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Optimizing inline coagulation to reduce chemical consumption in MF/UF systems

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ABSTRACT

Microfiltration/ultrafiltration (MF/UF) systems preceded by inline coagulation pretreatment are characterized by a high consumption of chemicals. For chemical consumption to be reduced, process conditions for inline coagulation (dose, pH, mixing, flocculation and temperature) should be optimized. In order to establish the optimum conditions for inline coagulation with different types of coagulants, their behaviour under different coagulation conditions needs to be understood. This study aimed at elaborating the behaviour of polyaluminium chloride (PACl) under different conditions of dose, pH, mixing (G) and flocculation (Gt) in the absence of turbidity and with low organic matter content.

Tap water, filtered through a 0.1 µm PVDF filter was used as model water. Flocculated solutions were filtered in dead-end mode using an Amicon unstirred cell device. The specific cake resistance was quantified by measuring the modified fouling index (MFI) at a constant pressure of 1 bar and at 20°C.

For low coagulant dose (1–5 mg Al^{3+}/L) and low shear rate (<10 min flocculation time), pH control appeared to be the most important factor. At 5 mg Al^{3+}/L, prolonged flocculation times (>30 min) resulted in lower MFI and higher pH values appeared to compensate for insufficient flocculation. In the presence of coagulant, the predominant filtration mechanism was cake filtration and pore blocking was not observed. The contribution of aluminium flocs to pressure increase in a UF membrane module, was not significant at dosing levels of 1–5 mg Al^{3+}/L, and ranged from 1% to 5% depending on dose, pH and mixing conditions.

Keywords: Inline coagulation; Specific cake resistance; Modified fouling index

1. Introduction

Microfiltration (MF) and ultrafiltration (UF) were originally introduced as chemical-free alternatives to conventional treatment schemes (coagulation, sedimentation, rapid sand filtration), in which product water quality is independent of the raw water quality. However, in order to ensure that MF/UF are cost effective alternatives to conventional treatment, relatively high flux values of approximately 80–120 L/m^2 h need to be maintained in pressurized systems. At such flux values, permeability can decrease rapidly, and as a result, various pretreatment methods such as inline coagulation (with metal salts) are frequently applied to enhance MF/UF performance [1–4]. Inline coagulation is a process whereby a coagulant is added to the

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Table 1
Mixing time and intensity for different shear rates

	Rapid mixing			Flocculation			
Exp. Set	G [s^{-1}]	t [s]	Gt RM	G [s^{-1}]	t [s]	Gt SM	Gt total
I	320	30	9600	50	65	3250	12,850
II	470	30	14,100	40	2100	84,000	98,100

feed stream, but the coagulated solids are not removed by sedimentation, flotation, etc. [5], and feed water reaching the membranes consists of colloidal and particulate matter and precipitated hydroxides of the salt.

One of the main drawbacks of coagulation pretreatment is the consumption of chemicals, which not only increase the operational costs but also the complexity of the process due to the need for sludge concentration, treatment and disposal [6]. When inline coagulation is not optimized a higher than necessary coagulant dose is required to achieve a stable operation which results in higher costs and environmental impacts. To minimize chemical consumption (coagulant dose), process conditions need to be optimized. It is well known that pH is a determining factor when the kinetics of flocculation (collision-agglomeration efficiency) is of concern, as it affects both the predominance of various hydrolysis species of aluminium and the surface charge of colloids, and consequently the predominating coagulation mechanism [7–9]. Camp (1955) introduced velocity gradient (G-value) and shear rate (Gt) and their effect on coagulation efficiency. He defined optimum ranges for both values in which the most suitable flocs for settling can be produced. In membrane pretreatment however, where the objective is to produce flocs with acceptable specific resistance and flocs that do not give rise to pore blocking, no guidelines for mixing and shear rates are available. Coagulant dose is another important process condition, as the volume fraction of particles (Ω) affects collision efficiency and the rate of agglomeration [2,7]. It is well known that coagulation is retarded at lower temperatures and hence temperature is of crucial importance for coagulation with hydrolyzing metal salts [10,11].

