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R&D AND DEMONSTRATION OF THE RESEARCH RESULTS OF THE BENCH-SCALE MEMBRANE (UF/NF) PRETREATMENT IN MODIFIED SOIL AQUIFER TREATMENT (SAT) SYSTEM

Membrane Pretreatment for Safe Water Reuse – Lab-scale

1. Introduction

Various types of water which are potentially to be treated by membranes, e.g. municipal effluents, sea water and spent filter backwash water, contain dissolved matter, colloidal suspensions and large particles, considerable part of them organic in nature. Particles and dissolved organic matter (DOM) separation can protect water transport systems and membranes from clogging and, as a result, make the process more economical. Coagulation has a significant effect on the particle size distribution (PSD) in comparison to the original PSD of the water. (Adin and Soffer, 1998). The efficiency of the coagulation affects directly the efficiency of the back transport mechanism of particles from the membrane surface and as a result the fouling intensity. In general, efficient flocculation produces larger flocs which can be transported away from the membrane by lateral migration or shear forces. At the same time, there is a probability to produce small flocs (colloid-colloid interaction) or small colloids which are created by complexion of DOM-iron or aluminum species. The above mentioned possibilities can affect the membrane fouling negatively, due to the fact that these new flocs are too large for brownian motion (another back transport mechanism) than with their primary size before the physical chemical treatment (Soffer et al., 1999). Electroflocculation (EF) is a coagulation/flocculation process in which active coagulant species are generated in situ by electrolytic oxidation of an appropriate anode material. Only in the past decade has awareness grown as to the advantages of using the technology for electrochemical treatment of water and wastewater. Today, electrochemical processes are being used in a wide range of applications (Mollah et al., 2001, Adin and Vescan, 2002). Treatment of wastewater by EF has been practiced for most of the 20th century, yet limited scientific research has explored the efficiency of the technology as an alternative process for water treatment. **The current report concerns in establishing the baseline for NF membrane post-treatment of SAT effluents by investigating UF and NF of the wastewater effluents prior to SAT.**

2. Objectives

The objectives of WP 3.2 are (a) to develop effective and sustainable treatment (and storage) technologies for integration into a semi-closed urban water cycle to promote safe wastewater reuse (b) to define the necessary attributes of appropriate environmental buffers as components and contaminant barriers in an indirect potable reuse system, and (c) to quantify design considerations for waste water recharge to aquifers and recovery considering (i) subsurface virus migration and (ii) production of potentially carcinogenic chlorine derivatives (e.g. THMs)

The role of HUJI in WP 3.2 is bench-scale examination of ultrafiltration/nanofiltration (UF/NF) membrane pretreatment in association with SAT (soil –aquifer treatment) of secondary effluents comparing a novel process of electroflocculation (EF) with conventional flocculation (CF).

3. Introductory preceded study

3.1 Materials and methods

This part of the work establishes a "baseline" by examining the possibility of improving quality of activated sludge effluents treated with iron coagulation (a CF process) before using membrane filtration as an advanced treatment process. It is done by right selection of physical-chemical treatment combinations and determination of pretreatment capability with iron salt as a substitute for aluminum salt, increasing membrane run time and providing better understanding of granular and membrane filtration mechanisms. Activated sludge effluents from the Dan Region Sewage Reclamation Project (Shafdan) served in the investigation. Two flocculants were tested separately: alum $Al_2(SO_4)_3$ which is long standing in water and wastewater treatment and ferric chloride $FeCl_3$. The flocculants were tested by the conventional Jar test procedure to define optimal conditions (pH, dose). Filtration tests were carried out in bench-scale columns, 20 cm and finally 100 cm deep, 1.79 mm sand grain size. Membrane filtration tests were performed in a stirred cell using different MWCOs (molecular weight cut off) (Fig. 1).

