# ULTRAFILTRATION OF SILICA SOLS*

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Ultrafiltration of three laboratory made silica and two commercial silica sols was studied using Amicon YC membrane in a 200 ml capacity batch-cell. The effect of silica particle size, stirring conditions, pressure, pH and silica contents on ultrafiltration was investigated.

The results obtained indicate that the smaller particles have, disregarding the stirring conditions, lower filtration flux. The differences observed in filtration flux are more pronounced in the conditions without stirring. The obtained value of the membrane resistance is independent of the conditions investigated (stirring, pressure, pH, silica contents and particle size). The values of the resistance of polarized solids, specific resistance, and the mass of gel per membrane surface unit were calculated for all experimental conditions.

Colloidal silica is largely applied in various industries[1]. There are different procedures for obtaining this material, but, in practice, the ion exchange method is most used. However, using this procedure diluted solutions of colloidal $SiO_2$ are obtained, while for commercial purposes concentrated colloidal $SiO_2$ is required. Concentrating of colloidal $SiO_2$ used to be performed by evaporation. A cheaper and simpler way of concentrating colloidal $SiO_2$ is ultrafiltration. The sudden development of ultrafiltration as a separation technique was carried out thanks to the elaboration of synthetic polymeric membranes.

There are only two papers[2,3] in literature devoted to the study of ultrafiltration of colloidal $SiO_2$. Fane[2] has investigated ultrafiltration of suspensions of various silica-based particulates (silica sol and diatomaceous earth). He reported that the effect of particle size on ultrafiltration of fine colloids is different for stirred and unstirred conditions. The use of acetate cellulose membrane type UAM-200 with $18-20$ nm pore diameter and polyamide membrane type MB1 $-10$ for concentrating of colloidal silica, have been studied by Frolov et al.[3]

In this paper, the results of investigations of the ultrafiltration of three laboratory made silica and two commercial silica sols have been summed up. The effect of the silica particle size, stirring conditions, pressure, pH and silica contents on ultrafiltration was investigated.

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**THEORETICAL**

According to filtration theory[2,4], the basic filtration equation can be written in the form:

$$J = \frac{dV}{dt}\frac{1}{A_m} = \frac{\Delta P}{(R_m + R_p)\,\eta}\,. \tag{1}$$

The resistance of the polarized solids, $R_p$, can be defined by the Carman–Cozeny dependence[5], provided that the layer of $SiO_2$ gel formed on the surface of the membrane, is considered as a small-particles porous layer though which the filtrate flows laminarly:

$$\frac{\Delta P_g}{\delta_g} = 180\frac{(1 - \varepsilon)^2}{\varepsilon^3}\frac{\eta J}{d_p^2}\,. \tag{2}$$

The thickness of the deposited gel layer can be approximately determined from:

$$\delta_g = \frac{m_p}{A_m(1 - \varepsilon)\,\varrho}\,. \tag{3}$$

Substituting Eq. (3) into Eq. (2) produces:

$$J = \Delta P_g \bigg/ \left(\frac{180(1 - \varepsilon)}{d_p^2\varepsilon^3\varrho}\frac{m_p\eta}{A_m}\right) = \Delta P_g\bigg/\frac{a m_p\eta}{A_m} = \Delta P_g/\eta R_p\,. \tag{4}$$

Therefore, $R_p$ can be determined through specific resistance, $a$:

$$R_p = \frac{a m_p}{A_m}\,. \tag{5}$$

The expression $a = 180(1 - \varepsilon)/(d_p^2\varepsilon^3\varrho)$ connects approximately the (gel) layer characteristics with the gel specific resistance. It can be concluded that the specific resistance increases with the decrease of the particle size.

The migration of particles towards the membrane surface (convective transport) brings about the so-called concentration polarization, i.e. an increase of the particles concentration next to the membrane surface. Under the concentration gradient's influence, there occurs a movement of the particles back to bulk concentration — the so-called back-transport. The back-transport increases with the increase of the concentration of particles next to the membrane surface, as long as the flux of colloidal particles, that are being carried towards the membrane surface, becomes equal with the diffusion flux of the particles in the opposite direction. When the convective

and the back-transport are equalized, there occurs a stationary state that can be expressed by the equation:

$$JC = D\frac{dC}{dx}.\tag{6}$$

The integration of Eq. (6) gives[6]:

$$J = \frac{D}{\delta}\ln\frac{C_g}{C_b} = K\ln\frac{C_g}{C_b}.\tag{7}$$

From Eq. (7), it follows that filtrate flux in the stacionary state does not depend either on the difference in pressure, or on the porosity of the membrane, but only on particles characteristics ($D$ and $C_g$) and the thickness of the boundary layer, $\delta$.