This study aimed at elaborating the behaviour of polyaluminium chloride (PACl) under different conditions of dose, pH, G and Gt in the absence of turbidity and with low organic matter content at a constant temperature of 20°C. PACl was chosen because it demonstrates a higher rate of flocculation than iron and aluminium salts [10], and is frequently used in inline coagulation applications. Quantification of floc filterability in terms of specific cake resistance and identification of different fouling mechanisms provides insight into the influence of inline coagulation with PACl on MF/UF performance.

Although residual foulants, that are not removed by backwashing or chemical cleaning, can significantly deteriorate MF/UF process performance [12], however the focus of this study was to assess the performance of UF with inline coagulation during a single filtration cycle with a clean membrane before backwashing. This was done to provide a preliminary investigation of the behaviour of flocculated aluminium particles in combination with membrane filtration. For a more concise analysis of the effect of process conditions on the performance of inline coagulation with membrane filtration, multiple filtration cycles with backwashing and chemical cleaning should be considered.

2. Materials and methods

2.1. Feed water

In order to get a clear understanding of the behaviour of aluminium flocs produced under different process conditions, raw water should be free of particles that may affect the structure and characteristics of flocs, e.g. suspended and colloidal particles and NOM. To this end, coagulation should ideally be carried out with ultra pure water (MilliQ water). However, from an experimental point of view (pH adjustment) it was more convenient to use tap water. Therefore, Delft tap water was used as source for all experiments conducted in this study. The average feed water characteristics of the source used for filtration experiments were: pH of 8.1; temperature 21°C; alkalinity 116 mg HCO_3/L; ultra violet adsorption (UVA_{254}) 2.7 m^{-1}; turbidity <1 NTU and dissolved organic carbon (DOC) 1.9 mg/L.

2.2. Jar test experiments

Jar tests were conducted with a calibrated jar test unit for the preparation of flocculated solutions under different process conditions. PACl (AquaRhône® 18D) was used as coagulant. Coagulant doses of 1, 2 and 5 mg Al^{3+}/L were used which correspond to 11.1, 22.2 and 55.6 mg/L as PACl dose respectively.

Flocculation was carried out in two different modes to study the effect of shear rate on floc formation and the characteristics of aluminium flocs. In the first mode only rapid mixing was undertaken for a very short time (10 s) at an intensity of 470 s^{-1} ($\sim$208 rpm), in an attempt to simulate inline coagulation (absence of flocculation). In the second mode rapid mixing was applied for a period of 30 s at the same intensity as the

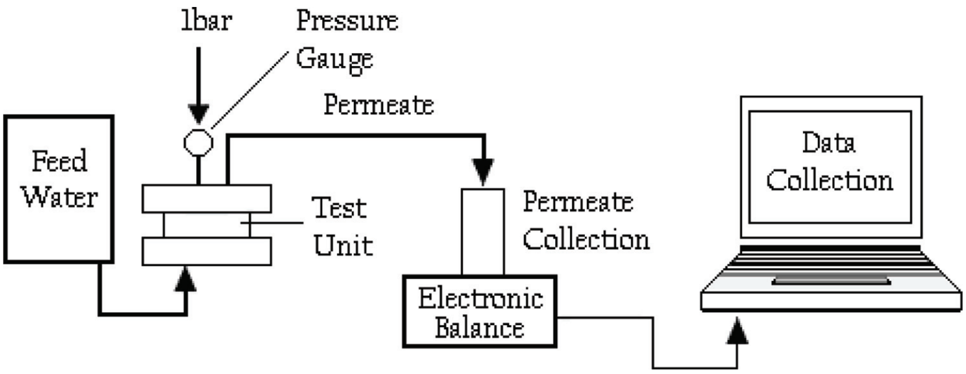

Fig. 1. Scheme of the filtration set-up.

first mode, followed by slow mixing for the purpose of flocculation. The latter set was exposed to longer flocculation times (up to an hour), in order to investigate the effect of prolonged flocculation time on floc filterability.

Temperature was recorded for each jar test and values ranged between 20°C and 22°C. Variation in temperature during flocculation was negligible. pH was adjusted to values between 5.5 to ambient (7.4–7.7 depending on dose) by the addition of 1 M HCl and 1 M NaOH.

