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Application of nanofiltration for removal of zinc from industrial wastewater

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ABSTRACT

Wastewater treatment which deals with pollution by heavy metals, especially pollution of zinc is still the main issue in many industries. It is necessary to apply effective separation technology for pollutant substances that enter into wastewater. The aim of the present study is to test a commercially available nanofiltration membrane (AFC 40) for the separation of zinc sulphate from aqueous solutions. The major focus of this study is to investigate the influence of various operating conditions on the membrane performance. Thereafter, the AFC 40 nanofiltration membrane was employed for experiments on real samples of industrial wastewater containing zinc. Experiments with binary aqueous solutions were performed at various process conditions. Important parameters such as concentration of zinc, transmembrane pressure, pH range of the solutions and feed flow rate were studied. High values of rejection were achieved during all the experiments. The rejection of the AFC 40 membrane was measured for real samples of zinc-containing industrial wastewater. High value of rejection during the experiments with real samples of wastewater was achieved. During all the experiments with real samples of industrial wastewater, the achieved value of rejection was above 98%. The results obtained shows that the tested commercially available nanofiltration membrane (AFC 40) is suitable for the removal of zinc from both model binary solutions and real samples of wastewater.

Keywords: Heavy metal; Zinc; Nanofiltration

1. Introduction

Contamination by heavy metals poses a high risk to the environment and human health. It represents a global problem. The main sources of pollution are found in various fields of human activity, especially in industries. Global industrial development contributes to the release of pollutants into components of the environment, primarily watercourses. Most wastewater pollution is caused by common metals, such as lead (Pb), copper (Cu), cadmium (Cd) and zinc (Zn) [1].

The major sources of heavy metals are tanning, petroleum refining, chemical manufacturing, electroplating, mining, the textile industry, fertilizer plants, the photographic process industry, battery manufacturing, metal and steel working and finishing and landfill leachates [2]. Other sources of heavy metals include vehicular traffic, the burning of fossil fuels, energy facilities, construction and demolition activity [3]. Heavy metals can accumulate in the human body and can cause damage to vital organs [4]. So, there is need to control the quality of drinking water. The limit for zinc in drinking water is 5 mg L^{-1} according to the National Secondary Drinking Water Regulations of the EPA [5]. The wastewater standard is 2.61 mg L^{-1} for zinc according to the EPA [6]. In the Czech Republic, the limits depend on the branch of industry. The allowable pollution for zinc is from 1.5 to 3 mg L^{-1} in surface and wastewater [7].

In wastewater treatment, the traditionally utilized processes include precipitation, ion exchange, electroplating, adsorption and membrane separation [8]. Membrane

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technologies have several advantages in comparison with other processes for the removal of heavy metals from wastewater. They are characterized by high efficiency, easy operation and low space requirements [9]. Pressure-driven membrane processes, including microfiltration, ultrafiltration, nanofiltration (NF) and reverse osmosis (RO), can be employed as treatment or pre-treatment technologies [10,11]. Combinations of different membrane processes [12,13] or with other processes [14] can also be applied.

NF is a very attractive pressure-driven membrane separation process because it can selectively retain divalent ions together with a low rejection of monovalent ions, and is operated at lower pressures when compared with RO. These features project NF as a promising, innovative and less energy-demanding separation technique that can be used in the treatment of drinking water and various industrial effluents [15,16]. The nominal molecular weight cut-off of NF membranes is within the range of 200–1,000 Da. The separation mechanism may be due to steric and "charge" effects. NF can be employed as a method for the removal of heavy metals from wastewater [2,17–22], especially for zinc [14,23–29].

The aim of this study is to experimentally test whether a commercially available NF membrane was applicable for the removal of zinc sulphate from aqueous solutions of model samples, and then to test its use on real samples of wastewater. The effects of various operating conditions on the membrane performance were investigated. This study is important because values of the rejection and other tested parameters of used membrane are not freely available and they are not declared by producer. It is important to experimentally test specific parameters and operating conditions of the entire NF system behaviour during experiments with model solutions and subsequently with real sample of industrial wastewater. Experiments were conducted at different concentration ranges from 25 to 150 mg L^{-1}, transmembrane pressures from 5 to 30 bar, a pH range of the solutions of 3–6.5 and various feed flow rates (9, 6 and 3 L min^{-1}).

