Evaluation of a Dead-end Ultrafiltration Membrane for the Removal of Organic Matter from a Water Reservoir in Colombia

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The removal of natural organic matter (NOM) from surface water sources was evaluated by a pilot ultrafiltration plant through 3 different treatment processes: UF, PAC/UF and PAC/C-S/UF. For UF, the ultrafiltration plant was used as the only treatment. For PAC/UF, tests were performed with powdered activated carbon (PAC) as pre-treatment to the ultrafiltration membrane. For the PAC/C-S/UF hybrid treatment process, adsorption with PAC and coagulation-sedimentation with aluminum sulphate as a coagulant was used as pre-treatment. The results indicated that the best hybrid treatment process was PAC/C-S/UF, since it maintained a stable transmembrane pressure (TMP) of around 0.5 bar in the operating time, while in the this process the flow remained constant at about 227 L / (m² · h). In addition, with this process, the highest removal efficiencies were obtained for the NOM parameters evaluated. Coagulation pre-treatment improved the removal of NOM in comparison with PAC and UF as the only treatment. The results showed that both hydrophobic and hydrophilic organic compounds could be removed by the action of the coagulant. When further comparing the increased removal rates of the hydrophobic material (HoM) and hydrophilic material (HiM) fractions, it was observed that the hydrophobic organic chromophores could be removed by coagulation more easily than the hydrophilic organic chromophore compounds. The molecular weight distribution (MWD) was evaluated, indicating that the PAC/C-S/UF treatment successfully removed medium-high molecular weight (MW) compounds (> 1701 Da), but the removal efficiency of medium-low MW compounds (≤ 1701 Da) began to decrease as the compounds had lower MW.

Keywords: dead-end ultrafiltration, natural organic matter removal, molecular weight distribution, chemical fractionation, drinking water.

Introduction

Natural water sources mostly contain natural organic matter (NOM) and organic matter from wastewater. However, the amount, character and properties of NOM present in the sources differ considerably depending on their origin, the biogeochemical environment, and the type of wastewater that is discharged (Fabris et al., 2008). Moreover, the nature of the organic compounds of NOM may vary with climatic changes like heavy rains, floods and droughts (Sharp et al., 2006). Floods and droughts have the largest impact on the availability and quality of water. These climatic changes are some of the reasons for the increase in the total amount of NOM in the water (Evans et al., 2005). The compositional, physical and chemical properties of NOM, such as its specific UV absorbance (SUVA), can also suffer changes due to these phenomena (Eikebrokk, 2004). NOM interferes with the removal of many pollutants, including metals. It is responsible for clogging membranes, contributes to corrosion and acts as a substrate for bacterial growth in distribution systems. Furthermore, it is the largest contributor to the formation of disinfection by-products (DBPs).

Dissolved organic carbon (DOC), UV254, pH, turbidity and colour are the most common parameters evaluated in water treatment plants to control NOM. However, this characterisation does not provide information on the composition and chemical structure of the NOM, such as its molecular weight and hydrophobicity. Using size exclusion chromatography, the molecular weight distribution of NOM (Sharp et al., 2006; Li et al., 2013) can be obtained. Furthermore, its hydrophicity and hydrophobicity can be determined with fractionation techniques with resins (Sharp et al., 2006; Matilainen et al., 2011). The hydrophilic fraction of NOM is composed mainly of aliphatic and nitrogen compounds, such as carboxylic acids, carbohydrates and proteins. The hydrophobic fraction primarily consists of humic and fulvic acids (humic substance), and is rich in aromatic compounds, phenolic structures and conjugated double bonds (Duan and Gregory, 2003). The composition of these fractions can help to select the correct removal process, coagulant dosage and oxidizing agent, and may affect the potential formation of the disinfection by-products (PFDBPs). DBPs have adverse effects on human health. More than 600 compounds have been identified,