2.3. Filtration experiments

Filtration tests were conducted using an unstirred cell device schematically presented in Figure 1. Batch experiments were performed using Amicon cells (8200) with a maximum process volume of 200 mL. The stirring assembly was removed from the cell to allow for cake formation. Filtration was achieved under dead-end, constant pressure mode. Flocculated suspensions prepared under different process conditions were transferred to the cell unit at the end of each flocculation time. The solution was filtered through a hydrophilic PVDF filter with a pore size of 0.1 μm (Millipore, USA) under $\triangle P = 100$ kPa (1 bar). Clean water flux was approximately 1450 L/m^2 h.

Permeate from the cell was collected in a beaker set on an electronic balance (Mettler Toledo, Model PB 602-S). Data sets of collected filtrate weight and filtration time were recorded.

2.4. Analytical methods

Permeate samples were collected, preserved, stored and tested per Standard Methods for the Examination of Water and Wastewater (19th ed.). All permeate samples were analyzed with Eriochrome Cyanine-R dye in accordance with APHA 3500 – Al D, using a spectrophotometer (Perkin Elmer, Lambda 20) for absorption at 535 nm. Minimum aluminium concentration detectable by this method in the absence of fluorides and complex phosphates is approximately 6 μg/L.

2.5. Calculation of specific cake resistance

Floc filterability was quantified using the concept of specific cake resistance per unit weight 'α'. Specific cake resistance is constant for incompressible cakes under constant pressure filtration and can be calculated according to the Carmen-Kozeny relationship (Eq. (1)) [13,14]. According to this relationship a decrease in particle diameter size (d_p) increases the specific resistance of the deposited cake.

$$\alpha = \frac{180(1-\varepsilon)}{\rho_p d_p^2 \varepsilon^3} \tag{1}$$

where
α = specific cake resistance of particles per unit weight (m/kg)
ε = cake porosity (%)
ρ_p = average density of deposited particles (kg/m^3)
d_p = average diameter of deposited particles (m)

Specific resistance can also be calculated from the modified fouling index (MFI). The MFI$_{0.45}$ was defined by Schippers and Verdouw [15] as an index of the fouling potential of a feed water containing particles when fixed reference values are used for pressure ($\triangle P = 2$ bar), viscosity ($\eta = \eta_{20°\mathrm{C}}$) and area ($A = 13.8{*}10^{-4}$ m^2), with a 0.45 μm membrane. MFI$_{0.45}$ is expressed as the gradient of the straight portion of the plot of t/V versus volume, as follows:

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

where

η = viscosity of water (Pa s)

α = specific cake resistance (m/kg)

C_b = particle concentration (aluminium dose in this case) (kg/m^3)

$\triangle P$ = transmembrane pressure (Pa)

A = membrane surface area (m^2)

Alternatively, MFI is calculated through Eq. (2b) when values of $\triangle t/\triangle V$ are plotted against volume.

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

For filtration at constant pressure, $\triangle t/\triangle V$ corresponds to instantaneous resistance. In this study plots of $\triangle t/\triangle V$ versus volume were used for higher accuracy, and consequently MFI was calculated through Eq. (2b).

MFI-UF [16], a slightly modified version of the MFI$_{0.45}$, was used to measure MFI with UF membranes. MFI-UF is corrected to the standard reference conditions through Eq. (3) and is therefore directly comparable with the MFI$_{0.45}$.

$$\mathrm{MFI}-\mathrm{UF} = \frac{\eta_{20^\circ C}}{\eta_T}\left(\frac{\Delta P}{\Delta P_o}\right)\left(\frac{A}{A_o}\right)^2\left(\frac{\Delta\left(\frac{\Delta t}{\Delta V}\right)}{\Delta V}\right) \tag{3}$$

For the calculation of specific cake resistance, C_b is considered as the amount of particles retained by the membrane. For this reason, Al^{3+} residual in permeate samples were measured to correct C_b for aluminium passage.

