equations. The general scheme of a *n*-stage integrated countercurrent membrane cascade with implementation of bypass stream in the last stage is depicted in Fig. 1 [31].

The simplified Kedem–Katchalsky equations for solvent and solute transport through reverse osmosis membranes [32] were selected to model the membrane separation performance:

$$J_V = L_p \Delta P \tag{1}$$

$$R = \frac{\sigma J_V}{J_V + \omega'} \tag{2}$$

Direct application of the Kedem–Katchalsky equations is enough to define the characteristics of the permeate streams (flow and metal concentrations) as function of the membrane area of the corresponding *i* stage:

$$P(i) = A_{(i)} J_{V(i)} \tag{3}$$

$$C_{P(i)}^{metal} = (1 - R_{(i)}^{metal}) C_{F(i)}^{metal} \tag{4}$$

The recovery ratio of each module $Rec_{(i)}$ and the bypass ratio T_{BY} , which are among the main operation decisions, were defined by the following expression:

$$Rec_{(i)} = \frac{P(i)}{F(i)} \tag{5}$$

$$T_{BY} = \frac{Q_{BY}}{P(n-1)} \tag{6}$$

The product quality was formulated as a dimensionless safety factor SF, defined as the quotient between the limit concentration (fixed by SEMI standard limiting requirements) and the product concentration:

$$SF = \frac{C_{SEMI}}{C_{EG}} = \frac{\text{Failure concentration}}{\text{Design concentration}} \tag{7}$$

According to the definition of the safety factor SF, each metallic solute implies a different safety factor. Nonetheless, the safety factor of the final product is the minimal value among the several individual factors corresponding to each metal.

The quality of the product was correlated with the safety factor by means of product quality-dependent prices:

$$Y_{EG} = \text{Mathematical function (SF)} \tag{8}$$

Five different quality-dependent price models were proposed: linear, parabolic, exponential, sigmoidal, and bisigmoidal relationships between product quality and price.

The mathematical equations of the product price as a function of the quality (given in terms of safety factor) covered the linear, parabolic, and exponential equations, indicating by simple fittings the significant increase of the hydrogen peroxide market price from Grade 1 to Grade 5. The sigmoidal and bisigmoidal

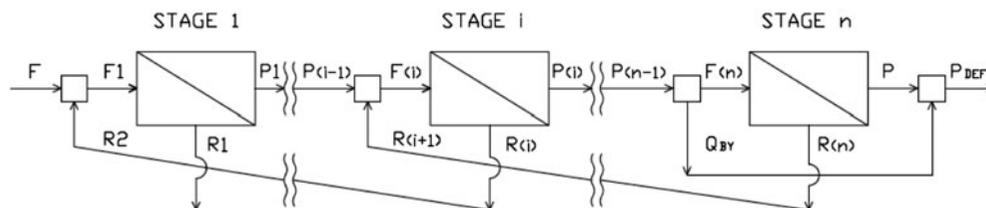


Fig. 1. General scheme of a *n*-stage integrated countercurrent membrane cascade.

models were also used in order to consider the customer requirements of a whole range of quality at the same product price (within the same Grade number), for covering different applications.

The economic profit (Z) of the process is defined as the difference between total daily revenues (Rev) and costs (TC):

$$Z = \text{Rev} - \text{TC} \quad (9)$$

The electronic grade hydrogen peroxide obtained as the final-stage permeate is the product of the ultrapurification system, but the retentate of the first stage can also be considered as a valuable by-product, since it can be commercialized as non-electronic grade chemical, useful for those applications where the metallic content of the hydrogen peroxide is not a limiting factor. According to this assumption, two terms have to be incorporated to assess the total daily revenues:

$$\text{Rev} = P_{i_{\text{DEF}}} Y_{\text{EG}} + R1Y_{\text{by}} \quad (10)$$

where $P_{i_{\text{DEF}}}$ represents the final permeate stream of a membrane cascade that integrates i total stages and $R1$ the retentate stream of the stage 1.