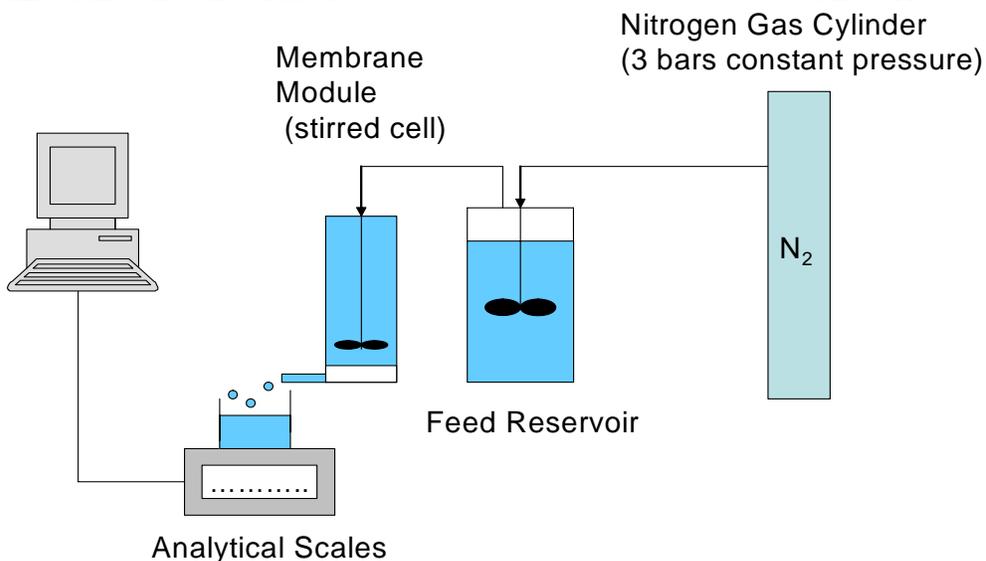


Fig. 1. Membrane filtration bench-scale system

The membrane inlet was fed with wastewater with or without coagulant addition (in-line flocculation). The effect of colloidal suspension pretreatment by EF on membrane fouling was measured by flux decline at constant pressure. A kaolin model suspension was used in this stage of the investigation. An EF cell was operated in batch mode and comprised of two flat sheet electrodes - an aluminum anode and stainless steel cathode - which were immersed in the treated suspension, and connected to an external DC power supply. The cell was run at constant current of 0.06A-0.2A.

3.2 Results and discussion

The flocculation tests showed that the efficient range of pH for alum is pH 6 - 8. In this range there is a direct relationship between the dose and the turbidity and TPC (Total Particle Count) residual ratio. The optimal point is pH 6 and alum dose of 30 mg/l. At this point the turbidity residual ratio was 0.11 and the residual ratio of TPC was 0.07. For ferric chloride at pH 4.5 exhibited the best for removal efficiency. At pH 5 the residual ratio of turbidity was 0.16 and the residual ratio of TPC was 0.01. For ferric chloride, in contrast to alum, it was found that the efficient pH range was larger, pH 3 - 10. Nonetheless, it has been found that the charge neutralization mechanism that prevails in the acidic pH range is more effective for higher removal. While with alum, the residual ratio curves of turbidity and TPC followed one another, for ferric chloride it was different. At low coagulant dosage the turbidity

residual ratio exceeded 1 (i.e. negative removal), leading to a second minimum in the turbidity curve. This phenomenon was probably a result of the formation of tiny colloids of iron hydroxide $\text{Fe}(\text{OH})_3(\text{s})$.

In light of the jar test results, ferric chloride was used in the contact filtration process. At first, filtration tests were done in small 20 cm deep filters, at filtration rate of 10 m/h which is in the middle of the economic domain. Initial tests led to filter media grain size selection of 1.79 mm. While the optimum pH and particle interaction mechanisms for contact filtration corresponded well with the above mentioned jar test results, the optimum doses that were observed in the filtration were half or less than half than the optimal doses produced by the jar tests. It was also found that overdose for filtration was noticed in lower dosages than for the jar test. From this it can be understood that the filtration process is more sensitive to deviations from effective conditions. This sensitivity becomes higher for the sweep coagulation (basic pH) domain as a destabilization mechanism. Head loss increased moderately with increasing coagulant dose and with pH decrease from 10 to 7. Under such conditions the filter experienced short filtration runs and deteriorated filtrate quality. Energy loss increased considerably below pH 7 (charge neutralization zone).