including trihalomethanes (THMs) and haloacetic acids (HAA) (Krasner et al., 2006; Tian et al., 2013). Hydrophobic fraction and low molecular weight compounds are considered to be larger generators of DBPs than hydrophilic fraction and high molecular weight compounds. However, it has been found that hydrophilic compounds also contribute to the formation of DBPs, particularly in water with a low content of humic acids (Cheng et al., 2005; Yang et al., 2013). Therefore, optimisation of water treatment should be carried out in order to remove both hydrophobic and hydrophilic organic compounds, and to mitigate the formation of DBPs. Problems in water quality caused by these compounds mean that regulations are becoming increasingly stricter. Consequently, as much NOM as possible should be removed from raw water through methods including coagulation, flocculation, sedimentation, adsorption, chemical oxidation, membrane microfiltration and ultrafiltration, ultraviolet radiation, photocatalysis, electrocoagulation and biofiltration, or a combination of these.

This article reports the pilot-level evaluation of an ultrafiltration membrane used in combination with conventional processes to improve the removal of NOM from a conventional drinking water treatment plant. Several physical and chemical properties of raw and treated water, such as MWD and chemical fractionation with DAX and DOC resins, were evaluated to better understand how different treatment processes alter the characteristics and reactivity of NOM.
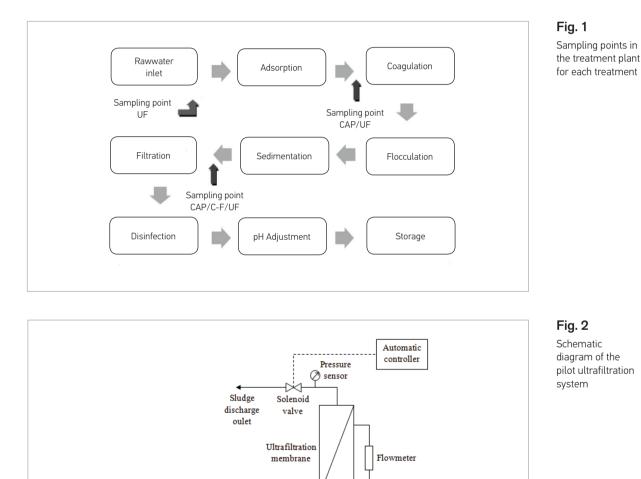
Materials and methods

Experimental treatments

UF, adsorption-UF and adsorption-coagulation-sedimentation-UF treatments were evaluated. For the treatments, doses of 2 mg/L PAC were used for adsorption, 36 mg/L aluminum sulphate (Al2 (SO4) 3 · 18H2O, liquid) were used as a coagulant and 0.019 mg/L of a non-ionic polymer (N-300 PWG) was used as a flocculation aid. The 3 treatments were denoted as UF, PAC/UF, and PAC/C-F/UF, respectively. To clean the ultrafiltration membrane, forward flush cycles were performed every 10 minutes with water from the feed tank; disinfection

was carried out after each test with sodium hypochlorite at a concentration of 400 ppm for 45 minutes; and a 2-hour cleaning with citric acid at a pH of 2 was done. When the membrane became excessively dirty, backwash cleanings were performed with water from the permeate tank. The forward flush cleanings were automatic, but backwash cleanings were manual as were chemical cleanings.

For the experiments of each treatment, the samples were taken in the potabilisation plant located in the northeast of Colombia during the period of study. Each test was batch made of 240 litres of permeate volume, and in triplicate. Fig. 1 shows the sampling points for each of the treatments.

A schematic illustration of the pilot ultrafiltration system is shown in Fig. 2. This modular ultrafiltration pilot system was designed and built to treat an average flow rate of 0.6 m³/h with an electric pump with a peripheral feed impeller with a capacity of 0.72 m³/h at 37 m.w.c. The pilot plant consists of a preliminary treatment with a filter of 100 μm pore diameter. The ultrafiltration module has a tubular polyether sulfone membrane (LFFB-4-3-1, LEGIO. filter) with an effective pore size of 0.02 μm, and a membrane filtration area of 2.65 m² operating at a nominal transmembrane pressure of 0.5 bar.

