Desalination and Water Treatment 8 (2009) 24–30
www.deswater.com

1944-3994/1944-3986

Removal of phosphorus from secondary effluents by coagulation and ultrafiltration

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Received 28 October 2008; accepted 2 July 2009

ABSTRACT

Secondary effluents of municipal wastewater in Israel contain on average 10 mg/L phosphorus, a concentration that is twice as high as a limit recently legislated by the Israeli Ministry of Environment. Reduction of phosphorus concentration to the required level is often performed by biological methods, or by flocculation followed by sand filtration. The current study explores a different path of coagulation with $FeCl_3$ followed by ultrafiltration. The results suggest a general applicability of the proposed treatment with a relatively significant phosphorus removal percentage of 54% achieved by a combination of 120 ppm of ferric chloride and polysulfone membranes with a molecular weight cut-off of 20 kDa. As the removal levels should be increased and concentration of the coagulant decreased, continuous optimization is warranted.

Keywords: Pretreatment; Ferric chloride; Alum; Ultrafiltration membrane; Phosphorus removal

1. Introduction

Many municipal wastewater treatment plants are required to remove phosphorus (P), in order to prevent eutrophication of receiving surface water bodies [1]. Typical concentration of phosphorus in secondary effluents lies between 5 and 10 mg/L when current Israeli legislation requires no more than 5 mg/L for unrestricted irrigation and no more than 1 mg/L for discharge into sensitive water sources [2]. Between 30% and 50% of phosphorus in wastewater originates from natural sources in a form of orthophosphates and polyphosphates, and the rest – 50–70% – comes from synthetic detergents. Phosphorus-containing detergents, such as sodium tripolyphosphate or sodium/potassium phosphates, have been used for a long period of time due to low toxicity, germicidal effect and surfactant activity helping to peptize and suspend particulate matter [3]. As up to 70% of phosphorus is provided by detergents, the first and simplest idea was to reduce or to even restrict usage of phosphorus-containing detergents. Although popular some time ago, the idea was not implemented to the levels that could result in any significant reduction of phosphorus concentration. The non-phosphorus detergents were unable to tie up calcium, magnesium, iron and manganese ions, thus leaving the overall washing performance low [4]. With a limited success in the lower release of phosphorus-containing detergents into wastewater streams, reduction of phosphorus from secondary effluents has to be achieved by a dedicated treatment.

In general, removal of phosphorus can be achieved by biological, chemical and physical methods when the biological treatment is the cheapest alternative. The biological treatment can be performed by either

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Presented at the First UK–Israeli Workshop and Research Event on the Application of Membrane Technology in Water Treatment and Desalination, 15–20 June 2008, University of Oxford, UK.

specific microorganisms such as tropical cyanobacterium *Phormidium bohneri* [5] and *Staphylococcus auricularis* [6], or by constructed wetlands with various plants such as *Eichornia crassipes* [7], *Gracilaria chilensis* [8] and *Lemna gibba* [9]. A detailed review of available biological treatments can be found in de-Bashan and Bashan [10]. Despite the fact that conventional activated sludge treatment can reduce effluent total phosphorus (TP) concentrations to 1–2 mg/L, in practice many of the synthetic detergents are not biodegradable and therefore physical–chemical treatment methods prevail.

One of the most successful and widely implemented methods to reduce concentration of phosphorus is by coagulation/flocculation, followed by filtration. Rybicki [11] found that addition of aluminum and ferric salts followed by filtration can cause up to 95% reduction in phosphorus concentration. It is generally acceptable to differentiate between two steps in the removal of phosphorus by aluminum sulfate (alum). In the first stage, alum quickly dissociates and reacts with phosphorus to create aluminum phosphate precipitates [12]:

$$Al^3+ + H_2PO_4^- + 2OH^- \rightarrow Al(H_2PO_4)(OH)_{2(s)} \quad (1)$$

$$Al^3+ + HPO_4^{2-} + OH^- \rightarrow Al(HPO_4)(OH)_{(s)} \quad (2)$$

The second stage is the formation of aluminum hydroxide flocs by deprotonation of the aqua aluminum ion usually formed with the introduction of aluminum salt in water [13]:

$$Al^3 + 3H_2O \rightarrow Al(OH)_{3(s)} + 3H^+ \quad (3)$$

The solid amorphous hydrous aluminum oxide is able to incorporate aluminum phosphate precipitates into its structure via coprecipitation or adsorption onto the surface. The adsorption capacity of aluminum hydroxide flocs to phosphorus is high [14,15], and the formed structure can be removed by sedimentation [16] or granular filtration [17]. Retention of phosphorus can be mitigated by changes in pH of the entire solution, alum dose, mixing time and speed.