Using different fluid flow procedures, it is possible to reduce the boundary layer thickness, and to increase the mass transport coefficient $K = D/\delta$.

In the case of ultrafiltration, the increase of the particles concentration next to the membrane surface causes an aggregation (coagulation) of colloidal particles, or macromolecules, and the formation of gel. As soon as the concentration value within which colloidal particles are able to exist as discrete spheres is exceeded, the particles join together spontaneously and the gel is formed. If the gel layer increases gradually, the filtrate flux will be decreasing.

Eq. (7) is valid only on condition that a gel layer is formed on the surface of the membrane. In cases of ultrafiltration without stirring, gel is formed even at very low pressures because of the low back-transport rate, and, in a briefer period of time, it is possible to neglect the back-transport. Then $m_p = VC_b$, and on condition that $\Delta P = $ const, Eqs (1) and (5) give the well-known filtration equation:

$$\frac{t}{V} = \frac{R_m\eta}{\Delta PA_m} + \frac{C_b a\eta}{2\,\Delta PA_m^2}\,V.\tag{8}$$

For ultrafiltration without stirring, a line may be constructed on the basis of experimental data in the $t/V$–$V$ coordinate system, from the slope of which, on the basis of Eq. (8), specific resistance is determined, while the membrane resistance is calculated from the intercept.

In the case of ultrafiltration with stirring, stationary state is quickly established, so that $m_p \neq VC_b$ and Eq. (8) is no longer valid. Therefore, specific resistance and membrane resistance may be determined only for unstirred ultrafiltration data.

The stirring increases the diffusion of particles from the membrane surface towards the bulk concentration considerably, so that, at low pressures, when the particles convective transport is small, gel formation next to the membrane surface does not occur. For the appearance of gel, a certain minimum pressure value, called threshold

transmembrane pressure, is required. The filtrate flux in the stationary state increases with the increase of pressure only up to the threshold pressure. Above the threshold pressure, gel is formed, so that the flux in the stationary state no longer depends on pressure, and Eq. (7) is then valid.

The threshold pressure value depends on: the type and concentration of the solution, the speed of stirring and the porosity of the membrane. The threshold pressure increases with the increase of the speed of stirring and with the decrease of the concentration of the solution and the decrease of the membrane porosity[6].

## EXPERIMENTAL

Five different forms of colloidal silica, three of which were laboratory made, while the remaining two were commercial silica sols, were chosen for ultrafiltration experiments. Laboratory made silica sols were prepared by ion exchange method[7], while for commercial silica sols, the products of Nalco Chemical Co., labelled Nalcoag 1030 and Nalcoag 1060, have been used.

Properties of all silica sols used are listed in Table I.

Colloidal solutions containing 10·0, 30·5, 62·2 and 106 kg m$^{-3}$ of $SiO_2$ were prepared for ultrafiltration experiments. In the case of laboratory made silica sols, colloidal solutions containing 10·0 and 30·5 kg m$^{-3}$ of $SiO_2$ were prepared by diluting the initial solutions with distilled water, while solutions with 62·2 and 106 kg m$^{-3}$ of $SiO_2$ were obtained by concentrating the inital solution by means of ultrafiltration.

Experiments were performed using an ultrafiltration cell produced by Amicon, type 8200 (capacity 200 ml) and with Diaflo membrane produced by Amicon, type YC 05. This was a non-cellulosic membrane with 2·1 nm pore diameter and a nominal molecular-mass cutoff 500. The cell diameter was 62 mm and the effective membrane area was 28·7 cm$^2$. The cell was supplied with a plasticized stirrer.

The initial solutions volume measured 100 ml in all investigations. In order to obtain a fixed pressure in the cell, compressed nitrogen was used. In all investigations, ultrafiltration was performed until 10 ml of the permeate were collected in a graduate cylinder.

All investigations were performed at a 22 ± 1°C temperature.

