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Optimization of inline coagulation in integrated membrane systems: A study of $FeCl_3$

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ABSTRACT

This research investigated the effect of different process conditions (pH, *G*, *Gt* and coagulant dose) on inline coagulation pre-treatment of ultrafiltration (UF) with $FeCl_3$. Delft canal water was coagulated with $FeCl_3$ and filtered through a 0.1 μm PVDF filter in an Amicon unstirred cell. Filtration was performed in dead-end mode at a constant pressure of 0.1 MPa and at 20°C. Membrane fouling was quantified by calculating the modified fouling index (MFI) of the coagulated solutions. For coagulation with $FeCl_3$, results showed that at higher coagulant dose (>10 mg Fe^{3+}/L), the effect of flocculation time on MFI becomes negligible such that MFI is almost constant for 15 mg Fe^{3+}/L for flocculation times ranging from 0 to 60 min. It was also observed that a relatively high dose (5 mg Fe^{3+}/l) and long flocculation time (60 min) can compensate for the adverse effect of low pH on MFI. While proper flocculation can be designed and achieved prior to membrane filtration, *Gt* and retention times within the pipes and membrane modules can affect floc size and structure. Calculations for a treatment plant revealed *G* values of approximately 4000 s^{-1} in the pipes (Ø 0.075 m) at a flow rate of 33 m^3/h and more than 500 s^{-1} in the UF hollow fibres (Ø 0.0008 m) at a flux of 80 l/m^2.h. From the results it may be concluded that process conditions are inter-related, such that careful selection of one parameter could compensate for the inadequacy of the others. Consequently, an important consideration for maintaining low coagulant doses for in-line coagulation is proper optimization of pH and flocculation time.

Keywords: Inline coagulation; Modified fouling index; Process conditions

1. Introduction

High chemical consumption is frequently encountered in integrated membrane systems (IMS) to ensure smooth operation and low fouling rates of the processes. The chemicals used for cleaning (CEB and CIP), pre-treatment (coagulants and flocculants) and antiscalants not only complicate the process and increase operational costs, but are also an environmental hazard. Consequently, minimizing chemical consumption while maintaining low fouling rates is an important consideration in such systems.

Inline coagulation pretreatment of ultrafiltration (UF) is frequently applied to overcome fouling in UF membranes and to reduce biofouling in subsequent RO membranes through the removal of DOC and phosphate.

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Inline coagulation is the injection of coagulant in the feed stream of a membrane process without removal of the coagulated solids by settling, flotation, etc. In order to maintain low fouling rates at low coagulant dose, process conditions must be optimized. The most relevant process conditions for inline coagulation with metal salts are pH, dose, velocity gradient (*G*), shear rate (*Gt*) and temperature.

pH affects the surface charge of colloids and determines the predominant coagulant species [1]. Therefore floc size and structure (porosity, density) may differ as a function of pH [2]. Flocs formed at lower pH and dose are reported to be denser and less porous than those formed at higher pH and dose [3]. Literature reports that for conventional coagulation schemes (coagulation/flocculation–settling process), optimal coagulation conditions should be a pH higher than 6, and a coagulant dose between 30 and 100ppm $FeCl_3$ [2]. Similar information on coagulant dose for inline coagulation with membrane applications was not found.

The physical properties of flocs are sensitive to flocculation conditions such as *G* and *Gt*. Increased shear reduces the average steady-state size of flocs [4]. Higher *G* values lead to larger fractal dimension, which is related to floc structure [5]. Increasing flocculation time results in loose flocs with low fractal dimension that impose less resistance to filtration [6].

An important consideration in inline coagulation applications is the fate of flocculated particles in the pipe network of a treatment plant and the membrane modules. It is of interest to consider whether flow regimes in the pipes and modules favour floc growth, or break the flocs. Studies on turbulent pipe flow for particle destabilization and aggregation show that for Reynolds number between 8,000 and 16,000, reaction rate for particle aggregation increased [7]. Beyond 16,000 however, reaction rate decreased with increasing Reynolds number. This effect may be attributed to a reduced collision efficiency of the primary particles and/or disruption of microflocs if the turbulence intensity in the pipe reactor exceeds a certain critical value. Reports on flocculation experiments in pipes of various diameters (8–600 mm) show that under steady-state conditions, a decreased floc size is observed with an increasing flow velocity [8].