Combining the fundamental equation for flow through a porous medium (Darcy and Poiseuille) and the concept of specific cake resistance per unit weight (Ruth), the following relation is established for estimating pressure build-up in a UF module.

$$\Delta P = \eta R_m J + \eta\cdot\alpha\cdot C_b\cdot J^2\cdot t \tag{4}$$

where

$\triangle P$ = transmembrane pressure in a UF module (Pa)

η = viscosity of water (Pa s)

R_m = membrane resistance (m^{-1})

J = flux (m^3/m^2 s)

α = specific cake resistance (m/kg)

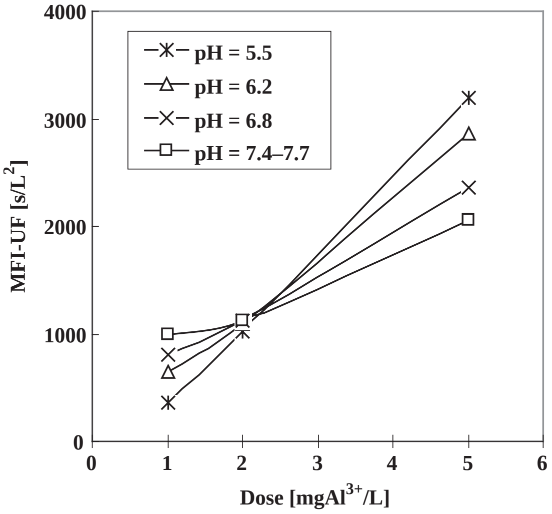

Fig. 2. MFI as a function of dose and pH at a constant shear rate of 12,850 (1 min flocculation time).

C_b = aluminium dose (kg/m^3)

t = filtration time (s)

3. Results and discussion

3.1. *Fouling potential of feed water at different dose and pH at a constant shear rate*

Fouling potential of feed water coagulated at ambient and suppressed pH values and coagulant dose ranging from 1 to 5 ppm as Al^{3+} was evaluated. Rapid mixing was performed at an intensity of 320 s^{-1} for 30 s followed by flocculation at an intensity of 50 s^{-1} for 65 s, bringing the total shear rate to 12,850.

Figure 2 demonstrates the effect of pH on floc filterability at a constant shear rate (at a very low flocculation time of approximately 1 min) for different doses of aluminium.

At a coagulant dose of 1 mg Al^{3+}/L, at a short flocculation time of 1 min, experimental results showed an increase in MFI from 354 s/L^2 at pH 5.5 to 994 s/L^2 at pH 7.7 (ambient). In the absence of colloids and organic matter, and at a low coagulant dose of 1 mg Al^{3+}/L, volume fraction of particles (Ω) is low and therefore rate of agglomeration is retarded. At lower pH, where preformed polymers in PACl hydrolyze and/or agglomerate more slowly [9], a significant fraction of the flocs remain small in diameter and will pass through a 0.1 μm membrane. This effect may be enhanced by the high flux (1450 L/m^2 h) in the filtration experiments. As pH increases, rate of particle formation increases, hence more flocs are formed and consequently retained by the membrane. These flocs

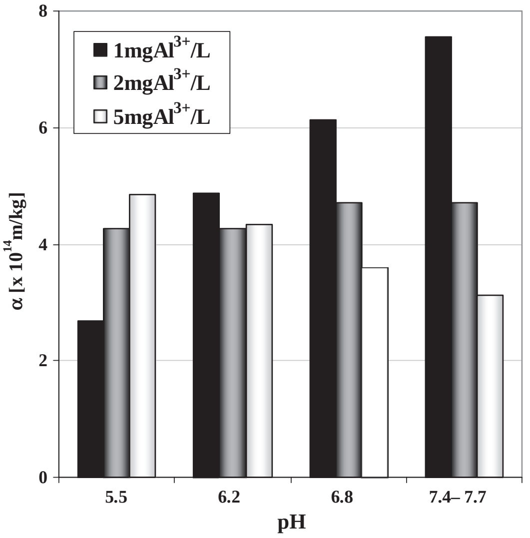

Fig. 3. Specific resistance of aluminium flocs at different dose and pH values at a total shear rate of 12,850 (1 min flocculation time).

would impart a higher resistance to filtration, therefore an increase in MFI is observed.

At a coagulant dose of 2 mg Al^{3+}/L, the change in resistance with increasing pH was negligibly small. This could mean that under these conditions, flocculation kinetics is dominated by dose (volume fraction of particles), such that an increase in pH does not have a significant impact on the rate of floc formation.