Jar test results, at pH 7.5 (natural pH) and pH 5.5 show that the optimum coagulant dosage which was required for DOM (expressed in terms of UV-254 nm or DOC-dissolved organic) removal was higher than for particle removal, and the removal percent was lower. Under such conditions, the positive influence of decreasing the pH level was even more significant than the case of particle separation as the above mentioned. It was observed that there is not a guarantee that applying UF (ultrafiltration), even in condition of coagulant addition, can succeed in improving DOM removal (e. g., membrane with MWCO of 50KDa gave DOM removal close to jar test results, around 30-50%). This MWCO membrane also was penetrated by small colloids of $\text{Fe}(\text{OH})_3(\text{s})$ and as a result supplied negative turbidity removal. From here, it was necessary to use LMWCO (low MWCO) UF. Significant improvement of DOM removal was achieved just with the decreasing of membrane MWCO to 4 KDa. The DOM removal increased with increasing coagulant dose and decreasing pH.

Usually, the contribution of membrane filtration on DOM removal efficiency relative to the jar test was higher in the case of the natural pH than in the case of acidic pH.

In order to reduce the sensitivity of membrane filtration to PSD varieties, in-line filtration of wastewater coagulant suspension was performed before the membrane filtration stage. The granular filtration system included 100 cm deep bed filters containing coarse sand of 1.79 mm average grain size. In most of the cases, this pre-treatment can reduce membrane fouling intensity. The fouling improvement was depending on the coagulant dose. In general, increasing the dose gave better improvement in membrane flux. Nanofiltration tests provided better removal efficiency, but the price was increasing sensitivity to internal fouling (Fig. 2). The letter should be further investigated.

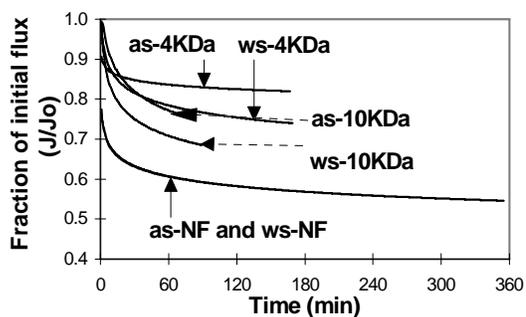
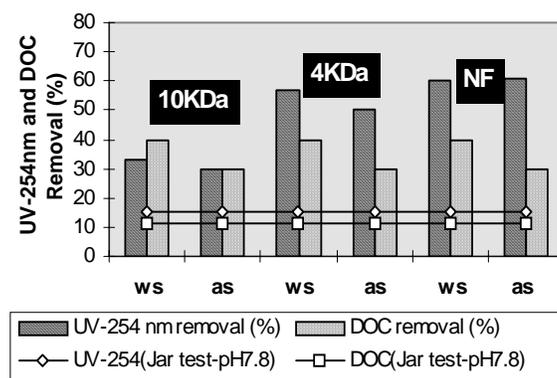


Fig. 2a. Summary of fouling curves for C-10 KDa, KDa, PES-4 KDa and NF with or without flocs separation of membrane inlet (pH 7.8, 150 mg/L).

Fig. 2b. Summary of DOM removal for C-10 PES-4 KDa and NF (pH 7.8, 150 mg/L).