The mechanism of phosphorus removal with iron salts is similar to that previously described with alum. Addition of ferric chloride to wastewater results in the formation of a number of precipitates such as $FePO_{4(s)}$ and $Fe(OH)_{2(s)}$ formed at acidic pH [18]. Additional species such as $FeHPO_4^+$, $Fe_{2.5}PO_4(OH)_{4.5(s)}$ and $Fe_{1.6}H_2PO_4(OH)_{3.8(s)}$ were observed at alkali pH later on [19]. The optimal pH for phosphorus removal was reported to be between 4 and 5 [20]. Removal of phosphorus was reported to vary as a function of stoichiometric Fe:P ratio from 63% achieved at Fe:P = 1:1 molar ratio to 100% with Fe:P = 2.55:1 [21]. Clark and Stephenson [22] reported that 88% removal of total phosphorous was obtained at a Fe:P 3:1 ratio at pH 7.4. Additional factors such as length of flocculation period and speed of mixing had only a minor effect on phosphorus removal [23].

The formed flocs are usually removed by either sedimentation or by granular filtration. Both operations can achieve excellent results. However, they demand a large footprint not always available for wastewater treatment plants. One of the elegant solutions is the introduction of a membrane filtration step that is more expensive than the former options but has an additional disinfection advantage [24]. One of the major obstacles in tertiary membrane treatment of municipal effluents is fouling that might result in a severe flux drop, frequent cleanings and a significant increase in operational costs [25–28]. Considering potential fouling, the coagulant concentration should be optimized to form either small dense or spacious voluminous flocs that will not interrupt the membrane filtration process [29]. The optimization can be performed in terms of changes of significant adjustable parameters such as coagulant dose and membrane molecular weight cut-off. Changes in pH that are also important are rarely considered due to possible complications with increased salinity of groundwater, imperviousness of soils, etc. Hence the question of optimal balance between maximum phosphorous rejection by chemical precipitation and minimal membrane fouling is of great interest.

Assuming that economical affordability of the membrane treatment plays an important role in the entire application, ultrafiltration (UF) and microfiltration (MF) membranes with low energy demands have an advantage. Intuitively, UF membranes should perform better due to a smaller molecular weight cut-off (MWCO). However, the size of typical flocs might be comparable with the size of membrane pore entries [30], thus suggesting optimization of the entire application by shifting to the MF arena. The performance of MF and UF membranes treating secondary effluent has been evaluated in many studies but there is no general agreement as to which is better. The cases of better UF [31], better MF [32] and equal importance were reported. The present research was aimed at reducing the concentration of phosphorus and minimizing membrane fouling by a combination of ferric chloride coagulation–UF process. Successful application of the proposed technology for a combination of aluminium salts and UF membrane had been reported recently [33,34]. The optimization of the proposed technology in terms of $FeCl_3$ dosage and membrane's MWCO is reported here.

Table 1
Characteristics of raw secondary effluents samples

Parameter	Average value
pH	7.7 ± 0.1
TSS (ppm)	11.4 ± 3.7
TOC (ppm)	13 ± 1.7
BOD (ppm)	8.6 ± 3.5
COD (ppm)	57.5 ± 10
Hardness (ppm)	281.3 ± 19.6
Turbidity (NTU)	5.9 ± 2.1
Alkalinity (ppm)	401.1 ± 25.2

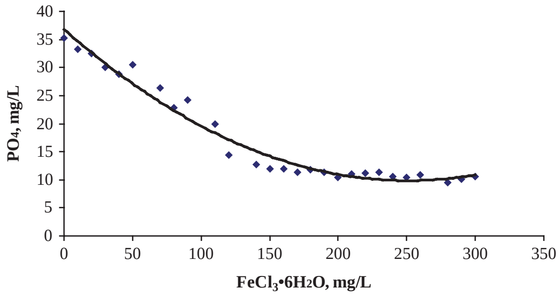

Fig. 1. Phosphate removal by flocculation with ferric chloride.