At 5 mg Al^{3+}/L, increase in pH resulted in a decrease in MFI values. MFI dropped from 3192 s/L^2 to 2056 s/L^2 as pH increased from 5.5 to 7.4 (ambient). At higher coagulant dose, more preformed polymers are available, therefore more collisions occur and more flocs are formed. Increasing the pH, increases the rate of precipitate formation (more hydroxyl ions), which in the presence of sufficient particles would allow for the formation of larger flocs that would impart less resistance to filtration.

The specific cake resistance 'α' was calculated for different coagulant doses. Results are presented in Figure 3.

For pH values ranging from 6.2 to ambient (7.4–7.7), specific resistance was highest at 1 mg Al^{3+}/L and decreased with increase in dose. At low coagulant dose, where Ω (volume fraction of particles) is small and rate of agglomeration is low, smaller particles are formed. A smaller particle diameter (d_p), results in a higher specific cake resistance (refer Eq. (1)). As the coagulant dose is increased, more collisions occur and floc formation and growth is enhanced. Hence specific resistance of the deposited cake at 5 mg Al^{3+}/L is less than that of 1 mg Al^{3+}/L.

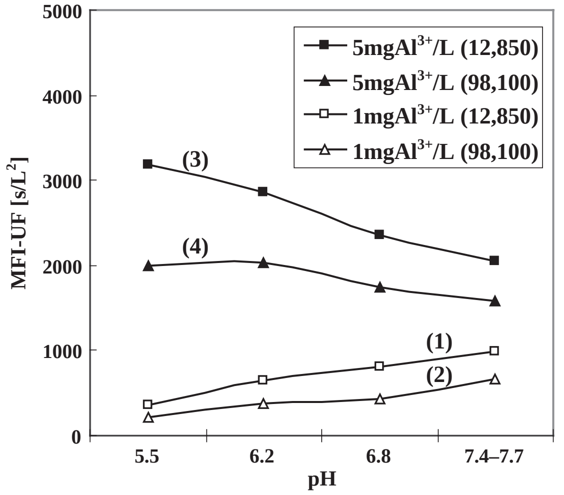

Fig. 4. Effect of increase in shear rate from 12,850 to 98,100 (1–35 min flocculation time) at different pH values for a coagulant dose of 1 and 5 mg Al^{3+}/L.

At pH 5.5 and low coagulant dose (1 mg Al^{3+}/L), a significant fraction of the particles are too small to be retained by a 0.1 μm membrane. Increase in dose compensates for the adverse effect of low pH on floc formation. Therefore at pH 5.5, an increase in specific cake resistance with increase in dose was observed.

3.2. *Effect of increased shear rate at different pH values for coagulant dose of 1 and 5 mg Al^{3+}/L*

Experiments were performed to compare the effect of a low (Gt 12,850, 1 min flocculation) and high shear rate (Gt 98,100, 35 min flocculation) on floc filterability at different pH values for coagulation at 1 and 5 mg Al^{3+}/L. Details of the mixing conditions are presented in Figure 4. Results are presented in Fig. 4.

At coagulant dose of 1 mg Al^{3+}/L, irrespective of the applied shear rate, MFI increased with increase in pH (Curves 1 and 2). The similar increasing trend observed in both curves indicates that at a low dose of 1 mg Al^{3+}/L, pH is the main controller of flocculation kinetics and increase in shear rate does not dampen the predominant effect of pH on floc formation.

Increasing the flocculation time from 1 (Curve 1) to 35 min (Curve 2), resulted in a reduction in MFI for all pH values. It was expected that a higher flocculation time would allow for particle growth and subsequent retention by the membrane, thereby imparting a higher resistance to filtration. However, a consistent decrease was observed for all pH values. Reduction in MFI was higher for the two intermediate pH values, i.e. 6.2 and 6.8, where aluminium solubility is low.