Fig. 1

Sampling points in the treatment plant for each treatment

Fig. 2

Schematic diagram of the pilot ultrafiltration system

Raw water

During the experimentation, the raw water was maintained at a temperature in the range of 17°C to 19°C, and the pH in the range of 6.5 to 6.7. Other water quality characteristics, e.g. turbidity, conductivity, COD, UV254 and SUVA (specific UV absorbance) were in the range of 9.69 ± 1.34 UNT, 40.25 ± 0.78 uS/Cm, 6.93 ± 0.05 mg-C/L, 0.172 ± 0.003 cm-1 and 2.48 ± 0.02 L/mgC · m, respectively.

Data treatment

The data obtained were analysed using the STAT-GRAPHICS Centurion XV version 15.2.11 software package, owned by the University of Antioquia. Analysis of variance (ANOVA) was used to compare the results and determine possible statistically significant relationships between them. The multiple range test was employed to search for different groups of statistically homogeneous behaviour. Both the analysis of variance and the multiple range test were used with a confidence level of 95%.

Analytical techniques

The analyses carried out in the tests with the pilot ultra-filtration system were done using standardised analytical techniques, in the laboratory of the GDCON group accredited by the IDEAM and ONAC accrediting agencies in Colombia. The turbidity was monitored using a turbidity meter (WTW 550 IR). COD was measured using a Total Organic Carbon Analyzer (TOC) (Apollo 9000, Teledyne Tekmar). UV absorbance at 254 nm, UV254, of the solution filtered through a 0.45-micron pore diameter filter was determined using an ultraviolet/visible spectrophotometer (UV-Vis Evolution 600, Thermo).

Molecular weight distribution by size exclusion chromatography (HPSEC)

The molecular weight distribution by size exclusion chromatography (HPSEC) was determined with an Agilent 1100-1200 Liquid Chromatograph equipped with a low pressure gradient quaternary pump (model G1311A), a mobile phase degasser (model G1379A), a diode array detector (model G1315B), a column oven (model G1316A) and an automatic injector (model G1329A). The data analysis software used was ChemStation Software Rev. B. 04. 03 with ChemSta-tion GPC Data Analysis Software Rev. B. 01. 01. A Bi-oSep-SEC-S2000 analytical column (Phenomenex, 7.8 x 300 Mm, 5 µm particle size, 145 Å pore size, stationary phase on silica) and BioSep-SEC-S2000 column guard (Phenomenex, 7.8 x 75 mm, 5 µm particle size, 145 Å pore size, stationary phase on silica) were used. The chromatographic conditions were 70 µL injection volume, 1.2 mL/min mobile phase flow, column temperature of 30°C, a mobile acetate buffer phase of 0.06 M, and detection wavelengths of 254 nm.

From each sample filtered by 0.45-µm pore diameter filter, 900 µL was taken and deposited in a 1-mL muffled amber vial. As the ionic strength of the sample must be equated with the ionic strength of the mobile phase, 100 µL of 0.6 M acetate buffer was added. It was covered with a PTFE cap and septum and put into a vortex for approximately 20 seconds. Each prepared sample was injected directly into the chromatographic system under the conditions described above. The mobile phase should be membrane filtrated 0.45-µm cellulose acetate. The method is isocratic, so there will be no elution gradient. The chromatographic system must be conditioned by a passing mobile phase through the column until a stable baseline is observed for each of the wavelengths to be analysed. Calibration and calculations should be done at a wavelength of 254 nm (Zhou et al., 2000).

Hydrophobicity of aquatic organic matter

The hydrophobicity of the aquatic organic matter was determined by the method of hydrophilicity (Thacker et al., 2005; (APHA) et al., 2012). Hydrophilicity was determined using an Amberlite DAX-8 resin (Sigma), a Pall agitator by Environcheck PN tubes 4821, Soxhlet type solvent extraction equipment, a Boeco U-320 R centrifuge with temperature control and maximum centrifugation rate of 5000 rpm, and a Thermo Evolution 600 UV-Vis spectrophotometer.