2. Materials and methods

Secondary effluents were obtained from Ashkelon wastewater treatment plant (in southern Israel). The plant typically treats 20,000 m^3/day of domestic wastewater by the activated sludge process (including mechanical and biological treatment). At present, no tertiary treatment is applied and, by definition, the secondary effluent cannot be used for irrigation of food crops. Samples were collected from the plant on different days during the period from July 2006 to December 2007. During the study period, the average raw wastewater quality was characterized by 400 mg/L biological oxygen demand (BOD) and 420 mg/L total suspended solids (TSS). Samples of the secondary effluent samples were sent to the laboratory at Ben-Gurion University on a weekly basis. Samples were stored at 4 °C until the experiments were conducted. Before each experiment, the temperature of the samples was adjusted to room temperature, i.e. the experiments were performed under isothermal conditions of 21 ± 1 °C. The raw effluent samples were characterized in terms of BOD, COD, TOC, pH, turbidity, temperature, PO_4^{3-}, alkalinity and hardness (Table 1). In terms of regulatory demands, the pH of the secondary effluent (7.3–7.7) fell within the allowed range, and the average values of 10 mg/L BOD and 20 mg/L TSS were lower than those required by current Israeli legislation of 20 and 30 mg/L for BOD and TSS, respectively.

The optimal dosages of ferric chloride ($FeCl_3 \times 6H_2O$, Sigma–Aldrich) were established in jar tests. The following experimental protocol was used: 500 mL of the effluent was dosed with various concentrations (in the range of 10–300 mg/L) of $FeCl_3 \times 6H_2O$, poured into 800-mL beakers, and then treated as follows: (i) mixed rapidly in a conventional multiple stirrer jar (Phipps and Bird 7790-402, Richmond, VA with 76 × 25 mm flat paddle impellers) at 100 rpm for 1 min; (ii) mixed slowly at 30 rpm for 20 min; and (iii) allowed to settle quiescently for 30 min. Samples collected by slow decantation from the upper part of the test jars were analyzed for phosphorus concentration, turbidity, and pH. Formed flocs were analyzed for zeta potential and floc size distribution with a ZetaPlus (Brookhaven Instruments Corporation, Holtsville, NY, USA) analyzer equipped with a 30 mW 657 nm laser (Hamamatsu Photonics K.K., Hamamatsu City, Japan) using (−150)-(+150) mV zeta potential range.

Filtration experiments were performed in a lab-scale 150-mL stirred cell operated in dead-end mode. The detailed description of the cell can be found elsewhere [35]. Three different polysulfone (PS) flat-sheet membranes of 20, 50 and 100 kDa manufactured by Alfa Laval (Copenhagen A/S, Denmark) were investigated. The membranes were supplied as flat sheets and of a size suitable for the membrane filtration cell. Filtration experiments were performed immediately after stages (i) and (ii) to prevent settling following a protocol described elsewhere [30]. Fouling experiments were performed on a pilot scale at 2 bars nitrogen pressure for times sufficient to allow filtration of at least 50 mL of the feed suspension, i.e. secondary effluent coagulated by ferric chloride. UF membrane performance was evaluated in terms of phosphorus removal (effluent quality) and in terms of flux drop (fouling). All fouling experiments were conducted at least twice. The variation between flux data from replicate experiments was found to be within a margin of 10%.

The ascorbic acid method [36] was used for phosphorus analysis. In general, ammonium molybdate and potassium antimonyl tartrate react in acid medium with orthophosphate to form a heteropoly acid (phosphomolybdic acid) that is reduced to an intensely colored molybdenum blue by ascorbic acid. A calibration curve was built using phosphate standard solution (KH_2PO_4, Merck, Germany). The minimum detection limit for this method is 10 μg P/l.

3. Results and discussion

Reduction in phosphate concentration as a function of added ferric chloride is depicted in Fig. 1. The results of jar-tests suggest that increased concentration of ferric chloride results in lower phosphorus residual levels. Along with that, two different removal areas were observed – the level below 120 mg/L of ferric chloride was a region of exponential decrease in phosphorus concentration. For concentrations above 120 up to 300 mg/L, a plateau with almost no additional removal was observed. The level of 120 mg/L in ferric chloride corresponds to a Fe:P ratio of 1.8 that according to previous reports should result in complete removal of phosphorus. However, even an increase of the Fe:P ratio from 1.8 to 5 did not result in complete phosphorus removal. The probable explanation of the observed phenomenon lies in the presence of additional components within the secondary effluents that might either interrupt precipitation or spend the coagulant. Another option could be a pH which was significantly higher than the optimal range. The experiments were performed at pH 7.2, a value that was achieved due to addition of ferric chloride. The main removal mechanism in that pH range is adsorption of phosphorus compounds onto previously formed ferric hydroxides and aggregation of compounds such as $Fe_{2.5}PO_4(OH)_{4.5(s)}$ and $Fe_{1.6}H_2PO_4(OH)_{3.8(s)}$ [19]. The most significant phosphate reduction observed was 73%, observed at Fe:P ratios of 3.3 and above.