This study aimed at elaborating the effect of pH, dose and flocculation time on floc filterability with $FeCl_3$ at a constant temperature of 20°C. Floc filterability was quantified using the Modified Fouling Index (MFI). In addition, an attempt was made to calculate *G* and *Gt* values imposed on flocs in pipes and UF modules in a treatment plant, to provide insight on the extent of shear and residence time that flocs are exposed to throughout a treatment scheme.

2. Materials and methods

2.1. Source water

Delft Westvest canal water prefiltered through a 122 μm sieve was used for the experiments. The average feed water characteristics of the canal water were pH of 7.98; TOC of 18.44 mg/l; DOC of 16.03 mg/l; TSS of 12.62 mg/l; and Fe of 0.28 mg/l. When preparing a test series uncoagulated prefiltered canal water was taken from the batch container stored at 4°C and allowed to adjust to room temperature (20°C). Before the sample was taken, the container was thoroughly stirred in order to avoid heterogeneity in suspended matter content for different tests.

2.2. Jar test experiments

A calibrated jar test unit equipped with a continuously adjustable speed regulator was used for the experiments. The test unit consisted of 6 containers with 4 baffles in each container. A flat-bladed stainless steel turbine impeller was used for shear induction. The rotation applied to the samples was measured by a digital contact tachometer, (Extech Instruments, Model 461891), which was directly applied to the axis propelling the blades of the stirrer.

Very short and relatively long flocculation times were employed to study the effect of flocculation time on floc filterability. Rapid mixing intensity was set to a constant value of 470 s^{-1} for 20–30 s, followed by flocculation at an intensity of 35 s^{-1} for durations of 2, 10, 30, 40 and 60 min.

The coagulant stock solution was prepared freshly for each test series in a concentration of 1 mg Fe^{3+}/l. Feed water pH was adjusted prior to dispersion of coagulant in the feed water. 1M HCl and 1M NaOH were used for pH adjustment.

2.3. Selection of independent process variables for coagulation

Values for *G* and *Gt* were selected by the traditional approach to coagulation, and by considering values cited by other studies performed under similar conditions. Camp (1955) suggested *G* values from 20 to 74 s^{-1} for flocculation and a total *Gt* of 20,000–200,000. The US Environmental Protection Agency recommends a mixing time of at least one minute and a mixing intensity of 300 up to 1000 s^{-1} to obtain instantaneous, uniform dispersion of the coagulant in raw water [9]. Several sources cite similar *G* and *t* values for mixing and flocculation as the ones used in this study [6,10].

Coagulant dose was chosen to cover a wide spectrum, from underdosing to relative overdosing (0.5–15 mg Fe^{3+}/l). pH values were selected to simulate naturally occurring pH ranges (6–8), thus limiting chemical use for pH adjustment.

2.4. Filtration experiments

After completion of the targeted flocculation time, the coagulated solution was immediately transferred to the filtration setup (Fig. 1), using a plastic ladle. The transfer was done with utmost care, avoiding disturbance of flocs as much as possible. For all tests an unstirred cell (Millipore Series 8000, model 8200) was used. The cell could take up a feed volume of up to 200 ml, with an effective membrane area of 27.8 cm^2. The magnetic stirring mechanism, available to control concentration polarization or accumulation of macromolecules on the membrane surface, was not used for the test runs (unstirred cell). The TMP was kept constant at 0.1 MPa by means of a manually adjustable pressure sustaining valve. Filtration was done with 0.1 μm PVDF filters (Millipore, USA). Filtrate was collected in a beaker placed on a digital scale (Mettler Toledo, Model PB 602-S). The scale had an RS 232 interface with a computer. Data sets of filtrate weight collected over time were recorded and imported into an MS Excel spread sheet by data acquisition software (WinWedge, www.taltech.com). The sampling frequency was set to 2 or 4 s. For each test run the membrane was flushed with 1 l of deionised water with a resistivity of 18.2 MΩ.cm at a TMP of 0.1 MPa. Before filtration of the coagulated solution, most of the remaining deionised water from the flushing procedure was removed by means of an air flush.