The total daily costs of the process are defined as the sum of the capital costs (CC) and the operation costs (OC). The capital costs attributable to membranes or to the rest of the installation are differentiated, while the operation costs are itemized into raw materials, labor, energy, and maintenance costs:

$$\text{TC} = \text{CC} + \text{OC} \quad (11)$$

$$\text{CC} = \text{CC}_{\text{memb}} + \text{CC}_{\text{ins}} \quad (12)$$

$$\text{OC} = \text{OC}_{\text{raw}} + \text{OC}_{\text{lab}} + \text{OC}_{\text{en}} + \text{OC}_{\text{m}} \quad (13)$$

The capital costs of the membranes, considering straight-line depreciation along their lifetime and constant transport properties, were expressed as function of the total membrane area of the installation. Once the membranes costs were defined, the capital costs corresponding to the rest of the installation were related to them by means of an empirical coefficient that expresses the contribution of the investment in membranes to the total capital costs [12].

The operation costs are essentially based on the consumption of the corresponding resource, except for the case of maintenance costs, which are function of the total capital costs. The only required raw material

was technical grade hydrogen peroxide as feed stream, and the complete installation was designed to be totally operated by a single worker.

The daily profit (Z), defined by Eq. (9), was chosen as the formulated objective function to maximize. All the model variables have been expressed in terms of the independent operation variables, that is to say, recovery rates (Rec_i), bypass ratio (T_{BY}), and applied pressures (ΔP_i). Constraints for the independent variables have been set to limit their values to defined ranges.

The problem resulted in mathematical terms as a nonlinear programming (NLP) one and the model can be expressed as follows:

$$\max N(x) = \{Z(x)\}$$

$$\text{s.t. } h(x) = 0$$

$$g(x) \geq 0$$

$$x_L \leq x \leq x_U$$

$$x \in R^n$$

where Z is the daily profit, x the vector of continuous independent variables $\Delta P(i)$, T_{BY} and $\text{Rec}(i)$, h the vector of equality constraint functions (material balance equations, transport equations, and economic considerations) and g the vector of inequality constraint functions that define the product requirements based on the concentration limits for each metal imposed by the SEMI Document.

General Algebraic Modeling System (GAMS) software was selected as optimization tool to manage the NLP model using CONOPT3 solver. The GAMS is a high-level modeling system for mathematical programming and optimization. It consists of a language compiler and a stable of integrated high-performance solvers.

3. Results and discussion

The definition of quality-dependent market prices is a common practice for both products and services. The assumption of this type of price policy can be a useful tool to adjust offer and demand, and a proper formulation should afford several advantages for both producers and customers. For the particular case of electronic grade hydrogen peroxide, the production of this chemical is included in a much customer-oriented sector where the presence of a much reduced number of clients is a typical circumstance that defines the

Table 2
Model equations for quality-dependent prices and the defined parameter values

Model	Equation	Parameters
Linear	$Y_{EG} = tb + a \cdot SF$	$a = 5.94$ $b = 2,494$
Parabolic	$Y_{EG} = b + a(SF - 1)^2$	$a = 0.06$ $b = 2,500$
Exponential	$Y_{EG} = b + a e^{k \cdot SF}$	$a = 3$ $b = 2,497$ $k = 0.0528$
Sigmoidal	$Y_{EG} = b + \frac{a}{1 + e^{(SF_m - SF)/k}}$	$a = 588$ $b = 2,500$ $SF_m = 55$ $k = 6$
Bisigmoidal	$Y_{EG} = b + \frac{a_1}{1 + e^{(SF_{m1} - SF)/k_1}} + \frac{a_2}{1 + e^{(SF_{m2} - SF)/k_2}}$	$a_1 = 150$ $a_2 = 438$ $b = 2,500$ $SF_{m1} = 15$ $SF_{m2} = 55$ $k_1 = 2$ $k_2 = 2$

semiconductor industrial sector. Under these conditions, the price negotiation between seller and buyer can be very concise, and identification of optimal conditions for both parts should be a common effort.