TABLE I
Characteristics of silica sols used for ultrafiltration experiments

| Characteristics | Laboratory made | | | Nalcoag | |
|---|---|---|---|---|---|
| | 1 | 2 | 3 | 1 030 | 1 060 |
| Silica content, kg m$^{-3}$ | 43·0 | 43·0 | 43·0 | 265 | 374 |
| pH | 9·2 | 10·2 | 10·2 | 10·2 | 8·5 |
| Particle size, nm | 8·5 | 14·1 | 20·3 | 17·0 | 41·0 |
| Specific surface area of silica, m$^2$ g$^{-1}$ | 320 | 193 | 134 | 160 | 66 |

## RESULTS AND DISCUSSION

Dependences of permeate volume with time for ultrafiltration with and without stirring for two different samples of colloidal silica are illustrated in Fig. 1. Similar dependences were found for other samples of colloidal silica, but for the sake of brevity these dependences are not presented in Fig. 1. It is evident that the permeate volume increases with the increase of particle size and the increase of the speed of stirring during a certain period of time.

When ultrafiltration with stirring is concerned, a linear dependence of volume and time is obtained, which indicates that, during stirring, stationary state, i.e. the constant value of the filtration rate, is quickly established.

In the case of ultrafiltration without stirring, the flux gradually decreases, while stationary state is practically never established. This is caused by the increase of the gel layer thickness, which, according to Eq. (5), brings about an increase in the polarization resistance. Since the membrane resistance and the applied pressure do not change, the increase of $R_p$, according to Eq. (1), brings about a decrease of the flux. Differences observed in the filtration rate, for various particle sizes, are more prominent for ultrafiltration without stirring.

The dependence of mean flux value, $J_m$, on pressure for two different colloidal silica samples in the case of ultrafiltration without stirring is given in Fig. 2. The mean flux value is equal to the total volume of permeate (in our case 10 cm$^3$) divided by ultrafiltration time. It can be seen that the flux increases linearly with the increase
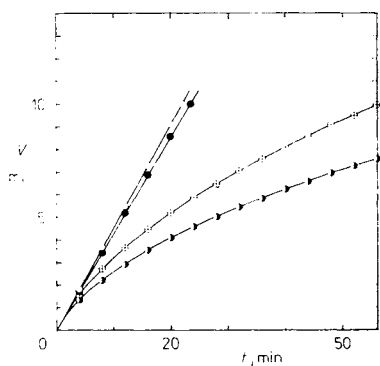


FIG. 1

Dependence of permeate vs time for colloidal silica solutions. $\Delta P = 0.2$ MPa, 106 kg m$^{-3}$ SiO$_2$. Particle diameter: ◑ 8.5 nm, unstirred; ◔ 41 nm, unstirred; ● 8.5 nm, stirred; 0.41 nm, stirred

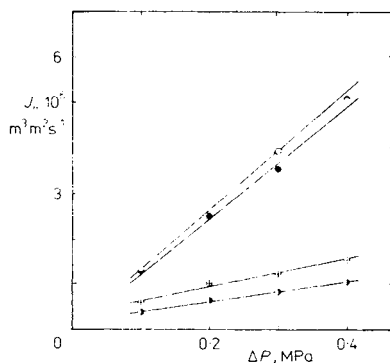FIG. 2

Dependence of mean flux on pressure for colloidal silica solutions. Unstirred. Particle diameter: ◑ 8.5 nm, 106 kg m$^{-3}$ SiO$_2$; ⊕ 41 nm, 106 kg m$^{-3}$ SiO$_2$; ● 8.5 nm, 10.0 kg . . m$^{-3}$ SiO$_2$; ○ 41 nm, 10.0 kg m$^{-3}$ SiO$_2$

of pressure. The flux increase is greater for lower $SiO_2$ concentrations. Flux is also somewhat greater for solutions containing $SiO_2$ particles of a larger diameter.

From Fig. 3 it can be noticed that, in the case of ultrafiltration without stirring, the mean flux value decreases with the increase of $SiO_2$ concentration. The initial flux is always the same at identical $\Delta P$ values, regardless of $SiO_2$ concentration. With the increase of $SiO_2$ concentration, flux decreases more quickly, and this influences a decrease in the mean flux value. The dependence of the mean flux value and the logarithm of $SiO_2$ concentration is linear, while the segment of the line on the abscissa gives the concentration of $SiO_2$ on the gel surface[2].

