



# Study on the behavior of nanofiltration membranes using for chromium(III) recovery from salt mixture solution

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## HIGHLIGHTS

- ▶ Low pH and high salt concentration in chromium solution modified NF membrane surface
- ▶ Two washing procedures to reproduce the properties of NF membranes was analyzed
- ▶ Potential zeta, SEM and permeability of membranes was determined
- ▶ Membrane bathing with HCl reduced its scaling but not the surface charge reproduced
- ▶ Mixed HCl–NaOH bath leads both reduce of membrane scaling and recovery its charge

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## ABSTRACT

Results of studies of the effects of concentrate salt solutions characterized by low pH, on the nanofiltration membrane surface properties used for the separation of chromium(III) have been presented in this paper. It was shown that the low pH of the concentrate salt solutions and cleaning bath with hydrochloric acid irreversibly altered the charge of tested membranes. As the consequence an instability of permeability and selectivity of the membrane during the process was noticed. The effect of alkaline bath used after cleaning with a solution of hydrochloric acid to regenerate the surface charge of tested membranes was also examined. The results showed that the use of bath in the form of sodium hydroxide leads to the partial recovery of a low surface charge of the membranes. In addition a significant improvement in the stability of the tested NF membranes used for the separation of chromium(III) from concentrate salt solutions at low pH was observed. Moreover, SEM images obtained for the tested membranes cleaned with solutions of HCl and NaOH indicated no mechanical defects in the structure of the examined membranes.

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## 1. Introduction

Nanofiltration is a pressure driven process, which assures great process efficiency, as well as enables its great selectivity [1]. On account of these advantages, the nanofiltration is successfully applied to the treatment of surface and underground waters, above all for their softening [2–5] and for the separation of metal ions from industrial wastewaters [6–8]. Several other studies [9–11] as well as our previous works [12–14] demonstrate very interesting possibilities of nanofiltration application for chromium(III) recovery from concentrated salts mixture characterized by low pH. Such solutions are used, among others, in a tanning industry. Separation of chromium(III) from concentrated salts mixture depends on the feed composition [12,13] and properties of the used nanofiltration membranes [14]. The presence of mono- and multivalent negative ions and sufficiently high number of

monovalent positive ions produces a Donnan phenomenon which can be additionally increased by the selection of the nanofiltration membrane demonstrating the proper chemical-physical capacity of surface layer. The NF membrane characterized by a negative zeta potential [14] is especially useful for chromium(III) recovery from concentrated salts mixture characterized by low pH. The use of such membrane lowers its polarization causing the increase of permeate flux, and at the same time growth of chromium(III) concentration factor. However, even at optimum feed composition and the use of membrane with appropriate surface properties decrease of its productivities during the nanofiltration process was observed [12]. It was caused by the change of membrane zeta potential by the low pH. The consequence of this fact is the adsorption of ions on the surface and/or in the pores and the deterioration of the membranes selectivity [12]. The change in the membranes selectivity causes changes in the retention of feed components, which may lead to the membrane polarization increase and as a consequence to reduction of its permeability. Because the maintenance of high membrane permeability is preferred for chromium(III)

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recovery, it seems to be necessary to remove the adsorbed ions layer from the surface and/or NF membrane pores to keep low values of zeta potential.

The aim of this study was to analyze the changes of surface properties of nanofiltration membranes used for chromium(III) recovery from concentrated salt solutions at low pH and to examine the different washing procedures to reproduce the properties of the tested nanofiltration membranes – characteristic zeta potential, as well as their hydraulic permeability.

## 2. Experimental

The experiments were carried out at laboratory scale in cross flow cell made of stainless steel operated in batch mode with circulation presented in Fig. 1.

The nanofiltration of model concentrated salt solutions containing  $2 \text{ gCr}^{3+}/\text{dm}^3$ ,  $10 \text{ gCl}^{-}/\text{dm}^3$ ,  $10 \text{ gSO}_4^{2-}/\text{dm}^3$  and characterized by a  $\text{pH} \approx 4$  was conducted for transmembrane pressure on the level of 14 bar and retentate flow equal to  $800 \text{ dm}^3/\text{h}$ . The composition of solution and operation conditions were fixed based on our previous studies [12–14]. The temperature of feed solution during the process was maintained at  $25 \pm 1^\circ \text{C}$  by a thermostat. The feed solution was pumped from the feed tank toward to nanofiltration membrane, obtaining a retentate that was returned to the feed/retentate tank and a permeate that was collected in the permeate tank.

Two kinds of commercial nanofiltration flat sheet membranes (under symbol DL and HL) provided by GE Osmonics were used in the experiments. The tested nanofiltration membranes had an active layer made of the poly(piperazine-amide) (Fig. 2). The isoelectric point (IP) was less than 3.0 and 3.3 for DL and HL membrane, respectively [14–16]. Such membrane IP indicated on its negative zeta potential under experimental pH. The DL membrane had active three-layer – dense membrane structure, while the HL membrane had active two-layer – loose membrane structure. Each of these membranes has an effective area of  $0.0155 \text{ m}^2$ .