From the point of view of this paper, a framework with quality-dependent prices implied an opportunity for integration of economic and product quality objectives in a unique economic objective, as other authors had investigated before [33].

Five different quality-dependent price models were proposed to illustrate a case study based on the production of SEMI Grade 1 hydrogen peroxide by a three-stage cascade. These models included linear, parabolic, exponential, sigmoidal, and bisigmoidal relationships between product quality and price. The model was formulated in such a way that the corresponding price values for the extreme products with lowest and highest quality (characterized by SF values 1 and 100, respectively) were identical in all cases. The model equations and the defined parameter values can be observed in Table 2.

The optimization results, including optimal profits, safety factors, and prices, are compiled in Table 3. As it can be observed, the optimal conditions for the linear, parabolic, and exponential models are very similar, as the maximum profits corresponded to the maximum prices, which can be achieved by the production of the highest purity chemical. However, the cases of the sigmoidal and bisigmoidal models are a bit different. In these scenarios, the maximum profits were not related to highest purity peroxide because prices very close to their maximal values can be found

before the highest safety factors were attained. Fig. 2 shows the evolution of the prices for the proposed models as function of the safety factor and includes the evolution of the maximum achievable profits for each product quality in order to have a better understanding of the system. The maximum profit followed mimetically the trend defined by the price model. As the sigmoidal and bisigmoidal fittings exhibit a flat plateau with nearly maximal prices for a range of safety factors before the highest value (Fig. 2(d) and (e)), least exigent operation conditions can afford revenues similar to ones under linear, parabolic, or exponential models, but with lower costs. As consequence, the optimal profits of the sigmoidal and bisigmoidal scenarios can be higher despite slightly lower prices.

After the analysis of the results, the bisigmoidal fitting was applied to other SEMI Grades. The parameters for proposed bisigmoidal model prices for Grades 2–4 are shown in Table 4, and the evolution of

Table 3
Optimization results of SEMI Grade 1 hydrogen peroxide, including optimal profits, safety factors, and product prices

Model	Z_{opt} (\$/d)	SF_{opt}	$Y_{EG opt}$ (\$/m ³)
Linear	44,814	100	3,088
Parabolic	44,815	100	3,088
Exponential	44,811	100	3,088
Sigmoidal	44,943	85	3,084
Bisigmoidal	45,262	67	3,087