2.5. Computational methods

The resistance in series model is often employed to describe filtration resistance in a UF process.

$$J=\frac{\Delta P}{\eta R_t}=\frac{\Delta P}{\eta\left(R_m+R_b+R_c\right)} \tag{1}$$

where J is the permeate water flux (l/m^{2}h), ΔP is the transmembrane pressure difference (Pa), η is the water viscosity (Pa.s), R_t is the total filtration resistance (1/m), R_m is the membrane resistance (1/m), R_b is the blocking resistance (1/m) and R_c is cake resistance (1/m). R_c is defined by Ruth [11], using the concept of "specific cake resistance" per unit weight 'α', as follows.

$$R_c=\frac{V}{A}\times\alpha.C_b \tag{2}$$

Combining Eqs. (1) and (2) results in the well known filtration equation.

$$\frac{\mathrm{d}t}{\mathrm{d}V}=\frac{\eta R_m}{\Delta P.A}+\frac{\eta\alpha C_b}{\Delta P.A^2}V \tag{3}$$

where dt is the differential filtration time (s), dV is the differential filtration volume (m^3), A is the effective filtration area (m^2), α is the specific cake resistance per unit weight of deposited particles (m/kg), C_b is the concentration of particulate matter (kg/m^3), and V is the cumulative filtered volume (m^3). When cake filtration is the predominant filtration mechanism, the slope of the filtration curve obtained from Eq. (3) is regarded as an index for predicting the fouling potential of a feed water, modified fouling index (MFI) [12], as given in Eq. (4).

$$\mathrm{MFI}=\frac{\eta\alpha C_b}{\Delta P.A^2} \tag{4}$$

MFI is proportional with 'α' and 'C_b'. As determining C_b was particularly not easy in this study and therefore α could not be calculated, MFI was considered as the indicator to investigate the effect of coagulation conditions on floc filterability.

According to the Carmen–Kozeny [13] definition of 'α' [Eq. (5)], a reduction in particle size and/or cake porosity increases the specific cake resistance and consequently the MFI.

$$\alpha=\frac{180(1-\varepsilon)}{\rho_p d_p^2\varepsilon^3} \quad \text{(Eq. 5)}$$

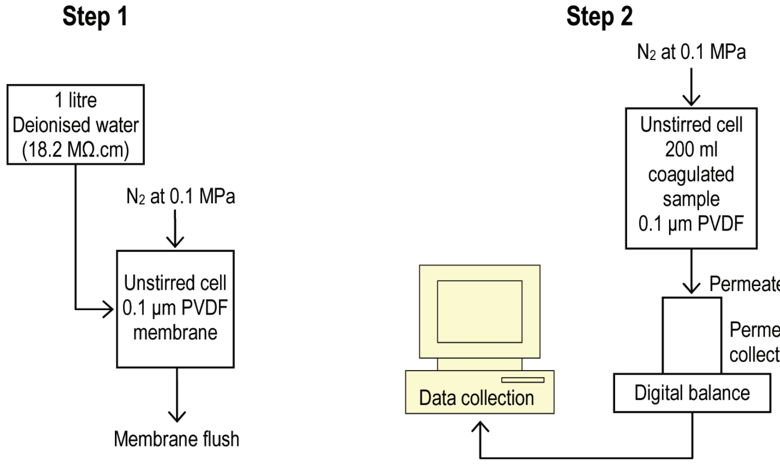

Fig. 1. Schematic representation of the filtration setup.

For the calculation of G values in a pipe or a hollow fibre of an ultrafiltration membrane element, pressure drop between the two ends of the pipe or fibre must be known. This may be determined by the Hagen–Poiseuille law for the calculation of voluminal laminar stationary flow of incompressible uniform viscous liquids through a cylindrical tube with constant circular cross-section.

$$\Delta P = \frac{128\,\eta L Q}{\pi D^4} \tag{6}$$

where ΔP is the head loss between two ends of a pipe (Pa), η is the viscosity of the fluid (Pa.s), L is the length of the pipe (m), Q is the flow (m^3/s), and D is the pipe diameter (m). Consequently, G can be calculated as follows.