2. Experimental setup

2.1. Material

A tubular NF membrane was utilized for all measurements. A commercially available type of membrane, AFC 40, PCI Membrane Systems (Poland), was utilized. This membrane is made of polyamide film with an effective membrane area of 240 cm^2 (two tubes, each with length of 30 cm and internal diameter of 1.25 cm). Other parameters of the tested membrane are summarized in Table 1.

Solutions of zinc sulphate ($ZnSO_4 \cdot 7H_2O$) were prepared from chemicals supplied by Lach-Ner, s.r.o. (Czech Republic) with the analytical grade (p.a.) purity and highly demineralized water (conductivity < 10 μS cm^{-1}). Sulphuric acid (96% H_2SO_4), which was used for the adjustment of pH, was supplied by Lach-Ner, s.r.o. (Czech Republic), also with the p.a. purity.

2.2. Experimental setup

The experimental setup is made of an NF apparatus (FT 18, Armfield, UK) and cooling equipment (TAEevo, Armfield, UK) (Fig. 1). The temperature of the feed solution was maintained at a constant value of 25°C. The volume of the feed was 10 L. The permeate was continuously weighed on digital scales (Balance KERN KB, Germany). The conductivity of the feed and the permeate were controlled by conductometers (WTW Cond 340i and WTW Cond 3210, Germany). Control of pH was performed using a pH meter (Accumet AB15 Basic, Fisher Scientific, USA). The concentrations of zinc in the samples were measured by inductively coupled plasma–optical emission spectroscopy (ICP–OES; Integra XL 2, GBC, Australia).

2.3. Methods

Two pieces of membrane were submerged into highly demineralized water (conductivity < 10 μS cm^{-1}). Before starting the main experiments, it was necessary to conduct compaction of the membranes. The tested membranes were stabilized for at least 2 h at the maximum pressure (30 bar) that was employed in this study. The permeate flow at different transmembrane pressures was measured by digital scales connected with personal computer (PC). Permeate flow was recalculated using density of permeate and membrane area

Table 1
Characteristics of the tested membrane

Structural parameters	
Membrane type	AFC 40
Material	Polyamide film
Maximum pH range	1.5–9.5
Maximum pressure (bar)	60
Maximum temperature (°C)	60
$CaCl_2$ retention (%)	60
Membrane surface charge (pH = 7)	Negative
Effective membrane area (cm^2)	240
Length of one tube (cm)	30
Internal diameter (cm)	1.25

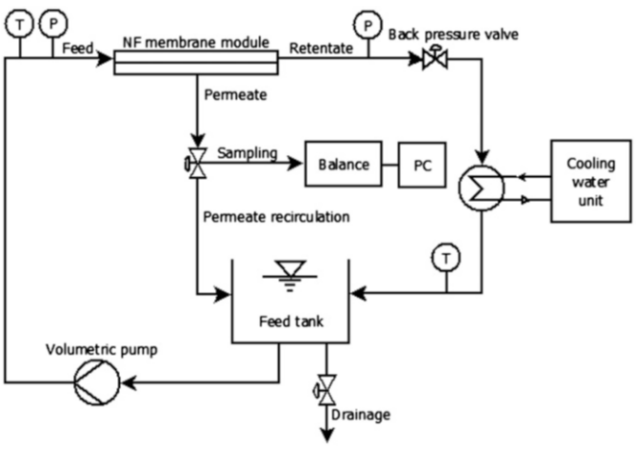

Fig. 1. Setup of nanofiltration experimental system.

to permeate flux. Permeate density was taken as being identical to pure water. After each experiment, the NF apparatus was cleaned with demineralized water until the permeate flux and permeability of the membrane were restored (3–4 h).