After nanofiltration the tested membranes were cleaned with acid and alkaline baths according with procedures presented in Tables 1 and 2.

pH of cleaning baths was chosen on the basis of the tested membranes characteristics regarding their chemical resistance [14]. Temperature of the acidic and alkaline cleaning bath was constant and equal to  $18 \pm 1^\circ \text{C}$ .

The changing of membrane permeability under the influence of concentrated salt solutions and the cleaning according to proposed procedures was analyzed based on the  $J_p = f(\Delta P)$  dependence designated for the deionized water ( $\text{TMP} = 10\text{--}24 \text{ bar}$ ,  $Q_R = 800 \text{ dm}^3/\text{h}$ ,  $t = 25 \pm 1^\circ \text{C}$ ) (Tables 1 and 2).

Samples of permeate and retentate have been collected for determination of chromium(III) concentration in well-defined time intervals.

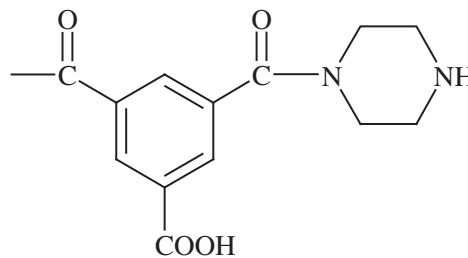


Fig. 2. Poly(piperazine-amide) formula [17–19].

After the end of the experiment, samples of permeate and retentate have been collected for determination of the chloride concentration.

The samples of permeate, feed and retentate have been analyzed using the following methods:

- chromium(III) – spectrophotometer NANOCOLOR UV/VIS using 1,5-difenylokarbazyde method with wave length  $\lambda = 540 \text{ nm}$ ,
- chlorides – the Mohr titration method.

The feed solution has been prepared using the following chemicals:  $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$  (Sigma-Aldrich), pure NaCl (Chempur®), pure  $\text{Na}_2\text{SO}_4$  (Chempur®) and the deionized water. The feed solution was characterized by  $\text{pH} \approx 4$ . For initial pH correction the pure HCl (Lachner) was used. The pH was measured by pH-meter (Mettler Toledo SevenEasy).

Membrane surface zeta potential was determined by streaming potential using an apparatus and the procedure described in the literature [20]. KCl (Chempur®) solution (0.001 M) was used as the electrolyte solution to measure the streaming potential of nanofiltration membranes. The pH was set by adding NaOH (Chempur®) and HCl (Chempur®). The zeta potential was calculated from the streaming potential using the Helmholtz–Smoluchowski equation taking into account a dielectric constant, viscosity and electrolytic conductivity of the solution.

The analysis of membranes surface was determined by scanning electron microscope PHENOM G2 (FEI). For SEM analysis, the deposition of a gold layer of about 2 nm in thickness was done using K550X Sputter Coater (Technologies Quorum).

## 3. Results and discussion

### 3.1. The effect of concentrated salt solution on nanofiltration membranes properties

In the first stage of the study, the influence of concentrated salt solutions on the surface properties of the tested DL and HL nanofiltration membranes was analyzed. For this purpose, nanofiltration membranes

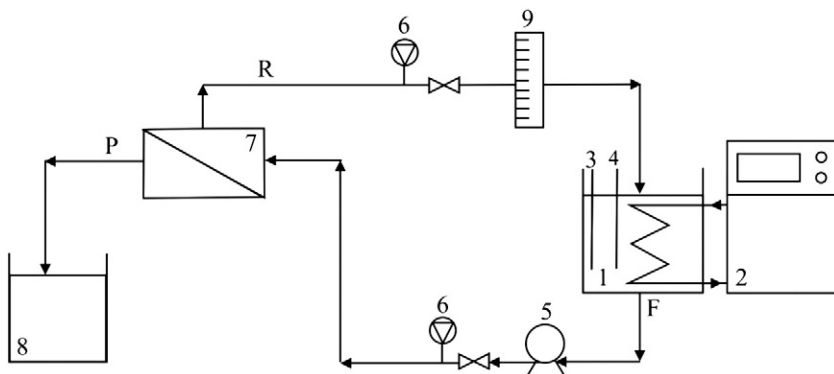


Fig. 1. Schema of the laboratory plant: 1 – feed/retentate tank, 2 – thermostat, 3 – measurement of temperature, 4 – measurement of pH, 5 – high pressure pump, 6 – manometer, 7 – NF membrane, 8 – permeate tank, 9 – flowmeter, P – permeate, F – feed, and R – retentate.

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