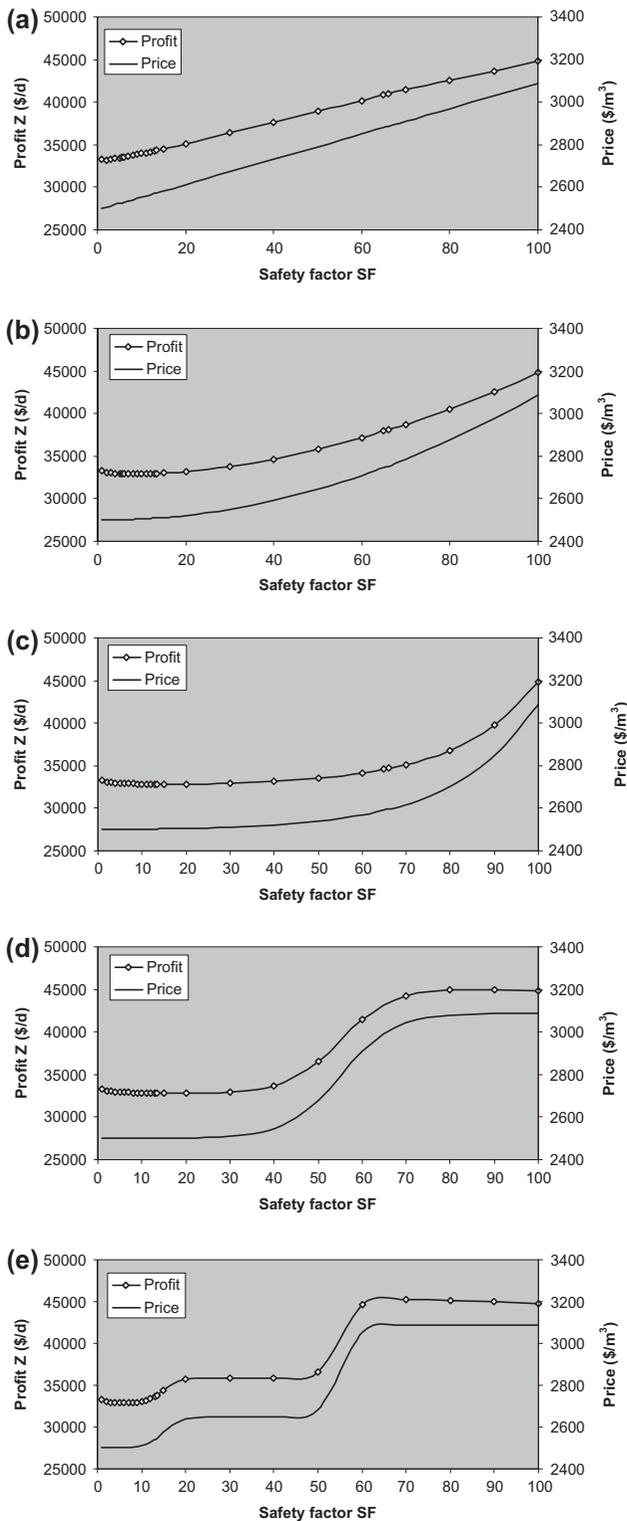


Fig. 2. Evolution of the prices for the proposed models as function of the safety factor. Fitting (a) linear, (b) parabolic, (c) exponential, (d) sigmoidal, and (e) bisigmoidal.

the prices for the proposed models as function of the safety factor is represented in Fig. 3, which also

Table 4

Parameters for bisigmoidal model prices for SEMI Grades 2–4 hydrogen peroxide

SEMI Grade	a_1	a_2	b	SF_{m1}	SF_{m2}	k_1	k_2
2	40	120	3,537	2.5	5.5	0.200	0.2
3	1,000	3,000	3,780	2.5	5.5	0.125	0.2
4	600	1,800	8,721	2.5	5.5	0.125	0.2