Experiments were performed at the following concentrations of zinc: 25, 50, 100 and 150 mg L^{-1}. Transmembrane pressures of 5, 10, 15, 20, 25 and 30 bar were applied on the NF apparatus. To investigate the maximum applicable pH range of the NF membrane, the pH values of the solutions in the experiments were adjusted to pH 3, 5 and 6.5. The dependence on the feed flow rate was also investigated by varying this parameter, with values of 9, 6 and 3 L min^{-1}. During all experiments, permeate fluxes were measured after stabilization of pressure and temperature and samples for ICP–OES analysis were collected after stabilization of the permeate conductivity. The last condition led to different duration of experiments.

2.4. Data analysis

By analyzing the solute concentrations in feed (c_F) and permeate (c_P), the efficiency of the membrane in the separation process can be evaluated by calculating the observed rejections as follows:

$$R_{obs} = 1 - \frac{c_P}{c_F} \tag{1}$$

In NF processes, the pressure applied on the feed side of the membrane leads to a solvent flow through the membrane pores accompanied by a partial permeation of the solutes. So, the solutes retained by the NF membrane are accumulated close to the membrane surface, and the solute concentration in the bulk of the feed (c_F) differs from the solute concentration in the near vicinity of the membrane surface (c_M). This phenomenon is called concentration polarization.

The intrinsic rejection (R_{int}), which characterizes the real performance of the membrane, can be evaluated (based on the film model for concentration polarization) by the following equation:

$$R_{int} = 1 - \frac{c_P}{c_M} = \frac{R_{obs}\exp\left(\frac{J}{k}\right)}{1 - R_{obs}\left[1 - \exp\left(\frac{J}{k}\right)\right]} \tag{2}$$

where c_P, c_F and c_M represent the concentrations of the solute in the permeate, in the feed (bulk) and in the feed solution at the membrane surface, J is the permeate flux and k is the mass transfer coefficient in the polarization layer.

The mass transfer coefficient (k) is calculated from the Sherwood relationship, generally calculated as:

$$Sh = \beta Re^{a} Sc^{b} \left(\frac{d_h}{L}\right)^{c} \tag{3}$$

where coefficients β, a, b, c are dependent on experimental conditions.

For our experimental conditions, turbulent flow (Re = 17,500 at 9 L min^{-1}) in the channel was used as the Deissler correlation for turbulent flow in channels or tubes [15]:

$$Sh = 0.023\,Re^{0.875}\,Sc^{0.25} \tag{4}$$

The Reynolds (Re), Schmidt (Sc) and Sherwood (Sh) numbers in Eq. (3) are given by:

$$Sh = \frac{k d_h}{D},\ Re = \frac{u \rho d_h}{\eta},\ Sc = \frac{\eta}{\rho D} \tag{5}$$

where u indicates the fluid velocity in the channel, whose hydraulic diameter is d_h (the inner diameter of the tubular membrane in our case), D is the diffusion coefficient of the solute and η and ϱ are the dynamic viscosity and density of the aqueous solution, respectively.

The solution properties (density, viscosity) utilized for computing the real rejections were taken as being identical with those of pure water, and the salt diffusion coefficient (D) was calculated based on the diffusion coefficients (D_+, D_-) and valences (z_+, z_-) of the individual ions (cation and anion) using the relationship:

$$D = \frac{(z_+ - z_-) D_+ D_-}{z_+ D_+ - z_- D_-} \tag{6}$$

where the diffusion coefficients are 7.03 × 10^{-6} and 1.065 × 10^{-5} cm^2 s^{-1} for zinc and sulphate, respectively [30].