The preliminary EF-EU results with kaolin suspension show that pre-EF enhances the permeate flux at pH 5 and 6.5, but only marginal improvement is observed at pH 8. At all pH values cake formation on the membrane surface was observed. The differences in membrane behavior can be explained by conventional coagulation theory and transitions between aluminum mononuclear species which affect particle characteristics and consequently cake properties. At pH 6.5, where sweep floc mechanism dominates due to increased precipitation of aluminum hydroxide, increased flux rates were observed. It is evident that EF can serve as an efficient pretreatment to ultrafiltration of colloid particles.

3.3 Conclusions

1. Activated sludge effluent. Charge neutralization mechanism was more effective for higher removal. While with alum residual ratio curves of turbidity and total particle count (TPC) followed one another, for ferric chloride it was different.
2. At low coagulant dosage, turbidity showed negative removal, leading to a second minimum in the turbidity curve. That phenomenon was probably a result of formation of tiny colloids of iron hydroxide $\text{Fe}(\text{OH})_3(\text{s})$.
3. Contact granular filtration reduced membrane fouling intensity. Increasing the dose resulted in higher improvement in membrane flux.
4. Nanofiltration provided better removal efficiency, but sensitivity to internal fouling increased greatly.
5. For kaolin suspension, pre-EF enhances the permeate flux at pH 5 and 6.5, but only marginal improvement is observed at pH 8. At all pH values cake formation on the membrane surface was observed.

4. The Use of Electrocoagulation to Remove Humic Acid from Water as Pretreatment in Membrane Filtration

4.1 Introduction

Humic substances, which are a major constituent of wastewater effluents, are normally difficult to be removed by infiltration through sand. This part of the work examined electrocoagulation of humic acid and the effect of some operative parameters on the process efficiency. Electrocoagulation was also tested as a pretreatment in membrane ultrafiltration (UF) of humics.

Results show, that electrocoagulation can be effectively used in the removal of humic acid from water, as demonstrated by using Leonardite at pH 7-8.1. Up to 90% of the segment absorbing UV at 254 nm was removed whereas the TOC was reduced up to 80%. Lowering the pH reduced the process

efficiency, particularly with respect to TOC removal; yet, a high iron concentration improved it. The process efficiency was found to be independent of current density.

Electrocoagulation as pretreatment for UF membrane filtration improved filtrate quality and reduced the fouling, particularly by lowering cake influence. The iron ions released from the electrode at pH 5.3-8.1 was found to be ferrous that probably oxidizes to ferric. More investigation is needed in relation to iron species formation in electrocoagulation to water pH and iron pC.

4.2. Materials and methods

4.2.1. Materials

The humic acid was Leonardite produced by the IHSS. The water used was tap water with a negligible TOC concentration. pH adjustments were done with NaOH or HCl. The electrodes were iron electrodes of different sizes (A_{elec}).

4.2.2 Batch tests

The sedimentation tests were conducted in a batch using a Phipps and Bird 7790-902b six paddle jar test system. The coagulant was generated by connecting two iron electrodes to a DC power supply, ADVISE P 3030, using an 800 ml cup. The valence of the ferric ions released from the electrode was determined by comparing the actual iron concentration in water (without humic acid) to the theoretical concentration according to Faraday's Law. Humic acid removal was examined by settling tests using TOC and UV_{254} as parameters with an initial concentration of 6-7 ppm and 0.200-0.250 cm^{-1} respectively. The removal was calculated by

$$\% \text{ removal} = \left(\frac{C_0 - C}{C_0} \right) \times 100 \quad (10)$$

where C was the final and C_0 the initial concentration. The tests were conducted at varying pH and current density values, using different electrodes sizes with the same current or the same electrode with a different current. A sample was taken after 10 minutes of slow mixing (30 rpm) and 1 hour of settling.

4.2.3 Pretreatment tests

Electrocoagulation pretreatment tests were carried out in a stirred cell, as described in Fig.2, using membranes with 4 KDa and 150 KDa molecular weight cut off made of PES.