$$G = \left(\frac{E}{\eta V}\right)^{1/2} = \left(\frac{Q\Delta P}{\eta V}\right)^{1/2} = \left(\frac{Q\dfrac{128\eta L Q}{\pi D^4}}{\eta V}\right)^{1/2} = \left(\frac{128 Q L}{\pi D^4 t}\right)^{1/2} \tag{7}$$

3. Results and discussion

3.1. *Effect of dose and* Gt *on MFI at constant pH for coagulation with FeCl*$^{3+}$

The influence of different flocculation times (ranging from 0 to 60 min), and coagulant dose (ranging from 0 to 15 mg Fe^{3+}/l) on filterability of prefiltered canal water was assessed. Feed water pH was adjusted prior to coagulation to achieve a final value of 8. Tests were performed at ambient temperature (21°C), and a TMP of 0.1 MPa. Details of mixing and flocculation conditions for the experiments are presented in Table 1.

Fig. 2 shows the impact of coagulant dose on MFI for different Gt values. A coagulant dose of 1 mg Fe^{3+}/l resulted in a minimum MFI for all Gt values, ranging from 589 s/l^2 at Gt 9400 to 2280 s/l^2 at Gt 140,100.

Reducing the coagulant dose to 0.5 mg Fe^{3+}/l resulted in an increase in MFI, regardless of the induced flocculation time. At a very low coagulant dose (e.g. 0.5 mg Fe^{3+}/l), floc formation may be inhibited, resulting in the formation of very small flocs. Smaller diameter flocs form cakes with higher specific resistance (α) which leads to higher MFI values [Eqs. (4) and (5)].

Table 1
Mixing and flocculation conditions for coagulation with FeCl$_3$, at pH 8 and 21°C

Rapid mixing			Flocculation			Gt total
G (s^{-1})	t (s)	Gt RM	G (s^{-1})	t (s)	Gt SM	
470	20	9400	0	0	0	9400
470	30	14100	35	120	4200	18300
470	30	14100	35	660	23100	37200
470	30	14100	35	1800	63000	77100
470	30	14100	35	2400	84000	98100
470	30	14100	35	3600	126000	140100

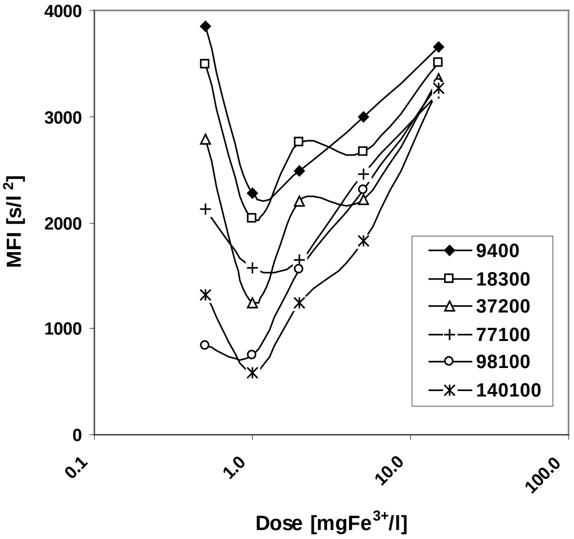

Fig. 2. MFI as a function of coagulant dose (mg Fe^{3+}/l) and total Gt at pH 8 and constant temperature (21°C).

Above a coagulant dose of 1 mg Fe^{3+}/l, MFI increased with increasing coagulant dose. For some Gt values a local maximum at 2 mg Fe^{3+}/l was observed (Gt 18300 and 37200), and for others increase in MFI was linear up to the maximum dose of 15 mg Fe^{3+}/l. One possible explanation for the increase in MFI with increase in dose is, since MFI is proportional to particle concentration 'C_b', an increase in coagulant dose (particle concentration) would result in an increase in MFI. Another possible explanation could be that next to the agglomeration of dissolved and suspended solids, precipitation of metal ions in the membrane may occur, which may add to the membrane resistance [14].

Results show that MFI generally decreased as Gt increased. The highest MFI values were recorded for filtration of solutions prepared with no flocculation, i.e. Gt 9400, and the lowest values were observed for flocculation periods of 60 min, i.e. Gt 140100. Prolonged flocculation time allows for more floc formation and growth. Consequently, flocs formed at Gt 140100 are probably larger than those formed at Gt 9400 and will form a less resistant cake.