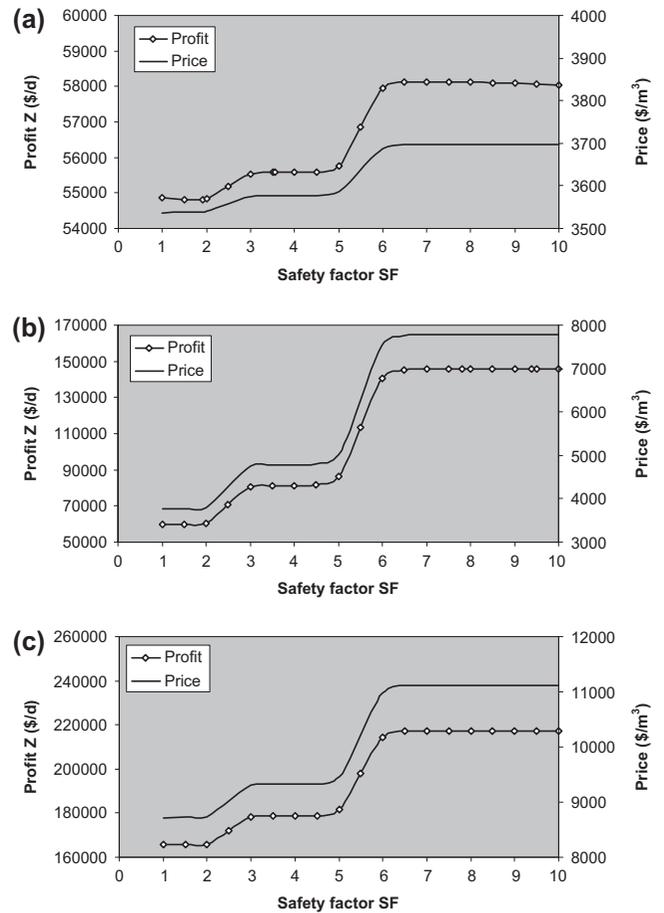


Fig. 3. Evolution of the prices for the bisigmoidal fitting as function of the safety factor. SEMI Grade H₂O₂ (a) Grade 2, (b) Grade 3, and (c) Grade 4.

Table 5

Optimization results of bisigmoidal fitting for SEMI Grades 1–4 hydrogen peroxide

	Z_{opt} (\$/d)	SF_{opt}	$Y_{EG\ opt}$ (\$/m ³)
Grade 1	45,262	67	3,087
Grade 2	58,136	7.9	3,697
Grade 3	145,682	7.8	7,780
Grade 4	217,275	7.6	11,121

includes the evolution of the maximum achievable profits for each product quality by different membrane cascades. The corresponding optimization results are compiled in Table 5. The price of the product increases 3.6 times from Grade 1 to Grade 4, while the process profit was multiplied by a factor of 5.

4. Conclusions

The consideration of quality-dependent prices implied an opportunity for the integration of economic and product quality objectives in a unique economic objective. The negotiation of a quality-dependent price model between producer and customer can be an advantageous option for both parts, and the formulation of this model would determine the target quality of the produced chemical.

Five different quality-dependent price models were proposed to illustrate a case study based on the production of SEMI Grade 1 hydrogen peroxide by a three-stage cascade. These fittings included linear, parabolic, exponential, sigmoidal, and bisigmoidal relationships between product quality and price.

The optimal conditions for the linear, parabolic, and exponential models were very similar, as the maximum profits corresponded to the maximum prices, which can be achieved by the production of the highest purity chemical. However, in the cases of the sigmoidal and bisigmoidal fittings, the maximum profits were not related to highest purity peroxide because prices very close to their maximal values can be found before the highest safety factors were attained. Considering sigmoidal or bisigmoidal fittings, least exigent operation conditions can afford revenues similar to ones under linear, parabolic, or exponential fittings, but with lower costs.

The bisigmoidal fitting was also applied to the production of SEMI Grades 1–4 hydrogen peroxide that resulted the process profit increase by a factor of 5 (Grade 4 related to Grade 1).

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Nomenclature

A	—	membrane area (m^2)
CC	—	capital costs ($\$/\text{d}$)
C_p	—	permeate concentration of metal (ppb)

CF	—	feed concentration of metal (ppb)
F	—	feed flowrate (m^3/s)
J_V	—	permeate flux (m/s)
L_P	—	hydraulic permeability coefficient ($\text{m}/\text{s bar}$)
OC	—	operating costs ($\$/\text{d}$)
OC_{raw}	—	raw materials cost ($\$/\text{d}$)
OC_{lab}	—	labor cost ($\$/\text{d}$)
OC_{en}	—	energy cost ($\$/\text{d}$)
OC_{m}	—	maintenance cost ($\$/\text{d}$)
P	—	permeate flowrate (m^3/s)
Q_{BY}	—	bypass flowrate (m^3/s)
R	—	rejection coefficient
Rec	—	recovery ratio
Rev	—	revenues ($\$/\text{d}$)
SF	—	safety factor
T_{BY}	—	bypass ratio
TC	—	total costs ($\$/\text{d}$)
Y_{EG}	—	price of electronic grade product ($\$/\text{m}^3$)
Y_{BY}	—	price of retentate product of the first stage ($\$/\text{m}^3$)
Z	—	process profit ($\$/\text{d}$)

Greek symbols

ΔP	—	pressure difference across the membrane (bar)
σ	—	reflection coefficient
ω'	—	modified coefficient of solute permeability (m/s)

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