Eq. ($1$) for ultrafiltration of distilled water is:

$$J = \frac{\Delta P}{R_m \eta} \quad (R_p = 0)$$

from which we obtain:

$$R_m = \frac{\Delta P}{J\eta} . \tag{9}$$

For experimental conditions, water viscosity is $\eta = 0.93 \; 10^{-3}$ Pa s, so that, on the basis of data obtained by ultrafiltration of distilled water at different pressure, the membrane resistance was calculated from Eq. ($9$):

$$R_m = (7.300 \text{ m} \pm 0.045) \; 10^{13} \text{ m}^{-1} .$$

The value obtained is in good agreement with the membrane resistance that has been calculated on the basis of Eq. ($8$):

$$R_m = (7.596 \pm 0.193) \; 10^{13} \text{ m}^{-1} .$$
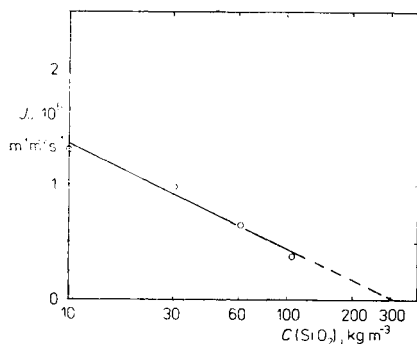


FIG. 3

Variation of mean flux with concentration for colloidal silica. $\Delta P = 0.1$ MPa. Unstirred; particle diameter 8.5 nm

The membrane resistance value is indenpendent of the investigations conditions (stirring, pressure, pH, concentration and size of $SiO_2$).

The membrane resistance and flux being known, polarization resistance can be determined from Eq. (1). Since in the case of unstirring ultrafiltration polarization resistance gradually increases, and reaches maximum value at the end of filtering, $R_p$ was determined at the end of ultrafiltration, for all unstirring experiments.

From Table II it can be noticed that polarization resistance increases with increase of pressure at identical (constant) $SiO_2$ concentration, and that it rises with the rise of $SiO_2$ concentration at the same pressure. It can also be seen that $R_p$ is greater for the colloidal $SiO_2$ solution having smaller particles. Increase of $R_p$ is influenced by a decrease of gel porosity. Gel porosity is smaller if colloidal $SiO_2$ particles are smaller, and pressure and concentration greater.

The share of polarization resistance, $R_p$, in the total resistance, $R_m + R_p$, increases with the increase of $SiO_2$ contents. Thus, for an $SiO_2$ having a particles diameter $d_p = 8.5$ nm, and at $10.0$ kg m$^{-3}$ of $SiO_2$ concentration, the polarization resistance share is from 11 to 14% (which depends on $\Delta P$), while at 106 kg m$^{-3}$ of $SiO_2$ concentration, it measures from 73 to 82%. Therefore, the gel layer resistance on the membrane surface presents the main resistance to permeate flow in the case of more concentrated $SiO_2$ solutions.

Using Eq. (8), and on the basis of experimental data, specific resistance was determined from the slope of the lines $t/V = SV + I$ (where $S$ and $I$ are slope and intercept, respectively). This specific resistance is presented in Table III. Dependence of specific resistance on pressure, on $SiO_2$ concentration and on particle size is identical to the ones established for polarization resistance. Using Eq. (5), the mass of gel per membrane surface unit $(m_p/A_m)$ was determined (Table IV).

Analyzing data in Tables II—IV, it can be seen that the increase of polarization resistance with the increase of pressure at same $SiO_2$ concentration, is not con-

TABLE II

Polarization resistance, $R_p$, values for ultrafiltration without stirring ($R_p \cdot 10^{-13}$, m$^{-1}$). $C(SiO_2)$ in kg m$^{-3}$

| $\Delta P$ MPa | $C(SiO_2)$, $d_p = 8.5$ nm | | | | $C(SiO_2)$, $d_p = 41.0$ nm | | | |
|------|------|------|------|------|------|------|------|------|
|      | 10.0 | 30.5 | 62.2 | 106  | 10.0 | 30.5 | 62.2 | 106  |
| 0.1  | 0.97 | —    | —    | 20.8 | 0.44 | —    | —    | 10.1 |
| 0.2  | 1.00 | 4.29 | 12.7 | 26.8 | 0.70 | 3.66 | 5.98 | 13.8 |
| 0.3  | 1.22 | —    | —    | 31.4 | 1.17 | —    | —    | 18.0 |
| 0.4  | 1.28 | —    | —    | 35.8 | 1.41 | —    | —    | 19.6 |