Each batch of resin was carefully washed to avoid the incorporation of organic material from the resin into the sample, which is the main disadvantage of any method that uses this material for the fractionation of NOM. The resin was washed with 5 successive 0.1 M HCl extractions, followed by 5 extractions with 0.1 M NaOH. Each extraction had a duration of 24 hours and the supernatant was removed after each extraction. The res-

in was then thoroughly cleaned by Soxhlet extractions with acetonitrile and methanol of 24 hours duration each. The cleaned resin was stored in methanol. Prior to use, the resin was rinsed and acidified for the experimental run in succession with 0.1 M NaOH, deionised water and 0.1 M H3PO4 in a ratio of 100:200:100 mL, respectively, for each 5 grams of resin. The purpose of the rinsing is for the resin to be free of dissolved organic carbon (DOC) residues that may interfere with the analysis.

Each sample was passed through a 0.45-μm pore diameter filter. 20 mL of the sample were taken and acidified with 2 drops of 6 M phosphoric acid until a pH ≤ 2 was obtained. 0.5 g of rinsed and dried DAX-8 resin was weighed. The sample was placed in contact with the resin and stirred at 100 rpm for 30 minutes.

The sample was then centrifuged at 5000 rpm for 60 minutes and the supernatant (containing the non-hydrophobic fraction) was removed for further reading in the spectrophotometer as was the initial sample. For reading, 1.5 mL of phosphate buffer and 1.5 mL of the sample were taken in the reading cell, mixed, and read at an absorbance at 340 nm. The hydrophobic fraction was calculated by the difference between the initial sample and the sample with the non-hydrophobic fraction.

Results and discussion

Treatment removal performance

The pollutant removal efficiencies of the 3 processes are summarised in Table 1. As shown in Table 1, the turbidity removal efficiency was approximately 98% for the 3 processes, indicating excellent particle removal behaviour. The removal efficiency of the total coliforms was 100%, indicating the total removal of these microorganisms and hence the optimal disinfection of the water.

The results also showed that the organic matter removal performance improved substantially in the PAC/UF and PAC/C-S/UF processes compared with UF without pre-treatment. In particular, the COD decrease rate was almost 70% higher in the PAC/C-S/UF (83.62 ± 0.43%) than the PAC/UF and UF because the coagulation-sed-imentation treatment could agglomerate and sediment part of the DOC, which was higher than in other previous treatments. The results of the PAC/UF and UF processes had similar low DOC removal efficiencies of about 13%, indicating that the dissolved organic material present in the raw water passes through the UF membrane pores. In addition, the rate of decrease of UV254 was approximately 10% higher in the PAC/UF process than that of UF because the PAC could adsorb part of UV254.

This further favoured coagulation in particle agglomeration, since the removal efficiency increased 7% more in the PAC/C-S/UF process than in the PAC/UF process.

TMP is an indicator of membrane fouling, which is required to maintain the desired potable water flow. In this investigation, the MTP increased with running time, while the flow in the 3 processes remained constant at about 227 L/(m$^2 \cdot$ h) before the TMP exceeded 2.5 bar. TMP performance for the different processes is shown in Fig. 3.

As shown in Fig. 3, there was an evident difference in the TMP evolution during the operation of the 3 processes. The TMP gradually increased over time from 0.5 bar for the PAC/UF and UF processes with a different trend for each. The PAC/UF process had a higher TMP increase rate than the other 2 processes because small PAC particles accumulated more and more on the surface of the membrane, forming a fine-grained gel layer which could not be effectively removed by washing the membrane (Liang et al., 2008).

In the case of the PAC/C-S/UF process, TMP was constant during the investigation at around 0.5 bar, which means that of the 3 processes the PAC/C-S/UF was generally the best configuration for the control of membrane fouling.