The obtained phospho-ferric aggregates were characterized by size and zeta potential. The results are depicted in Fig. 2. Here, the size and zeta potential of particles or flocs were plotted for secondary effluents without treatment, and for secondary effluents treated with 120 and 200 mg/L ferric chloride corresponding to Fe:P ratios of 2.1 and 3.6, respectively. The smallest average particle size of 0.48 mm was observed for untreated effluents. The treatment caused formation of flocs with sizes of 3.69 and 3 mm for 120 and 200 mg/L ferric chloride, respectively. Measurement of zeta potential of secondary effluents showed average values of −22 mV for secondary effluents with no treatment, and levels of 0 and −3.5 mV for 120 and 200 mg/L ferric chloride, respectively. An interesting and not obvious tendency of formation of bigger flocs and the absence of charge was observed at a lower concentration of 120 mg/L ferric chloride, where higher concentration caused lower floc dimension and minor floc charge. The observed can be explained by the formation of immense flocs at higher concentration that were subject to breakage due to continuous mixing. Thus the broken floc has smaller dimensions and minor charge resulting from the presence of charge groups on the floc-solution interface. The zeta potential value below 20 mV has no influence on adsorption to the membrane surface [37].

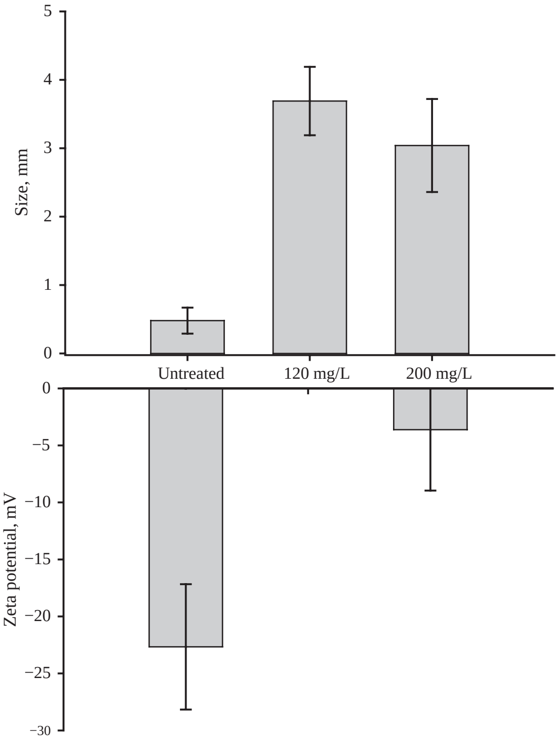

Fig. 2. Changes in size and zeta potential as a function of ferric chloride dose.

The size of 0.48 mm observed for raw particles in secondary effluents is already big enough to be retained by UF membrane without flocculant addition. Fig. 3 depicts phosphorus retention experiments performed for PS membranes with a MWCO of 20, 50 and 100 kDa. A membrane with a MWCO of 20 kDa was able to achieve approximately 24% phosphorus retention, reducing the concentration from 6.7 to 5.1 mg/L. Almost no phosphorus retention by 50 and 100 kDa membranes was observed, suggesting that there are some inorganic phosphorus compounds with a

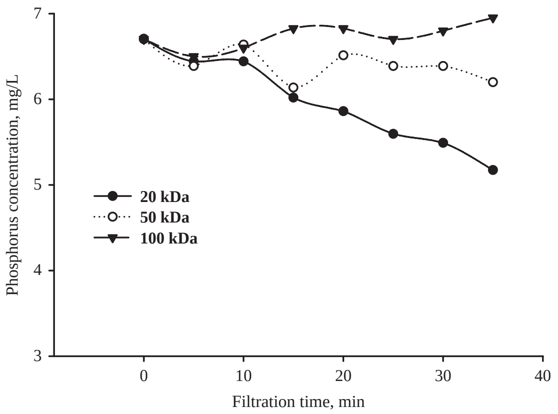

Fig. 3. Removal of phosphorus by UF alone.