TABLE III
Specific resistance, $a$, values for ultrafiltration without stirring ($a \cdot 10^{-14}$, m kg$^{-1}$). $C(SiO_2)$ in kg m$^{-3}$

| $\Delta P$ MPa | $C(SiO_2)$, $d_p = 8.5$ nm | | | | $C(SiO_2)$, $d_p = 41.0$ nm | | | |
|---|---|---|---|---|---|---|---|---|
| | 10·0 | 30·5 | 62·2 | 106 | 10·0 | 30·5 | 62·2 | 106 |
| 0·1 | 5·76 | — | — | 11·1 | 2·40 | — | — | 5·42 |
| 0·2 | 6·24 | 8·05 | 11·7 | 14·6 | 3·67 | 6·67 | 5·45 | 7·51 |
| 0·3 | 6·44 | — | — | 17·0 | 6·15 | — | — | 9·77 |
| 0·4 | 6·70 | — | — | 19·5 | 7·47 | — | — | 10·7 |

TABLE IV
Mass of gel per membrane surface unit for ultrafiltration without stirring ($m_p/A_m$, kg m$^{-2}$). $C(SiO_2)$ in kg m$^{-3}$

| $\Delta P$ MPa | $C(SiO_2)$, $d_p = 8.5$ nm | | | | $C(SiO_2)$, $d_p = 41.0$ mn | | | |
|---|---|---|---|---|---|---|---|---|
| | 10·0 | 30·5 | 62·2 | 106 | 10·0 | 30·5 | 62·2 | 106 |
| 0·1 | 0·017 | — | — | 0·186 | 0·018 | — | — | 0·186 |
| 0·2 | 0·016 | 0·053 | 0·109 | 0·184 | 0·019 | 0·055 | 0·110 | 0·184 |
| 0·3 | 0·019 | — | — | 0·184 | 0·019 | — | — | 0·184 |
| 0·4 | 0·019 | — | — | 0·184 | 0·019 | — | — | 0·185 |

TABLE V
Influence of pH solution on the polarization resistance, $R_p$, value for ultrafiltration without stirring at $\Delta P = 0.2$ MPa ($R_p \cdot 10^{-13}$, m$^{-1}$). $C(SiO_2)$ in kg m$^{-3}$

| pH | $C(SiO_2)$, $d_p = 8.5$ nm | | pH | $C(SiO_2)$, $d_p = 41.0$ nm | |
|---|---|---|---|---|---|
| | 10·0 | 106 | | 10·0 | 106 |
| 2·95 | 0·75 | — | 2·60 | 1·15 | — |
| 5·50 | 0·79 | — | 8·50 | 0·70 | — |
| 9·64 | 1·00 | — | 2·35 | — | 21·0 |
| 2·45 | — | 49·1 | 5·70 | — | 20·0 |
| 5·85 | — | 39·0 | 8·50 | — | 13·8 |
| 9·64 | — | 26·8 | | | |

sequence of the increase of the gel mass, but of its specific resistance. At the same $SiO_2$ concentration, with the rise of pressure, the rate of gel formation rises too, but the time of ultrafiltration is reduced. The result of this is that the deposited mass of gel is approximately the same as the one that is obtained at a lower pressure, where gel formation is slower, and ultrafiltration lasts longer.

The increase of polarization resistance, influenced by an increase of $SiO_2$ concentration at a constant pressure, is the result of the increase of the gel mass and of its specific resistance. The size of particle does not influence the mass of gel that has been formed, but it does influence its porosity, and, consequently, its specific resistance. Because of a smaller gel porosity for colloidal solutions of $SiO_2$ with smaller

TABLE VI

Influence of pH solution on the specific resistance, $a$, value for ultrafiltration without stirring at $\Delta P = 0\cdot2$ MPa ($a \cdot 10^{-14}$, m kg$^{-1}$). $C(SiO_2)$ in kg m$^{-3}$

| pH | $C(SiO_2)$, $d_p = 8\cdot5$ nm | | pH | $C(SiO_2)$, $d_p = 41\cdot0$ nm | |
|---|---|---|---|---|---|
| | 10·0 | 106 | | 10·0 | 106 |
| 2·95 | 4·05 | — | 2·60 | 5·96 | — |
| 5·50 | 4·48 | — | 8·50 | 3·67 | — |
| 9·64 | 6·24 | — | 2·15 | — | 11·4 |
| 2·45 | — | 26·7 | 5·70 | — | 11·0 |
| 5·85 | — | 21·3 | 8·50 | — | 7·51 |
| 9·64 | — | 14·6 | | | |