3. Results and discussion

3.1. Dependence of rejection on concentration

The permeate flux was measured as a function of the pressure difference. The line in Fig. 2 indicates the pure water flux (PWF). The permeability (7.14 L h^{-1} m^{-2} bar^{-1}) is the slope of this line. The feed volumetric flow was constant, at 9 L min^{-1}, during this experiment. This value of the feed flow rate was chosen to minimize the effect of concentration polarization. In the concentration range of zinc sulphate (25–150 mg L^{-1}),

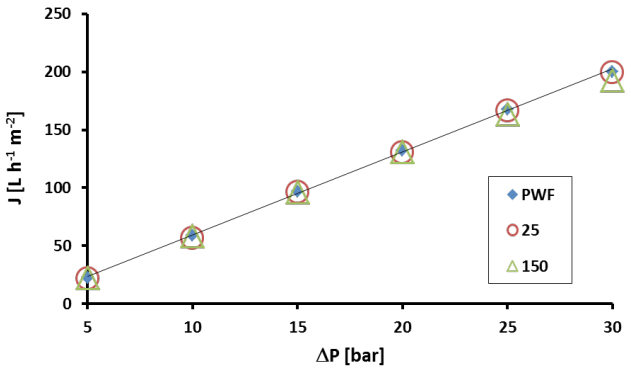

Fig. 2. The comparison of fluxes, PWF, c = 25 and 150 mg L^{-1}, membrane AFC 40, feed flow 9 L min^{-1}, T = 25°C.

it can be assumed that the permeate flux varied very little, because very low concentrations were utilized. For this reason, small differences in osmotic pressure were the driving force, while the net pressure difference was almost constant (in the range of experimental error).

The observed rejection increased very slightly with increasing concentration, especially at the smallest pressure difference (from 97.5% to 98.5% at 5 bar). The observed rejections at higher permeate fluxes were almost constant, and independent of the feed concentration (Table 2). Rejection was higher than 98% with all tested concentrations, except for the experiment with the lowest pressure and lowest concentration. Figs. 3 and 4 illustrate a comparison between the intrinsic and the observed rejection for the concentrations of 25 and 150 mg L^{-1}. The intrinsic rejection is calculated on the basis of Eq. (2). It is obvious that the decrease of the observed rejection at higher permeate fluxes was caused by concentration polarization, because the intrinsic rejection increased further. When testing the influence of other parameters (pH, feed flow rate and testing of real wastewater), values of observed rejection are shown.

3.2. Dependence of rejection on feed flow rate

The dependence of the observed rejection on the feed flow rate was investigated as the second parameter. The conditions of the experiment were as follows: feed Zn(II) concentration, ~100 mg L^{-1}; pressure, 10 or 20 bar; pH, 6.5 and temperature, 25°C. The feed flow rate was set at three values: 9, 6 and 3 L min^{-1}. The dependence of observed rejection on the feed flow rate at different pressures (10 and 20 bar) is summarized in Tables 3 and 4.

Rejection was slightly decreased when the feed flow rate was reduced from 9 to 6 L min^{-1} at 10 bar. There was an obvious reduction in the rejection when the feed flow rate was reduced from 6 to 3 L min^{-1}. Increasing the pressure from 10 to 20 bar led to an increased concentration polarization effect and a greater influence of the feed volumetric flow.

Table 2
Observed rejections for all measured Zn(II) concentrations

Pressure (bar)	Rejection (%)			
	Zn(II) concentration of feed (mg L^{-1})			
	25	50	100	150
5	97.50	98.10	98.60	98.50
10	98.10	98.40	98.60	98.60
15	98.40	98.60	98.60	98.50
20	98.00	98.50	98.60	98.50
25	98.00	98.60	98.60	98.50
30	98.00	98.40	98.40	98.40

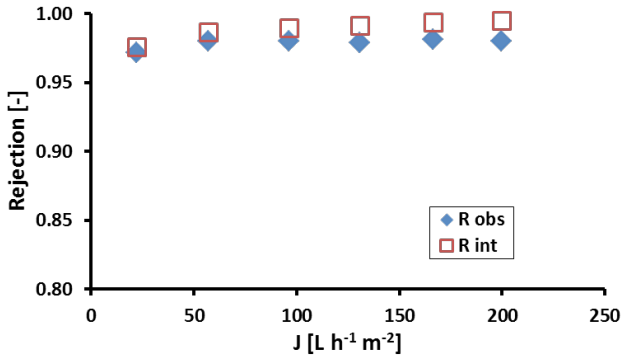

Fig. 3. Dependence of rejection on permeate flux, membrane AFC 40, c = 25 mg L^{-1}, feed flow 9 L min^{-1}, pH 6.5, P = 5–30 bar, T = 25 °C.