From Fig. 2 it was also observed that the influence of flocculation time on MFI is greatest at 0.5 mg Fe^{3+}/l and decreased with increasing dose. For 0.5 mg Fe^{3+}/l, MFI dropped by 66% as Gt increased from 9400 to 140100. At 15 mg Fe^{3+}/l, the drop in MFI between the lowest and highest Gt values reduced to a mere 10%. The large variation in MFI values at 0.5 mg Fe^{3+}/l was attributed to the difference in flocculation times. At such coagulant dose, where flocculation kinetics is most likely retarded, shorter flocculation times (0–2 min) may result in smaller flocs and a less permeable cake (as indicated by higher MFI values of 3500–3900 s/l^2), whereas longer flocculation times, e.g. 60 min, assist floc formation and growth, ultimately producing larger flocs and low MFI (1320 s/l^2). The smaller variation in MFI values as a function of *Gt* at 15 mg Fe^{3+}/l may be attributed to faster flocculation kinetics due to the higher coagulant concentration.

At coagulant dose of 10 mg Fe^{3+}/l and higher, increasing the flocculation time beyond 2 min did not result in a considerable reduction in MFI (3650 s/l^2 at 2 min flocculation time as opposed to 3260 s/l^2 at 60 min flocculation time). Consequently, proper selection and induction of *Gt* is very critical at low dose (0.5 mg Fe^{3+}/l) and less critical at higher coagulant dose (≥10 mg Fe^{3+}/l).

3.2. *Effect of flocculation time and coagulant dose on MFI at different pH values*

The influence of 3 flocculation times (0, 30 and 60 min) combined with varying coagulant dose (0, 1 and 5 mg Fe^{3+}/l) on MFI was assessed at different pH values. Feed water pH was adjusted prior to coagulation in order to obtain a final pH of 6, 7, and 8. The tests were performed at ambient temperature (21°C) and a TMP of 0.1 MPa. Details of mixing and flocculation are given in Table 2.

Table 2
Mixing and flocculation details for experiments at different pH values

Rapid mixing			Flocculation			*Gt* total
G (s^{-1})	*t* (s)	*Gt* RM	*G* (s^{-1})	*t* (s)	*Gt* SM	
470	30	14100	0	0	0	14100
470	30	14100	35	1800	63000	77100
470	30	14100	35	3600	126000	140100

MFI values obtained from the filtration tests are presented in Fig. 3 as a function of total *Gt* and coagulant dose, grouped for different pH values.

Results showed that increasing pH from 6 to 7 resulted in a drastic reduction in MFI values. Further increment in pH from 7 to 8, reduced the MFI only marginally. Coagulated canal water at pH 8 showed slightly lower MFI (e.g. for a *Gt* of 140100, 464 s/l^2 and 1490 s/l^2 at a dose of 1 and 5 mg Fe^{3+}/l, respectively) compared to pH 7 (for *Gt* 140100, 471 s/l^2 and 2000 s/l^2 at a dose of 1 and 5 mg Fe^{3+}/l, respectively).

At pH 8, MFI increased by 474% and 180% when the imposed *Gt* was decreased from 140,100 to 14,100 for a coagulant dose of 1 and 5 mg Fe^{3+}/l, respectively. As explained previously, to increase the *Gt* from 14,100 to 140,100, the flocculation time was increased from 0 to 3600 s, probably resulting in larger flocs and a more permeable filter cake, and thus a lower MFI.

At pH 7 and 8, MFI increased as the coagulant dose was increased from 0 to 5 mg Fe^{3+}/l. Increasing coagulant dose from 0 to 1 mg Fe^{3+}/l at pH 8 and a *Gt* of 77,100 doubled the MFI. For increase in dose from 1 to 5 mg Fe^{3+}/l, MFI increased by 75%.

A rather marginal increase in MFI (112%) occurred when the pH decreased from 8 to 7 for an imposed *Gt* of 77,000 at a coagulant dose of 1 mg Fe^{3+}/l. A similar trend was observed for a *Gt* value of 140,100 — the increase in MFI was even more marginal (101%) when pH decreased from 8 to 7. The gap gradually widens at 5 mg Fe^{3+}/l for a change in *Gt* from 77,000 to 140,000 — the increase in MFI was 125% and 134%, respectively.