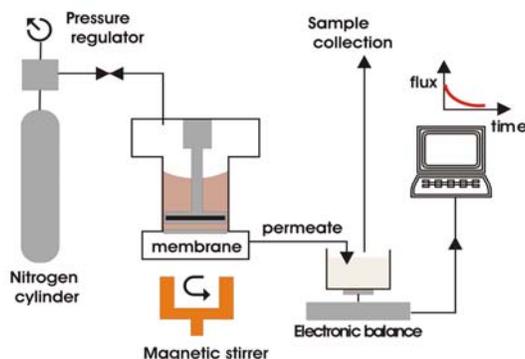


Fig.2 Stirred dead end cell for pretreatment tests

These experiments were performed in pH 7 with a constant ferric concentration (10 ppm Fe), and a constant TOC and dissolved organic carbon (DOC) concentration (9-10 ppm from which 4 ppm DOC). The fouling parameter was measured by relative flux with an electronic balance connected to a PC. The initial flux was measured using distilled water, after which humic acid was filtrated until 200 ml of filtrate was collected. The cake impact was investigated by gently removing it with a delicate wiper after rinsing with distilled water and measuring the flux with distilled water. The inner clogging was examined after soaking the membrane in 0.1 N NaOH for 24 hours and measuring the flux again with distilled water.

4.2.4 Analytical procedures

The TOC and DOC were measured with TOC-V SHIMADZU. DOC was the TOC concentration after filtration through a 0.45 μm membrane. A UV-Vis spectrophotometer HP model 8452A was used to measure UV_{254} absorption, and ferric concentration was measured with Perkin Elmer Optima XP ICP.

4.3. Results

4.3.1 Jar test experiment

4.3.1.1 Correlation with Faraday Law

The iron concentration that was measured in all conditions, pH 5-8 and various current densities ($j = 62.5-750 \text{ A/m}^2$) using the same electrodes or electrodes of different sizes, correlated with divalent iron ($n=2$) and matched within 85-95% the theoretical concentration according to Faraday Law (Fig.3).

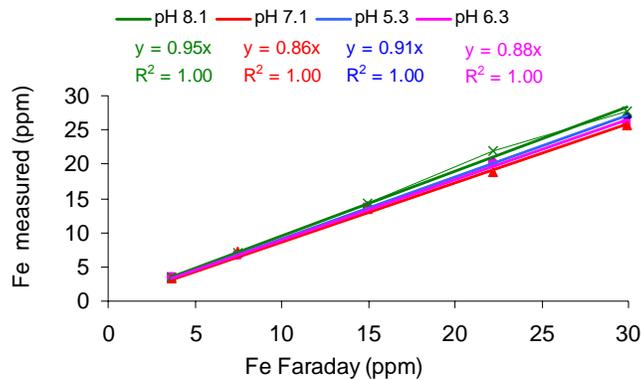


Fig.3. Fe measured vs. Fe calculated by Faraday Law
pH 5-8, $I=0.6\text{A}$, $A_{elec}=9 \text{ cm}^2$.

After adding the iron to the water, different colors were observed. These colors changed with pH and coagulant concentration suggested different iron species, according to the mechanisms for the oxidation of iron electrode. At pH5 a light yellow color appeared only at high coagulant concentration while an increase in the pH values caused a darker yellow brown color to appear even at low coagulant concentrations. This difference in the iron species under different conditions affects the removal of the humic acid as will be demonstrated later.

4.3.1.2 Sedimentation tests

The sedimentation tests resulted in an 80-90% UV_{254} removal at a low coagulant concentration and up to 80% TOC removal, although a higher coagulant concentration was required (Figs. 4 and 5).