The order of the rate of increase of TMP in the 3 processes is given as follows: PAC/C-S/UF < UF < PAC/UF, indicating that previous treatment with PAC/C-S was more appropriate for the control of the fouling of the UF membrane than PAC because the C-S agglomerates and sediments both the PAC particles and the organic material present in the raw water and largely prevents the formation of layers on the surface of the membrane, reducing fouling of the membrane by NOM (Yu et al., 2016).
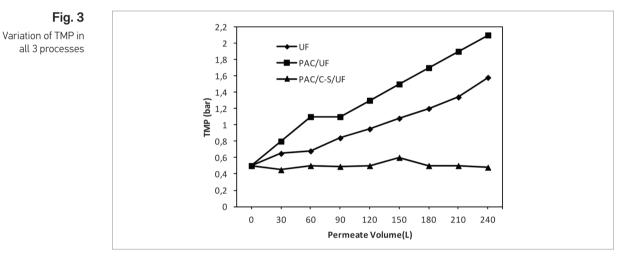
Table 1

Efficiency of pollutant removal for water purification processes

Water quality indexes	Raw water	UF		PAC/UF		PAC/C-S/UF	
		Effluent	Total removal (%)	Effluent	Total removal (%)	Effluent	Total removal (%)
1	2	3	4	5	6	7	8
Turbidity (NTU)	9.68 ±1.34	0.15 ±0.01	98.50±0.15	0.13±0.02	98.67±0.17	0.14±0.01	98.53±0.12
DOC (mg/L)	6.928±0.055	6.011±0.119	13.24±1.72	6.027±0.108	13.00±1.56	1.135±0.030	83.62±0.43
UV_{254} (cm^{-1})	0.172±0.003	0.151±0.001	12.43±0.37	0.134±0.002	21.95±1.02	0.125±0.001	27.54±0.28
Total coliforms (UFC/100 mL)	1900±58	0±0	100±0	0±0	100±0	0±0	100±0

Fig. 3

Variation of TMP in all 3 processes

Chemical fractionation of MON

Different treatment processes have different factors that influence water quality. The fractionation of dissolved organic matter (DOM) in the raw water after the UF, PAC/UF and PAC/C-S/UF processes was investigated. Fig. 4 shows the chemical fractions of DOM for the raw water after each treatment.

The DOM in terms of UV340 nm absorbance was reduced from 0.0595 m^{-1} for the raw water to 0.0473 m^{-1}, 0.0415 m^{-1} and 0.0056 m^{-1} after UF, PAC/UF and PAC/C-S/UF treatments, respectively. This indicates that that the PAC/C-S/UF hybrid treatment process had the highest removal efficiency of DOM that absorb light at a wavelength of 340 nm, as is the case with aromatic compounds, quinone and conjugate structures present

in the water. The treatment processes of UF, PAC/UF and PAC/C-S/UF had a DOM removal rate of 20.59%, 30.25% and 90.64%, respectively.

However, as shown in Fig. 4, the UF treatment process had the lowest removal rates of HoM and HiM, with 22.22% and 12.50%, respectively. This indicates the low capacity of the UF membrane when used as the only treatment to remove the HoM and HiM fractions from DOM. The PAC/UF hybrid treatment had very similar removal efficiencies of 30.30% and 30.00% for HoM and HiM, respectively. This shows an increase in the removal of DOM for HoM of 8.08% and for HiM of 17.50%. This is because hydrophobic organic components such as humic substances may have less capacity to be absorbed at the PAC surface than hydrophilic organic

components such as quinones and amino acids (Zhang et al., 2015). The PAC/C-S/UF hybrid treatment had an excellent performance with the highest removal rates of HoM and HiM of 93.80% and 75.00%, respectively. In the PAC/C-S/UF process, there was a considerable improvement in the removal rate of HoM and HiM (63.49% and 45.00%, respectively) when compared with PAC/UF.

This is because both hydrophobic and hydrophilic organic compounds could be removed by the action of the coagulant. When further comparing the increase of the removal rates of HoM and HiM, it is observed that the hydrophobic organic chromophores could be removed by coagulation more easily than the hydrophilic organic chromophore compounds.
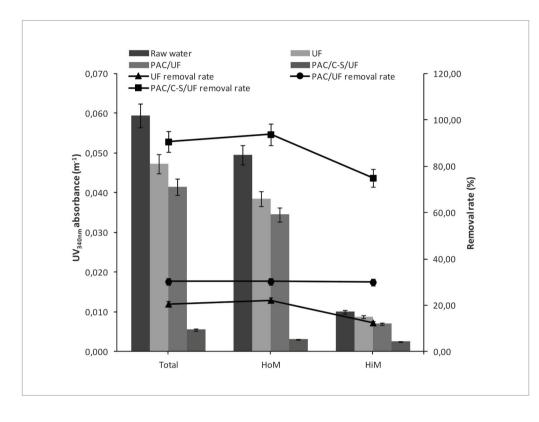