TABLE VII

Influence of pH solution on gel formation in the case of ultrafiltration without stirring at $\Delta P = 0\cdot2$ MPa ($m_p/A_m$, kg m$^{-2}$). $C(SiO_2)$ in kg m$^{-3}$

| pH | $C(SiO_2)$, $d_p = 8\cdot5$ nm | | pH | $C(SiO_2)$, $d_p = 41\cdot0$ nm | |
|---|---|---|---|---|---|
| | 10·0 | 106 | | 10·0 | 106 |
| 2·95 | 0·019 | — | 2·60 | 0·019 | — |
| 5·50 | 0·018 | — | 8·50 | 0·019 | — |
| 9·46 | 0·016 | — | 2·35 | — | 0·184 |
| 2·45 | — | 0·184 | 5·70 | — | 0·182 |
| 5·85 | — | 0·183 | 8·50 | — | 0·184 |
| 9·64 | — | 0·184 | | | |

particles, specific resistance, i.e. polarization resistance, is greater, while the flux value, i.e. the filtration rate value, is consequently the smaller.

Results from Table V indicate that the polarization resistance increases with the decrease of the pH value. This dependence is more pronounced in larger contents of the disperse phase $(SiO_2)$. This is in accordance with the dependence of the surface charge densities in the function of the pH solution[8]. At lower pH values there occurs a stronger interaction between $SiO_2$ particles, because of the lower surface charge values.

An analogous dependence was obtained in the course of investigations of the influence of pH on the specific resistance, $a$, value (Table VI). The dependence given can also be explained with the help of the surface charge of $SiO_2$ particles.

Results from Table VII indicate that the solution pH, at the same $SiO_2$ concentration and the pressure, has no influence on the deposited mass of gel. The explanation is similar to the one given in Tables II—IV.

On the basis of results obtained by means of ultrafiltration with stirring, and literature data[6], it was established that, due to the characteristics of the membrane used, the pressure threshold was not reached in the course of the experiments. Therefore, the threshold pressure for our experimental conditions is certainly higher than 0·4 MPa. Such a high threshold pressure value is a consequence of poor porosity of the membrane used. Fane[2], who performed experiments with colloidal $SiO_2$ and amorphous $SiO_2$ (diatomaceous earth) in the same concentration are a and at the same stirring speed, but using a membrane of a 50 times greater porosity (produced by Amicon, type PM 30), obtained a threshold pressure below 0·1 MPa.

So in our case, there occured no gel formation, and stationary state, during which the filtrate flux does not depend on pressure, was not established. Therefore, Eq. (7), i.e. Eq. (8), cannot be applied for data obtained with stirring involved.

It was demonstrated that, in the case of unstirring ultrafiltration, the filtrate flux increases with the increase of the particle size (for greater $d_p$, $\varepsilon$ is greater, and $R_p$ are smaller, so that the flux is greater). When ultrafiltration with stirring is concerned, it was established on the basis of data obtained that the particle size does not influence the flux value. This can be explained by the fact that, in the course of stirring, there occurred no gel formation. A certain increase of the flux, depending on particle size, was observed only for greatest concentrations of colloidal $SiO_2$ (106 kg m$^{-3}$).

**LIST OF SYMBOLS**

| | |
|---|---|
| $a$ | specific resistance, m kg$^{-1}$ |
| $A_m$ | membrane area, m$^2$ |

| | |
|---|---|
| $C$ | colloidal $SiO_2$ concentration, $kg\ m^{-3}$ |
| $C_b$ | $SiO_2$ concentration in bulk, $kg\ m^{-3}$ |
| $C_g$ | $SiO_2$ concentration on the gel surface, $kg\ m^{-3}$ |
| $d_p$ | mean particle diameter, m |
| $D$ | diffusion coefficient, $m^2\ s^{-1}$ |
| $J$ | flux, $m^3\ m^{-2}\ s^{-1}$ |
| $J_m$ | mean flux, $m^3\ m^{-2}\ s^{-1}$ |
| $K$ | coefficient of mass transfer, $m\ s^{-1}$ |
| $m_p$ | mass of particles in gel layer, kg |
| $\Delta P$ | pressure drop, Pa |
| $\Delta P_g$ | pressure drop through gel layer, Pa |
| $R_m$ | membrane resistance, $m^{-1}$ |
| $R_p$ | polarization resistance, $m^{-1}$ |
| $t$ | time, s |
| $V$ | volume of permeate, $m^3$ |
| $\delta$ | layer thickness, m |
| $\delta_g$ | gel layer thickness, m |
| $\varepsilon$ | porosity (fraction voids) |
| $\varrho$ | particle density, $kg\ m^{-3}$ |
| $\eta$ | permeate dynamic viscosity, Pa s |

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