3.3. Dependence of rejection on pH

The tested AFC 40 NF membrane can be employed in the pH range of 1.5–9.5. Experiments were performed at pH values of 3, 5 and 6.5. The conditions of the experiment

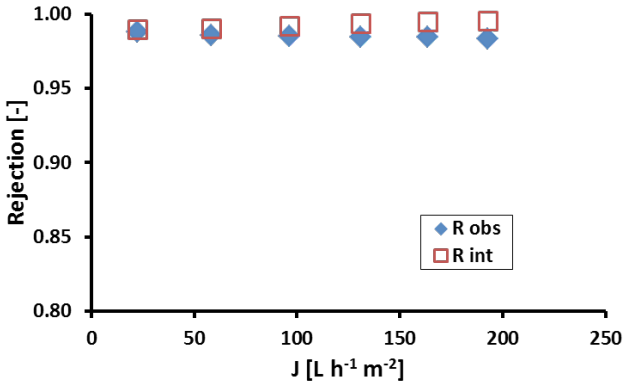

Fig. 4. Dependence of rejection on permeate flux, membrane AFC 40, c = 150 mg L^{-1}, feed flow 9 L min^{-1}, pH 6.5, P = 5–30 bar, T = 25°C.

Table 3
Dependence of observed rejection on feed flow, P = 10 bar, membrane AFC 40, pH 6.5, T = 25°C

Zn(II) concentration (mg L^{-1})	Feed flow rate (L min^{-1})		
	9	6	3
Feed	99.49	98.95	100.60
Permeate	1.330	1.475	2.097
Rejection (%)	98.66	98.51	97.92

Table 4
Dependence of observed rejection on feed flow, P = 20 bar, membrane AFC 40, pH 6.5, T = 25°C

Zn(II) concentration (mg L^{-1})	Feed flow rate (L min^{-1})		
	9	6	3
Feed	100.00	100.00	105.90
Permeate	1.519	1.975	3.827
Rejection (%)	98.48	98.03	96.39

were as follows: feed concentration, ~100 mg (Zn) L^{-1}; pressure, 10 bar; feed flow, 9 L min^{-1} and temperature, 25°C. The dependence of the rejection on the variation in pH is presented in Table 5.

From the rejection values obtained, it is obvious that the highest rejection was obtained at pH 3. The smallest rejection was achieved at pH 5, which is close to the isoelectric point of this membrane (the point at which the membrane does not carry charge, which is at pH 4.1) [16]. Below this pH (i.e., for the experiment at pH 3), the membrane was positively charged and rejection increased again as a result of repulsive electrostatic forces. At pH 3, the feed concentration was higher due to evaporation during the long experimental period, which concentrated the feed. This may have increased the rejection (see Section 3.1); nevertheless, we assume that the effect is not significant. At pH 6.5, membrane has negative charge and rejection is enhance by electric charge also (now for sulphate). It can be seen from the comparison of rejection at pH 5 and 6.5.

3.4. Real sample of industrial wastewater

Real wastewater was obtained from the production of viscose rayon; it had been designated as suitable washing water. Prior to the NF experiment, the real sample was filtered utilizing a filter with a pore size of 0.7 μm for the removal of viscose residues. The concentration of zinc before filtration was 64.90 mg L^{-1} and was 61.88 mg L^{-1} after filtration. This decrease may be attributed to the adsorption of zinc on the viscose. The concentrations of other metals were determined as follows: for Ni, 0.027 mg L^{-1}; for Fe, 0.54 mg L^{-1} and for Cr, <0.01 mg L^{-1}. The values of conductivity were in the range of 4.05 ± 2.02 mS cm^{-1}. The other characteristic parameters of the composition of the real industrial wastewater were as follows: COD = 93.7 ± 6.3 mg L^{-1}, BOD = 27.7 ± 3.5 mg L^{-1} and SO_4^{2-} = 95.35 mg L^{-1}. A new sample of the AFC 40 NF membrane was utilized for this experiment, and the pH of the wastewater was not adjusted. The conditions of the experiment were as follows: volume of feed, 8.5 L; pressure, 10, 15 and 20 bar; pH, 4.5; feed flow rate, 9 L min^{-1} and temperature, 25°C. The experiment lasted for approximately 2 weeks, with a minimum of 6 h of work on each day (except weekends). The total working time was 55.5 h.