For filtration at 5 mg Al^{3+}/L and 1 min flocculation time (Curve 3) increase in pH from 5.5 to 7.4 resulted in

Table 2
Mixing time and intensities tested to assess the effect of prolonged flocculation on fouling

	Rapid mixing			Flocculation			
Exp. Set	G [s^{-1}]	t [s]	Gt RM	G [s^{-1}]	t [s]	Gt SM	Gt total
I	470	10	4700	0	0	0	4700
II	470	30	14,100	40	578	23,100	37,200
	470	30	14,100	40	2100	84,000	98,100
	470	30	14,100	40	3150	126,000	140,100

a significant reduction in MFI, i.e. 36%. For the same dose at 35 min flocculation time (Curve 4) the difference in MFI between the two pH extremes dropped to 21%, indicating that longer flocculation times can dampen the predominant effect of pH on flocculation kinetics by allowing for more floc formation.

At 5 mg Al^{3+}/L, when flocculation time was increased from 1 to 35 min, a reduction in MFI was observed at all pH values. The difference was greater at lower pH values (38% at pH 5.5) and gradually decreased with increasing pH (23% at pH 7.4).

With reference to Eq. (2), for similar experimental conditions, MFI at 5 mg Al^{3+}/L is expected to be five times higher than that of 1 mg Al^{3+}/L, as it is directly proportional to particle concentration 'C_b'. However, experimental results showed a nine-time increase in MFI as dose increased from 1 to 5 mg Al^{3+}/L at pH 5.5. This is attributed to the difference in flocculation kinetics at different doses. As pH increased, the difference in MFI between the two doses became smaller, such that at ambient pH (7.4–7.7), MFI was doubled as dose increased from 1 to 5 mg Al^{3+}/L.

3.3. Effect of pH and shear rate on fouling at a constant dose of 5 mg Al^{3+}/L

The effect of shear rate on floc formation and growth at a constant dose (5 mg Al^{3+}/L) was investigated. The range of mixing time and intensities (rapid mixing and flocculation), used for this set of experiments are given in Table 2. pH values varied from 5.5 to 7.4 (ambient).

Experimental results (Figure 5) revealed that in the absence of flocculation (total shear rate of 4700), an increase in pH from 5.5 to 7.4 resulted in a 53% decrease in MFI; from 3725 s/L^2 to 1737 s/L^2. As shear rate increased from 37,200 (10 min flocculation) to 140,100 (60 min flocculation), difference in resistance between the two pH extremes dropped from 21% to 15%. This shows that higher flocculation periods can dampen the effect of pH on the rate of floc formation. When flocculation time is longer, floc growth continues and larger flocs are produced. Cakes deposited from large flocs have low specific resistance and consequently low MFI.

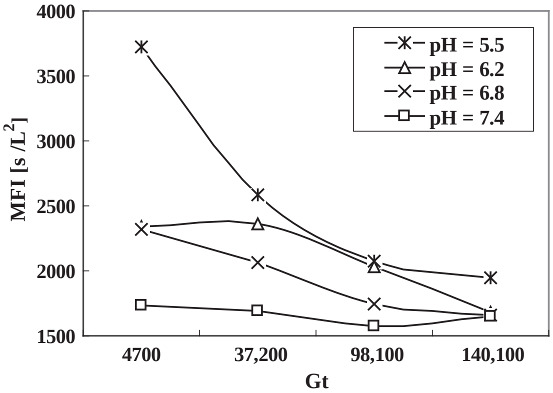

Fig. 5. Effect of pH and shear rate on MFI at 5 mg Al^{3+}/L.

At pH 5.5, increase in flocculation time from 0 to 60 min, resulted in a 48% reduction in MFI. When pH was increased to 7.4, the difference in MFI from 0 to 60 min flocculation time dropped to 5%. This indicates that at higher pH values, the effect of flocculation time on floc growth and filterability becomes negligible.

3.4. Measurement of aluminium residual in permeate samples

Figure 6 illustrates the results of Al^{3+} measurement in permeate samples for feed water coagulated at 5 mg Al^{3+}/L at different shear rates and pH values. At pH 5.5 and 7.4, where solubility is high, a higher passage was observed. Aluminium residual at pH 6.2 and 6.8, where solubility is less, were consistently lower than the two pH extremes.