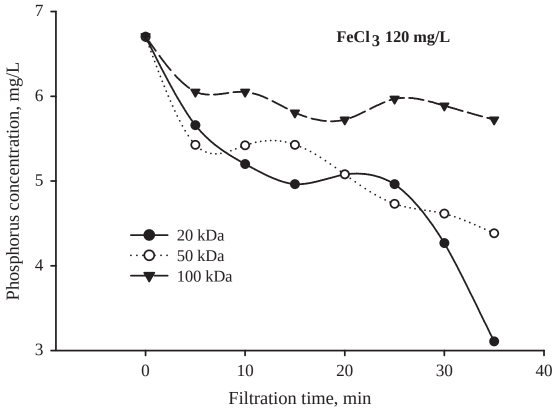

Fig. 4. Removal of phosphorus by a combination of 120 ppm $FeCl_3$ and UF with MWCO of 20, 50 and 100 kDa.

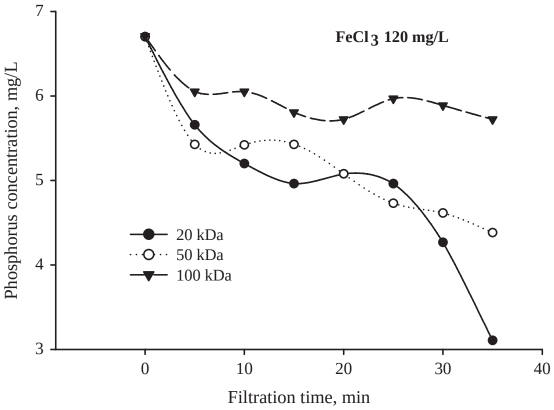

Fig. 5. Removal of phosphorus by a combination of 200 ppm $FeCl_3$ and UF with MWCO of 20, 50 and 100 kDa.

molecular weight between 20 and 50 kDa. At the same time, the hydrodynamic radius of the compounds is on the order of 10 nm, significantly lower than the inorganic particles of 0.48 mm diameter observed by size measurements. Another interesting tendency is a time-dependent character of phosphorus retention. When at the beginning the retention was close to zero, after half an hour of filtration significant retention was observed. That might indicate that there is a cake formed on the top of the membrane, and retention of phosphorus is performed by the cake and not by the membrane itself. Despite a negligible cut-off size difference between the 20 and 50 kDa membrane, insignificant retention on membranes with MWCO of 50 and 100 kDa suggest a high monodispersivity of the particles. A significant flux drop for all membranes was observed (data not shown). Flux reduction was on the order of 30–40% for both 20 and 100 kDa membranes and on the order of 25% for 50 kDa membranes.

Reduction in phosphorus concentration after addition of 120 and 200 mg/L, as a function of filtration time, is depicted in Figs. 4 and 5, respectively. The plots are for the polysulfone membranes with MWCO of 20, 50 and 100 kDa. Better retention was observed for 20 kDa membranes where phosphorus concentration was reduced from 6.7 to 3.1 mg/L, i.e. retention of 54% was received. Membranes with molecular cut-offs of 50 and 100 kDa were able to reduce the phosphorus concentration from 6.7 to 4.4 and 5.7 mg/L, or by 35% and 15%, respectively. A similar trend was observed with the addition of 200 mg/L ferric chloride (Fig. 5). A higher removal level was observed with membranes with a lower MWCO of 20 kDa. Concentration of phosphorus was reduced from 6.7 to 4.4 mg/L level, i.e. by 35%. Reduction to the levels of 5.4 and 5.7 mg/L, i.e. by 20% and 15%, was observed in experiments with 50 and 100 kDa membranes, respectively. Retention of phosphorus with 120 mg/L ferric chloride was significantly better than the retention obtained with 200 mg/L. Although the tendency was solid and was observed with all membrane pore sizes, it is difficult to attribute it to a variation in zeta potential values of the flocs or to a size difference between the flocs formed at two coagulant concentrations.

Flux dependence on filtration time and on concentration of flocculant is depicted in Fig. 6. The plots for raw effluents and effluents treated with 120 and 200 mg/L $FeCl_3$ are depicted for 20 kDa membrane. The highest flux drop of 33% from 600 to 400 L/m^2 h was observed for untreated effluents. Addition of both 120 and 200 mg/L ferric chloride resulted in a flux drop from 600 to 500 L/m^2 h. The fouling was observed in the first 15 min, and followed by a stable filtration with no further flux drop.