The removal efficiency decreased with pH and increased with iron concentration along with a change

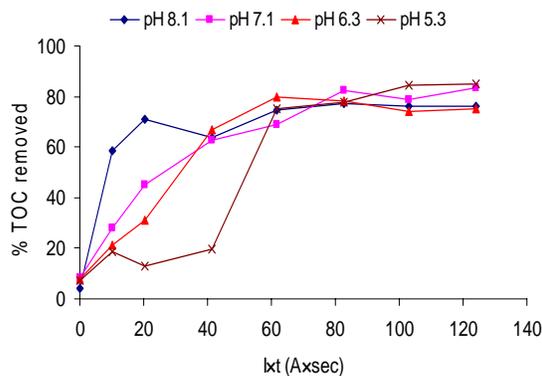


Fig.4. % TOC removed vs. iron concentration.
pH 5-8, $I=0.6A$, $A_{elec}=9\text{ cm}^2$.

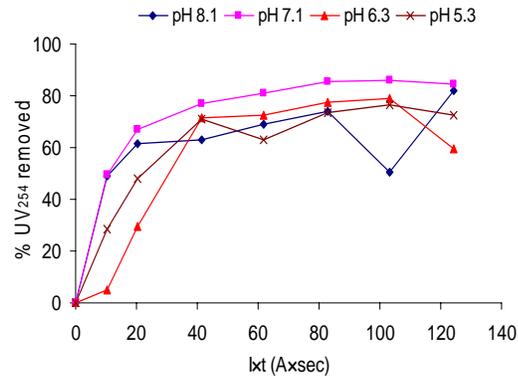


Fig.5. % UV₂₅₄ removed vs. iron concentration.
pH 5-8, $i=0.6A$, $A_{elec}=9\text{ cm}^2$.

in the removal trend. At pH value of 8 the optimal removal, from 6.8 ppm to 1.7 ppm TOC and from 0.200 cm^{-1} to 0.040 cm^{-1} UV₂₅₄, occurred at low iron concentration of 7.2 ppm when a further addition of coagulant had a little effect, while at pH 5.3 the same removal efficiency was obtained at 22 ppm iron concentration. Moreover below this coagulant concentration there was a poor humic removal while above it the efficiency did not change. At pH 6-7 the removal trend showed an even incline expressed by the higher humic removal with the addition of coagulant, up to a constant value. It is well known that in chemical coagulation the iron species, normally Fe^{+3} , change with pH or iron concentration according to the pH-pC diagram, when the best pH value for removing humics with iron is pH 4.5-5.5 (Edwards, M., 1997; Edwards, G.A. and A. Amirtharajah, 1985). In our case the dominant ion is Fe^{+2} which can oxidize to Fe^{+3} , because of the oxygen produced by the water oxidation on the anode, or can remain as Fe^{+2} . This difference, compared with the chemical coagulant, might explain our findings. At pH 7-8 the iron is probably found as amorphous ferric oxyhydroxide or ferric hydroxide received from the oxidation of ferrous and seen by the yellow brown color in the solution. This ferric species are responsible for the removing of humics. At a lower pH the absence of color in the water at low iron concentration suggests that the ferrous ions, are dominant; whereas at higher concentration the water color changes to a light yellow-brown thus leading to the assumption that either ferric or ferrous hydroxide exists causing the humic acid removal. Larue and Vorobiev, 2003 have also found that coagulation with electrocoagulation and FeSO_4 behave similarly in kaolin sedimentation with a minimum level of turbidity at pH 7-8. Other researches have shown that electrocoagulation removes COD and organic pollutants from wastewater at pH 7-9 (Mahesh S., B. et al, 2006; Kobya, M. et al, 2003).

The current density did not affect the overall removal indicating that the final iron concentration rather than the pace (change in current density) introduced into the water, determines the process efficiency. The current density was changed while maintaining the coagulant concentration constant, by keeping the multiplication of time and current constant (Fig. 6) or by changing the size of the electrodes (Fig. 7), in both cases the outcome was the same - the electrocoagulation efficiency, under the same coagulant concentration, at different current density, were similar. This is inconsistent with the findings of others who state that the current density influences the electrocoagulation efficacy in the removal of pollutants (Mahesh S., B. et al, 2006; Monser, L. and N. Adhoum, 2004). However, those tests were conducted by changing the current without changing the time so that it is the coagulant concentration (charge loading) rather than the current density that is actually examined (Chen X. et al, 2002). Others showed that at a low current density the efficiency decreases slightly but above it there is almost no effect on the overall removal (Mahesh S., B. et al, 2006). We must restrict the finding that the current density does not influence electrocoagulation only to the overall removal because as was found by Harif et al., 2006 and Hult, 2002 the current density effects flocs size and shape the size of the bubbles that form during the process thus on the removal of the flocs mechanism and increase in the operational cost; nevertheless, these changes did not influence our results.