At pH 5.5, increase in flocculation time, resulted in a consistent decrease in aluminium passage. Aluminium

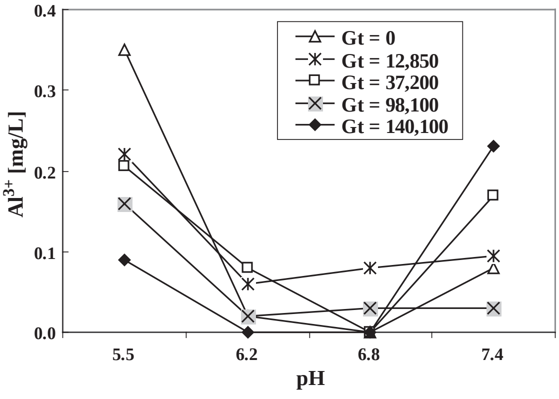

Fig. 6. Aluminium residual as a function of pH at coagulant dose of 5 mg Al^{3+}/L for different shear rates.

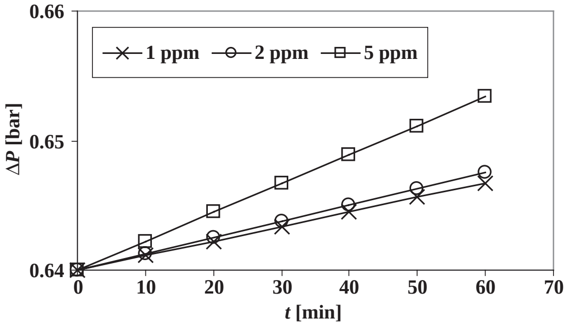

Fig. 7. TMP increase at $J = 80$ L/m^2 h for coagulant dose of 1, 2 and 5 mg Al^{3+}/L, at 20°C, total shear rate of 12,850, at ambient pH (7.4–7.7).

residual decreased from 0.35 mg/L at no flocculation to approximately 0.1 mg/L at 60 min flocculation. At pH 7.4, an opposite trend was observed; longer flocculation times resulted in a higher passage of aluminium. It may be that aluminium flocs formed at higher pH are weak and can rupture when exposed to high shear rates and pass through 0.1 μm pores. At lower pH values particles are dense, less porous [17] and consequently less apt to break up.

Maximum aluminium passage is 0.35 mg/L and occurs at pH 5.5 when no flocculation is applied. This amounts to about 7% of the aluminium dose, i.e. 5 mg Al^{3+}/L.

3.5. Predicting pressure increase in a UF module

For fluxes of 80 and 100 L/m^2 h and 'α' obtained under different process conditions, pressure build-up in a UF module was calculated according to Eq. (4). Results are illustrated in Figure 7 and Figure 8.

In Figure 7, 5 mg/L of Al^{3+} gives the highest increase in pressure for 60 min filtration time. The increase in pressure is about 2% for 5 mg Al^{3+}/L, and about 1% for 1 and 2 mg Al^{3+}/L. At a flux of 100 L/m^2 h, coagulant dose of 5 mg Al^{3+}/L and no flocculation (Figure 8), filtration for 60 min results in a 5% increase in TMP at pH 5.5, 3% at pH 6.2 and 6.8 and 2% at pH 7.4. Similar calculations for results obtained from other conditions of pH and flocculation time, revealed TMP increments in the same range or lower. It may therefore be concluded that the contribution of aluminium flocs to pressure build-up in a UF module is marginal. The narrow range is attributed to the low organic matter, low turbidity and experimental temperature of 20°C. At lower temperatures the effects are expected to be more pronounced.

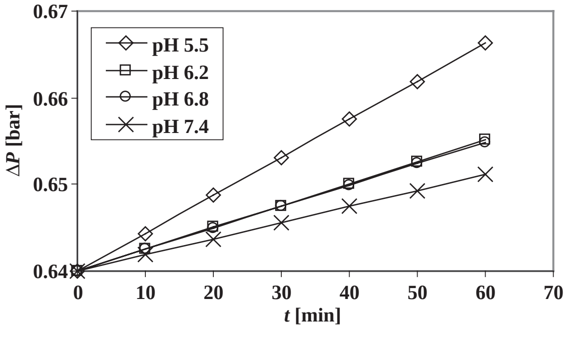

Fig. 8. TMP increase at $J = 100$ L/m^2 h for different pH values at coagulant dose of 5 mg Al^{3+}/L, 20°C and no flocculation.