Samples were collected after reaching a stable value (i.e., the same value four times) of the permeate conductivity. The concentration and rejection of Zn at different pressures are presented in Table 6.

It can be seen that the feed concentration was considerably higher than 61.88 mg L^{-1}; this is again due to evaporation during the long experimental period. From the high values of rejection, the high ability of the tested membrane to separate zinc from wastewater at different pressures is evident.

The average values of permeate flux for the demineralized (PWF) and wastewater were measured after stabilization of pressure and temperature. A comparison of the PWF and the permeate flux of the real sample of wastewater is presented in Table 7.

From the comparison of the PWF and the values from the experiment, it can be seen that there was a tendency to fouling. It has not yet been tested whether this was of reversible or irreversible type. For real applications, more experiments will be necessary, especially for the development of superior pre-treatment methods (for instance, using a 0.1 μm filter instead of 0.7 μm) or simple and effective methods for cleaning the membrane.

Table 5
Dependence of observed rejection on pH for membrane AFC 40, cZn(II) = 100–120 mg L^{-1}, P = 10 bar, T = 25°C

Zn(II) concentration (mg L^{-1})	pH		
	3	5	6.5
Feed	120.00	100.00	101.10
Permeate	0.850	1.800	1.400
Rejection (%)	99.29	98.20	98.62

Table 6
Concentration and observed rejection of Zn from the real sample of wastewater, membrane AFC 40, P = 10, 15 and 20 bar, pH 4.5, feed flow 9 L min^{-1}, T = 25°C

Pressure (bar)	Zn(II) concentration (mg L^{-1})		Rejection (%)
	Feed	Permeate	
10	99.10	1.235	98.75
15	92.84	1.121	98.79
20	91.82	1.140	98.76

Table 7
Pure water flux and permeate flux during experiment with real sample of wastewater, membrane AFC 40, P = 10, 15 and 20 bar, feed flow 9 L min^{-1}, T = 25°C

Flux (L h^{-1} m^{-2})	Pressure (bar)		
	10	15	20
Demineralized water	66.2	104.5	141.4
Real wastewater	54.5	85.9	120.5
Decrease (%)	17.7	17.8	14.8

4. Conclusion

The selected membrane, AFC 40, is suitable for the separation of zinc from aqueous solutions. It is characterized by high permeate flow and high rejection. The rejection rates achieved during the experiments were higher than 98%, except at the lowest pressure and lowest concentration (25 mg L^{-1} at 5 bar). Rejection is decreased with decreasing zinc concentration in the feed. The observed rejection depended on the feed flow rate, where the highest rejection (98.66%) was achieved at a feed flow rate of 9 L min^{-1} and a pressure of 10 bar. The highest value of rejection, 99.29%, was achieved at pH 3.

Thereafter, the tubular AFC 40 NF membrane was employed for experimentally testing a real sample of industrial wastewater. The experiment with the real sample of wastewater revealed that NF using AFC 40 membrane was an effective process for the separation of zinc from industrial wastewater. In proof of this, it obtained values of rejection

were higher than 98% during all the experiments. The AFC 40 membrane was confirmed by the experiment with real wastewater to be suitable for the removal of zinc from wastewater.

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Symbols

c_F	—	Concentration in feed
c_M	—	Concentration at the membrane wall
c_P	—	Concentration in permeate
J	—	Permeate flux
k	—	Mass transfer coefficient
P	—	Pressure
Re	—	Reynolds number
R_{int}	—	Intrinsic rejection
R_{obs}	—	Observed rejection
Sc	—	Schmidt number
Sh	—	Sherwood number
t	—	Temperature

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