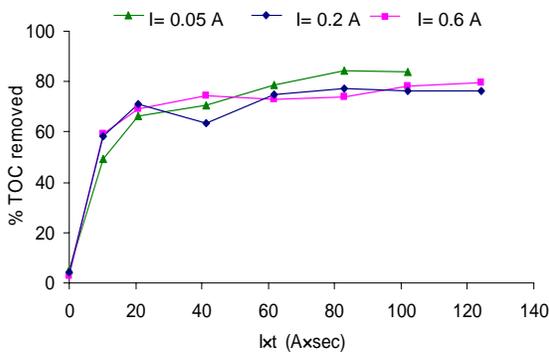


Fig.6.. % TOC removed vs. iron concentration at different current densities. pH 8.1, $A_{elec} = 9 \text{ cm}^2$.

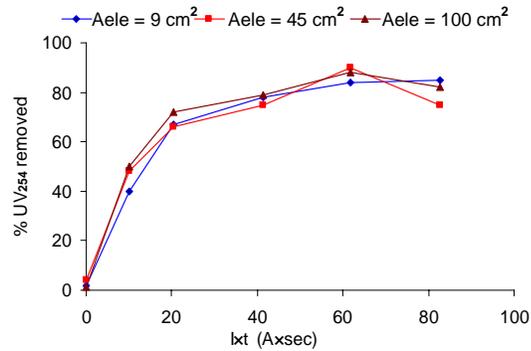


Fig.7. % UV₂₅₄ removed vs. iron concentration at different current densities. pH 7.1, $I = 0.4$, $A_{elec} = 8, 45 \text{ and } 100 \text{ cm}^2$.

Electrocoagulation as a pretreatment to UF membrane displayed improvement both in humic acid removal and fouling minimization. TOC and DOC removal after filtration with pretreatment was 50% higher with the two membranes tested (Table 1).

The residual TOC after filtration with the 150 KDa membrane was 2-3 ppm and 4 ppm from an initial concentration of 10 ppm and DOC from an initial concentration of 4 ppm to 2 ppm and 3.5 ppm with and without pretreatment respectively. DOC and TOC concentrations after filtration remained the same and were similar with all membranes, raising the assumption that that was the fraction that could not be removed. The 150 KDa exhibited 70% removal of the UV_{254} fraction, from 0.250 cm^{-1} to 0.08 cm^{-1} , after electrocoagulation while almost no change occurred without the pretreatment. Although the 4 KDa membranes were capable of removing UV_{254} without pretreatment; still, the pretreatment improved its performance by 50%.

Table 1. TOC, DOC concentration and UV_{254} absorption before and after filtration with 150 KDa and 4 KDa membranes with and without pretreatment.

	TOC _i (ppm)	TOC _f (ppm)	DOC _i (ppm)	DOC _f (ppm)	UV _{254 i} (cm ⁻¹)	UV _{254 f} (cm ⁻¹)
150 KDa	9.3	4.35	4.8	4.35	0.270	0.234
150 KDa + EF	9.70	2.30	3.50	1.80	0.280	0.080
4 KDa	11.30	3.10	3.05	2.75	0.200	0.050
4 KDa + EF	9.80	1.90	2.80	1.40	0.200	0.025

TOC_i initial concentration

TOC_f concentration after filtration

EF with electrocoagulation

pH 7.1, $i = 0.6A$, $A_{elec} = 9\text{ cm}^2$, $[Fe] = 10\text{ ppm}$

The electrocoagulation reduced the fouling after filtration. The fast decline in the flux simulated by the slope of the curve at the beginning of the filtration, resulting from internal clogging, decreased with the pretreatment and the flux at the end of the filtration was higher (Figs. 8 and 9).