3.6. Filtration mechanisms

Figure 9 shows results obtained from filtration of (a) tap water at pH 6.8 and (b) coagulated tap water at 5 mg Al^{3+}/L, pH 6.8 and a total shear rate of 12,850. Pore blocking was observed for filtration with tap water without coagulant (Fig. 9a). This trend was observed at all pH values. MFI values obtained from filtration with tap water were constant at approximately 91 s/L^2 for the tested pH range.

Results obtained from the filtration of coagulated solutions were assessed for pore blocking by (a) visual inspection of the filtration curves ($\triangle t/\triangle V$ versus V curves), (b) re-calculation of initial membrane resistance from filtration results and (c) evaluation of the slope of $\triangle t/\triangle V$ versus V graphs. Pore blocking was not detectable by any of the mentioned approaches. It cannot be excluded that due to the high flux, the time span of the phenomenon was too short to be recorded at the start of the filtration run.

4. Conclusions

For low coagulant dose (1–5 mg Al^{3+}/L) and low shear rate (<10 min flocculation time), pH control appeared to be the most important factor.

At coagulant dose of 5 mg Al^{3+}/L, in the absence of flocculation, increase in pH resulted in a drastic reduction in MFI. Increase in flocculation time (up to 60 min) dampened the predominant effect of pH on floc formation. At coagulant dose of 1 mg Al^{3+}/L, flocculation kinetics was retarded. Increase in pH allowed for more floc formation at this low dose, but an increase in flocculation time did not improve flocculation kinetics.

Specific cake resistance of aluminium flocs decreases (but not proportionally) with increase in dose. From TMP predictions in an actual UF module, it may be concluded that aluminium flocs impart little resistance to flow and contribute a small fraction to increase in pressure. The predominant filtration

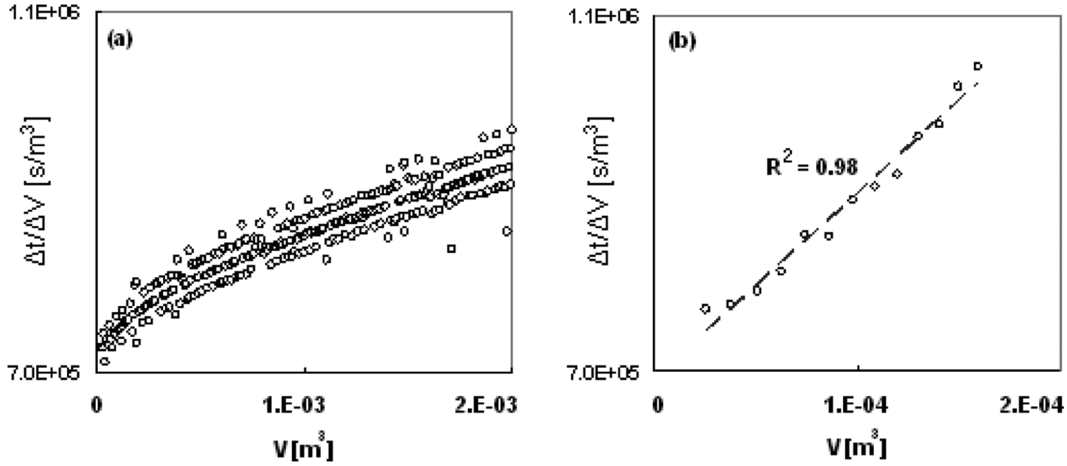

Fig. 9. Filtration curves for (a) tap water and (b) coagulated tap water at 5 mg Al^{3+}/L, pH 6.8 and shear rate of 12,850.

mechanism for solutions prepared under different coagulation conditions was cake filtration and no pore blocking was observed.

List of symbols

α	specific cake resistance of particles per unit weight (m/kg)
ε	cake porosity (%)
ρ_p	average density of deposited particles (kg/m^3)
d_p	average diameter of deposited particles (m)
η	viscosity of water (Pa s)
C_b	particle concentration (aluminium dose in this case) (kg/m^3)
$\triangle P$	transmembrane pressure (Pa)
A	membrane surface area (m^2)
R_m	membrane resistance (m^{-1})
J	flux (m^3/m^2 s)
t	filtration time (s)
Ω	volume fraction of particles during flocculation ($m^3/\ m_0^3.m_0^3$)

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