At pH 6, MFI decreased with increase in dose. Increase in coagulant concentration improves the kinetics of floc formation and growth and consequently larger flocs may form, imparting less resistance to filtration.

3.3. *Fate of flocculated particles in a full-scale treatment plant*

The objective of this section is to highlight the importance of continuous flocculation throughout the entire

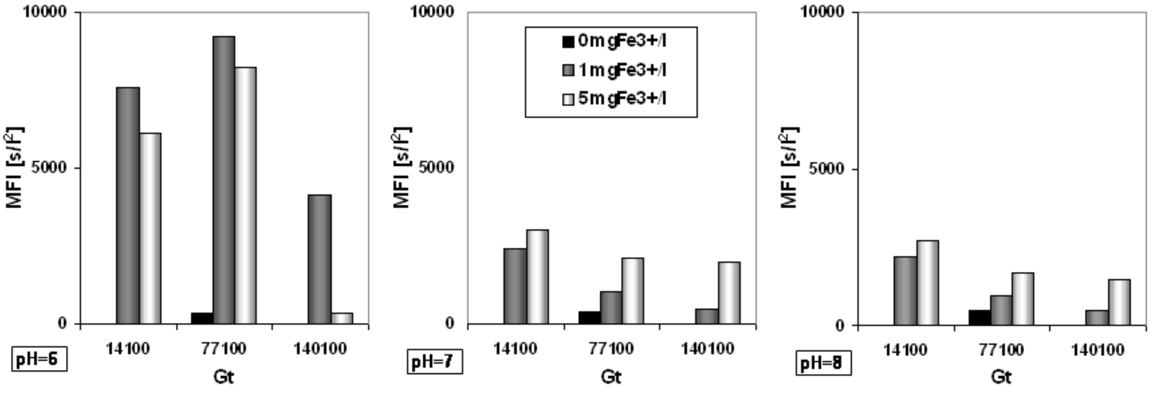

Fig. 3. MFI as a function of total *Gt* and coagulant dose at different pH values.

treatment process and its possible effect on floc size and structure. It is important to realize that regardless of how optimum the flocs are created, their exposure to *G* and *Gt* values in pumps, pipes, valves and even within the hollow fibres of a membrane module may break the flocs or affect their structure. To elaborate this concept, calculations are made for *G* and *Gt* values within the pipe network and hollow fibres of an arbitrary treatment plant considering the scheme presented in Fig. 4.

The UF system is comprised of three skids in parallel which operate in dead end mode; each skid accommodates 12 elements (6×2). Considering a membrane area of 35 m^2 per element, the total filtration area of each skid will be 420 m^2. Therefore, at a permeate flux of 80 $l/m^2.h$, the flow to each skid will be 33 m^3/h. With reference to this flow rate, velocities in different pipe sections in the system were calculated and the results are presented in Table 3.

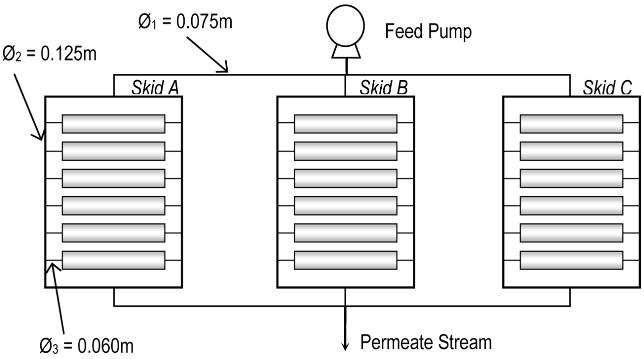

Fig. 4. Scheme of the UF system in an arbitrary full-scale plant.

Table 3
Velocity and Reynolds number in the pipe network of a treatment plant

Pipe	Diameter, Ø (m)	Velocity (m/s)	Reynolds No.
1	0.075	2	155000
2	0.125	0.75	93000
3	0.060	3	193400

Calculations revealed turbulent flow regimes for all pipe diameters. *G* values in the pipes [Eq. (7)] were at about 4000 s^{-1}, which is approximately 8 times higher than the values proposed in literature [15] for microfloc formation and 40 times higher than those proposed for particle aggregation into macroflocs. Overall retention time up to the first module in Skid A was about 15 s.