Fig. 4

Chemical fractions of the effluent NOM for the 3 processes

MWD of NOM

The molecular weight distribution of DOM both in the raw water and in the effluent of the UF, PAC/UF and PAC/C-S/UF processes were investigated. Fig. 5 shows the MWD for the raw water after each treatment. The DOM compounds present in the analysed water were found to be distributed according to their apparent molecular weight in 4 marked fractions > 32900, 2560-2328, 1701-1543 and 868-790 Da. It can be observed that DOM of high, medium and low molecular weight are present in the raw water, especially compounds of medium and low molecular weight, which can be removed from the water as it is passed through the 3 processes of treatment. This is clearly seen in Fig. 5, where
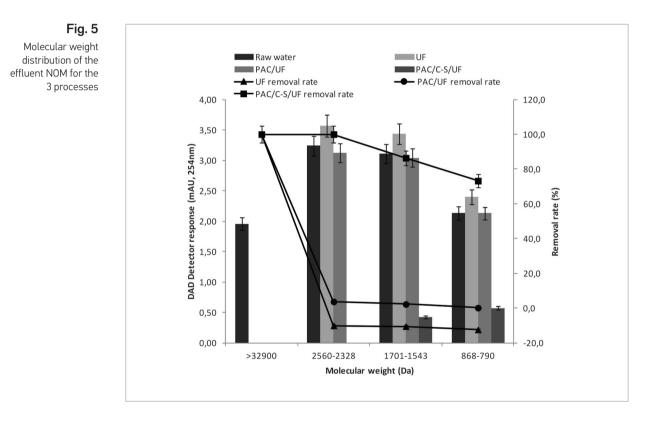
the apparent high molecular weight compounds, fraction > 32900 Da, are removed by the 3 treatment processes evaluated, with a removal rate of 100% for all three UF, PAC/UF and PAC/C-S/UF processes. Of the 3 treatment processes, the one with the highest removal rate of fractions 2560-2328, 1701-1543 and 868-790 Da of MWD of DOM in the raw water was the PAC/C-S/UF process with the rates of removal of 100.00%, 86.42% and 73.21% for the respective fractions. As can be seen, the PAC/C-S/UF process completely removes the MW fractions > 32900 and 2560-2328 Da, but the removal efficiency decreases for the MW fractions 1701-1543 and 868-790 Da. This indicates that PAC/C-S/UF could satisfactorily eliminate medium-high MW compounds

(> 1701 Da), but the removal efficiency of medium-low MW compounds (≤ 1701 Da) begins to decrease as the compounds have lower MW. This is because the action of the coagulant can more easily agglomerate medium-high MW compounds than those of medium-low MW (Gibert et al., 2015; Lai et al., 2015).

The UF and PAC/UF treatment processes had low removal rates for the MW fractions of 2560-2328, 1701-1543 and 868-790 Da. In the UF process, the removal rate was -10.10%, -10, 52%, and -12.42% for the respective fractions, and in the PAC/UF process it was 3.67%, 2.22% and 0.12%.

This indicates the low capacity of the UF membrane alone and PAC as the only pre-treatment to UF to remove organic compounds from the MW DOM approximately ≤ 2560 Da.

According to the above results, in one way or another, an indication of the efficiency of the UF, PAC/UF and PAC/C-S/UF treatment processes can be found in the removal of by-product precursors from disinfection. This is because it is well known that many of the materials directly responsible for this phenomenon are located in the fractions MW > 32900, 2560-2328, 1701-1543 and 868-790 Da.