The flux drop (over 30 min) of 50 and 100 kDa membranes for effluents treated by $FeCl_3$ and

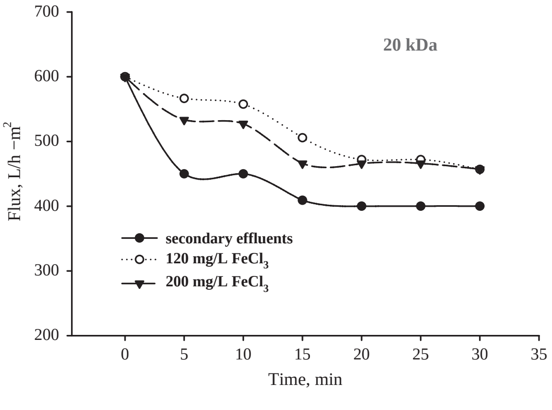

Fig. 6. Variation with time of the permeate flux through PS membrane as a function of pretreatment for a 20-kDa membrane.

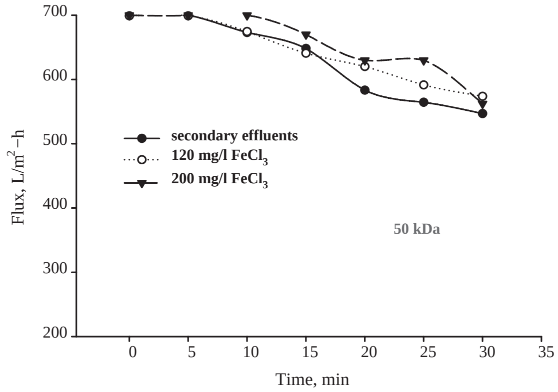

Fig. 7. Variation with time of the permeate flux through PS membrane as a function of pretreatment for a 50-kDa membrane.

untreated secondary effluents are presented in Figs. 7 and 8. The general trend of flux drop as a function of time was observed for all the membranes. As well, the most severe fouling was always observed with untreated secondary effluents. The flux drop reached up to 33%, 37% and 47% for 20, 50 and 100 kDa membranes, respectively. At the same time, when flocculation was applied, membranes with higher MWCO values demonstrated the highest reduction in flux, e.g., if 33% drop in flux was observed for 100 kDa membranes, then only 24% and 20% was observed for 50 and 20 kDa membranes, respectively. This phenomenon can be explained by the fact that the membranes with low and high MWCO have different fouling patterns. In contrast to the membranes with high MWCO, where flux drop is caused by the flocs that enter into the pores and adsorb on pore walls, membranes with low MWCO were protected from long-term fouling by formation of the porous cake on their surface.

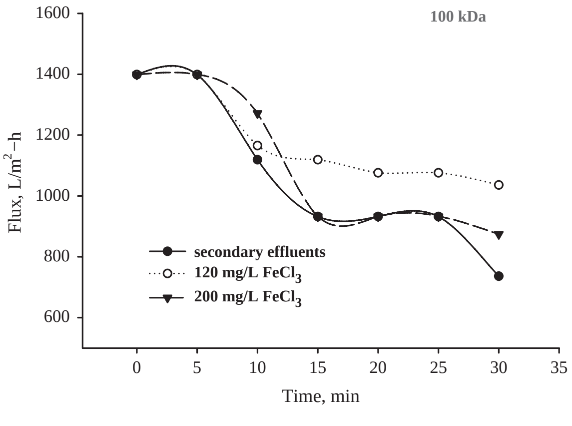

Fig.8. Variation with time of the permeate flux through PS membrane as a function of pretreatment for a 100-kDa membrane.

4. Conclusions

The current research shows that the combination of 120 ppm of ferric chloride and polysulfone membrane with MWCO of 20 kDa provides 54% of phosphate removal versus the 23% that were achieved by UF alone. The combination of a flocculation and UF process not only demonstrated a synergy effect, but also minimized flux drop, while membranes with higher MWCO values showed a higher reduction in flux.

This interesting tendency was explained by different fouling patterns for membranes with high and low MWCOs. While flux drop in high MWCO membranes was caused by flocs entering the pores and adsorbing on pore walls, membranes with low MWCO were protected from long-term fouling by formation of the porous cake on their surface.

However, the obtained results did not ultimately provide a satisfactory reduction in phosphate values and therefore continuous search for an optimal flocculation–membrane filtration combination is warranted.

Acknowledgements

Alfa-Laval Corporation and Dr. Frank Lipnizki are thanked for free membrane samples. E.A. thanks Israel Ministry of Science for an Eshkol Ph.D. fellowship.

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