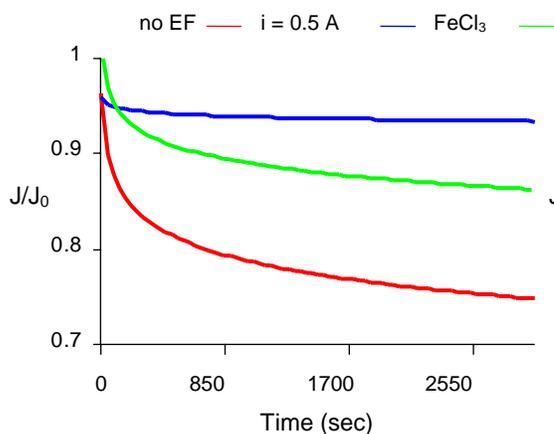
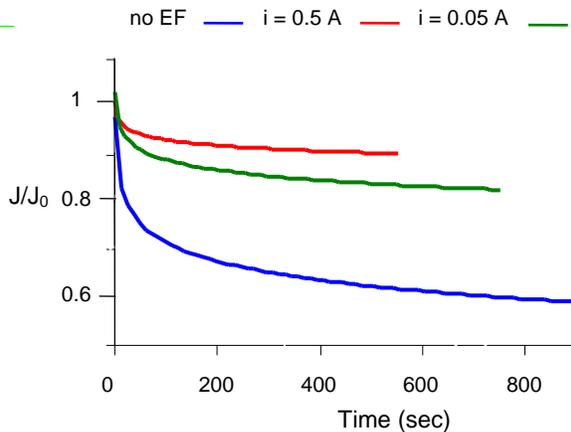


Fig.8. 4 KDa membrane relative flux vs. time, without pretreatment (no EF) and with pretreatment electrocoagulation ($I=0.5A$) and chemical coagulation ($FeCl_3$). pH 7.1, $[Fe]= 10$ ppm.



Fi.9. 150 KDa membrane relative flux vs. time, without pretreatment (no EF) and with pretreatment with two current densities ($I=0.5 A, 0.05 A$). pH 7.1, $[Fe]= 10$ ppm.

The higher flux at the end of the filtration with the pretreatment than without it leads to suggesting that a less dense cake with a higher permeability was formed on the membrane surface. In addition, the less dense cake after filtration with the pretreatment made it easy to clean it from the membrane surface simply by rinsing the membrane with water compared with the need of wiping the membrane surface with a wiper, in the case where there was no pretreatment. Removing the cake reconstructed the flux up to 90% of the clean membrane's initial flux, with or without pretreatment, indicating that this was the main fouling factor. After soaking the membrane in NaOH the flux demonstrated a small change and therefore the irreversible inner clogging was insignificant.

Pretreatment with chemical coagulation gave similar results in humic acid removal, but was not as good in the cake fouling reduction.

4.4. Conclusions

Electrocoagulation can be effectively used in the removal of humic acid from water, as demonstrated by using Leonardite at pH 7-8.1. Up to 90% of the segment absorbing UV at 254 nm was removed whereas the TOC was reduced up to 80%. Lowering the pH reduced the process efficiency, particularly with respect to TOC removal; yet, a high iron concentration improved it. The process efficiency was found to be independent of current density. Electrocoagulation as pretreatment for UF membrane filtration improved filtrate quality and reduced the fouling, particularly by lowering cake

influence. The iron ions released from the electrode at pH 5.3-8.1 was found to be ferrous that probably oxidizes to ferric. More investigation is needed in relation to iron species formation in electrocoagulation to water pH and iron pC.

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