Fig. 5

Molecular weight distribution of the effluent NOM for the 3 processes

Conclusions

During the period from 0 to 240 L of permeate volume, the TMP rate of the 3 processes increased as follows: PAC/C-S/UF < UF < PAC/UF. This indicates that the PAC/C-S/UF process was the most appropriate for the control of UF membrane fouling compared with the other 2 processes.

In the evaluation of the removal efficiency of the HoM and HiM fractions of the NOM, the treatment processes used in the present investigation increase in removal efficiency in the following order UF < PAC/UF < PAC/C-S/UF. This indicates that the hybrid treatment process of PAC/C-S/UF is suitable and highly effective to remove the HoM and HiM fractions of the NOM that absorb light at 340 nm. The PAC/UF hybrid treatment improves re-

moval of the HiM fraction in greater proportion than the HoM fraction of the raw water DOM.

The raw water studied in the present investigation contains DOM compounds of different molecular weight grouped into 4 fractions > 32900, 2560-2328, 1701-1543 and 868-790 Da.

The efficiency of removal of the NOM in MWD increases according to the processes studied as follows UF < PAC/UF < PAC/C-S/UF. This indicates that the hybrid treatment process of PAC/C-S/UF is suitable and highly effective to remove fractions > 32900, 2560-2328, 1701-1543 and 868-790 Da from the light absorbing NOM at 254 nm.

Acknowledgements

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Organinių junginių šalinimas iš Kolumbijoje esančių vandens telkinių, naudojant aklinojo rėžimo membraninę ultrafiltraciją

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Atlikti bandomieji-pilotiniai, natūralių organinių junginių šalinimo iš paviršinių vandens telkinių, tyrimai, naudojant tris skirtingus vandens valymo procesus: (1) ultrafiltaciją (UF); (2) ultrafiltraciją kartu adsorbcija miltelinio pavidalo aktyvintaja anglimi (MAA/UF); (3) ir naudojant mišrų valymo procesą t. y. kombinuojant ultrafiltraciją, koaguliaciją-sėsdinimą ir adsorbciją milteline aktyvintaja anglimi (MAA/K-S/UF). Pirmuoju atveju (UF) buvo naudojama tik ultrafiltracija, antruoju atveju (MAA/UF) prieš ultrafiltraciją vanduo buvo paruošiamas (apvalomas) adsorbuojant organines medžiagas aktyvintaja anglimi. Trečiuoju atveju (MAA/K-S/UF) prieš ultrafiltraciją vanduo buvo apdorojamas keliais būdais – pirmiausia adsorbcija aktyvintaja anglimi ir po to organinių medžiagų koaguliacija-sėsdinimu naudojant aliuminio sulfatą kaip koaguliantą. Lyginant pirmųjų dviejų metodų valymo efektyvumą, efektyviau organinės medžiagos buvo šalinamos MAA/UF metodo. Tyrimo rezultatai parodė, kad efektyviausias yra mišrus valymo procesas (MAA/K-S/UF), kurio metu ne tik efektyviausiai išvalomos organinės priemaišos, tačiau ir valymo metu išlaikomas stabilus transmembraninis slėgis (TMS) apie 0,5 baro, taip pat veikimo metu išlaikomas ir pastovus tekėjimo srautas 227 l/(m² · h). Taip pat tyrimai, parodė, kad tiek hidrofilinės tiek fidrofobinės organinės medžiagos gali būti išvalomos naudojant koaguliaciją. Tačiau gilesni koaguliacijos tyrimai atskleidė, kad hidrofobinės medžiagos koaguliacijos pagalba pašalinamos efektyviau negu hidrofilinės. Vertinant organinių medžiagų šalinimo efektyvumą pagal molekulinio svorio pasiskirstymą, paaiškėjo, jog MAA/K-S/UF proceso metu sėkmingiausia buvo išvalytos vidutinės ir didelės molekulinės masės junginiai (> 1701 DA), kai vidutinės ir mažos molekulinės masės organinių junginių (≤ 1701 DA) valymo efektyvumas mažėjo, mažėjant molekuliniam svoriui.

Raktiniai žodžiai: aklinojo rėžimo ultrafiltracija, natūralių organinių medžiagų šalinimas, molekulinės masės pasiskirstymas.