For a single hollow fibre fed from both ends, flow rate, velocity and *G* values were calculated at a flux of 80 l/m^2h. The hollow fibre was split into 20 arbitrary sections of equal length (0.075 m), for better elaboration of *G* and *Gt* development along the fibre length (Fig. 5).

For each arbitrary section of the fibre, the mean flow and head loss were calculated. Reduction in flow is based on the assumption that permeation is constant in all sections. Based on head loss, *G* was calculated for each section. Results are illustrated in Fig. 6.

Calculated average velocities were around 0.08 m/s at

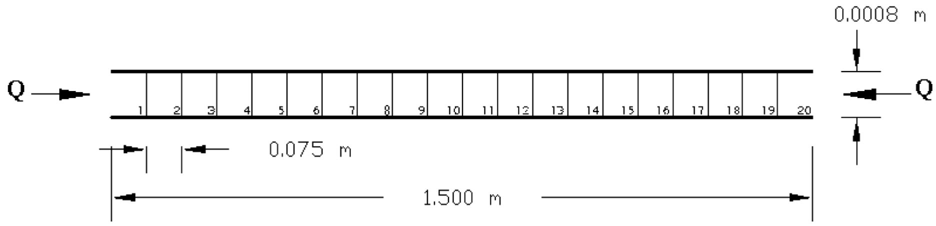

Fig. 5. Longitudinal section of a UF hollow fibre.

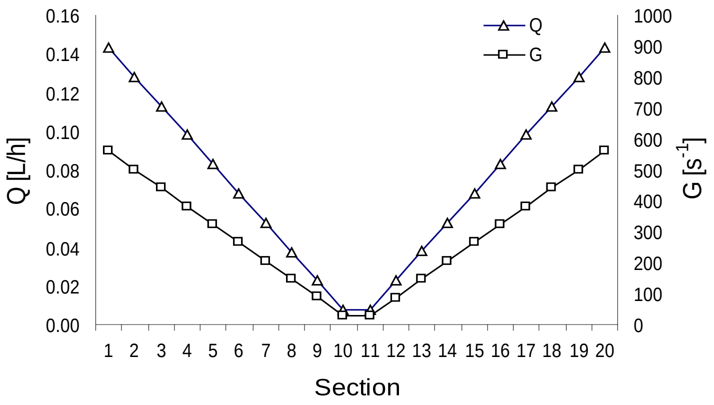

Fig. 6. Change in flow and *G* along a UF hollow fibre.

the fibre entrance and approached zero at the centre of the fibre. Calculations showed that *G* value decreased proportionally with decreasing flow along the fibre length; such that *G* was maximum at the fibre entrance at about 560 s^{-1} and this value decreased along the fibre, till it reached its minimum at 30 s^{-1} (fibre midsection). Residence time in the fibre is about 18 s just before mid-span.

G values in the first few sections of the fibre could be high enough to break the flocs and it is unlikely that they can re-form due to the very short residence time in the fibre.

Further calculations showed that flocs entering the fibres can be exposed to a total *Gt* of about 5500 by the time they reach fibre mid-section.

4. Conclusions

The effect of flocculation time on floc filterability is lessened with increasing dose. At higher coagulant dose (>10 mg Fe^{3+}/l), increasing flocculation time from 0 to 60 min did not have a pronounced effect on the filterability of ferric flocs. At low coagulant dose of 0.5 mg Fe^{3+}/l, however, increase in flocculation time improved filterability drastically.

MFI was very high for low pH (i.e. 6) and decreased with increasing pH (i.e. 8). A relatively high dose (5 mg Fe^{3+}/l) and long flocculation time (60 min) can compensate for the adverse effect of low pH on MFI.

From the results it may be concluded that process conditions for coagulation are interrelated such that proper selection of one parameter could compensate for the inadequacy of others.

Calculations for *G* and *Gt* values in an arbitrary treatment plant revealed that at a typical flux of 80 l/m^2.h, flocs are exposed to high *G* values in the pipe network (4000 s^{-1}) and in the hollow fibres of UF membranes (560 s^{-1}). Such high *G* values can affect floc size and structure, and should be taken into account while creating flocs.

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