

pH and Temperature Effect on Transport Parameters of Zn Retention by NF Membrane

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Abstract: The objective of our study was to take knowledge about the possible influence of various operating parameters on the performance of the membrane process through evolution of transport parameters in order to understand the mechanism which governs the separation of the zinc ions by nanofiltration. The operating parameters to be considered in this study were temperature and pH. The experiments were performed on zinc nitrate solutions prepared at 10 mg/L in Zn^{2+} . The results have shown that an increase in the temperature generates a reduction in the solute retention (68% at 13°C and 23% at 36 °C). The study on the pH effect have shown that the Zn ions retention decreases when the pH increases (90% at pH 2 and 60% at pH 6), while that of the nitrate ions retention increases (17% at pH 2), reached a maximum (67% at pH 4) and then decreases (56% at pH 6). These results were related to the calculation of solute transport parameters in order to know more comprehensive retention and transfer mechanism.

Key words: Nanofiltration, zinc, temperature, pH, transport parameter.

Introduction

The water pollution by zinc is primarily due to the rejections of the industrial effluents. These effluents are not treated in a satisfactory way and generate a pollution of the rivers which deposit the muds polluted by metals, in particular zinc. This has aroused an increasing attention, translated by the installation of a legislation obliging the industrialists responsible for this pollution, mainly those of the surface treatment, to face this problem. This legislation imposes limiting contents in the rejections as well as a system of royalty aiming at taxing the industrialists responsible in the event of non-compliance with standards.

To better produce and less pollute are the challenges with which the industrialists of all sectors are confronted,

in particular those of the surface treatments, by the political officials responsible for the environment, as well as by the public opinion. The constraints in legislative and normative forms are increasingly drastic.

To solve this problem and answer the increasing severity of the standards of the thresholds of rejection, imposed by the national and European public authorities, the industrialists are confronted with the implementation of increasingly sophisticated processes of purification. The membrane separation process using highly effective NF membranes is considered a competitive method for the wastewater treatment.

Conceived at the origin for desalination or softening water, the nanofiltration showed thereafter an important potential for the applications in the liquid waste processing. The choices of the membrane and the

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List of symbols

J_v :	permeate flux, $\text{m}^3 \cdot \text{m}^{-2} \cdot \text{s}^{-1}$
S :	surface membrane area, m^2
TR :	observed retention
C_p :	salt concentration in the permeate, $\text{kg} \cdot \text{m}^{-3}$
C_0 :	salt concentration in the feed solution, $\text{kg} \cdot \text{m}^{-3}$
D_S :	diffusion coefficient, $\text{m}^2 \cdot \text{s}^{-1}$
K_S :	sorption coefficient, $\text{m} \cdot \text{s}^{-1}$
L_p :	water permeability, $\text{m}^3 \cdot \text{m}^{-2} \cdot \text{s}^{-1} \cdot \text{Pa}^{-1}$
ΔP :	transmembrane pressure, Pa
$\Delta \pi$:	osmotic pressure, Pa
ΔP_{eff} :	effective pressure, Pa
T :	operating temperature, $^{\circ}\text{K}$
T_{ref} :	reference temperature, $^{\circ}\text{K}$
T_{CF} :	temperature correction factor
η :	dynamic viscosity, Pa.s
E_L :	apparent activation energy, $\text{J} \cdot \text{mol}^{-1}$
R :	universal gas constant, $\text{J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$
J_s :	solute flux, $\text{kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}$
B :	solute permeability, $\text{m} \cdot \text{s}^{-1}$
X :	total fixed charges density, $\text{mol} \cdot \text{m}^{-3}$
δ :	length of active layer, m

geometry of the module constitute the first stage in the implementation of a process of nanofiltration and there exists today a certain choice for material constitutive of a membrane. However, if the permeability and the MWCO are always specified by the manufacturer, other properties related to the characteristics of the material membrane surface or the transfer of molecules or salts are not provided. The choice of a membrane for a given application thus remains a difficult step.

Indeed, considering the complexity of the composition of certain industrial effluents, this step must be carried out according to effluent treatment characteristics. The wastewater treatment of the industrial rejections by nanofiltration allows extraction of water or solvents and concentration of the aqueous solutions in order to limit volumes, to even recycle solutes or molecules with strong added value. It is thus necessary to develop specific tools, if possible simple and reliable, in order to acquire additional data on the characteristics of materials used and their behaviour towards solutions. Therefore, the physicochemical study of the phenomena occurring on the surface of the membrane by experimental studies becomes necessary to understand the mechanisms governing the transfer of the species within material.

The objective of this research is to study the influence of temperature and pH on the behaviour of transport parameters in order to know more comprehensive retention mechanism and membrane performances.

Materials and Methods

Description of the Pilot

The pilot used is a commercial apparatus Millipore Proscale with 12 L capacity (Figure 1). It is equipped with a spiral wound polymeric membrane (Nanomax 50, Millipore USA) with a filtration area of 0.37 m^2 . The Nanomax 50 is a composite membrane having a negatively charged thin skin layer ($0.4 \mu\text{m}$) made of polyamide arylene on a polysulfone support layer. The data of the manufacturer announce a MWCO about 300 Da for uncharged solutes and a pore diameter of 0.5 nm.

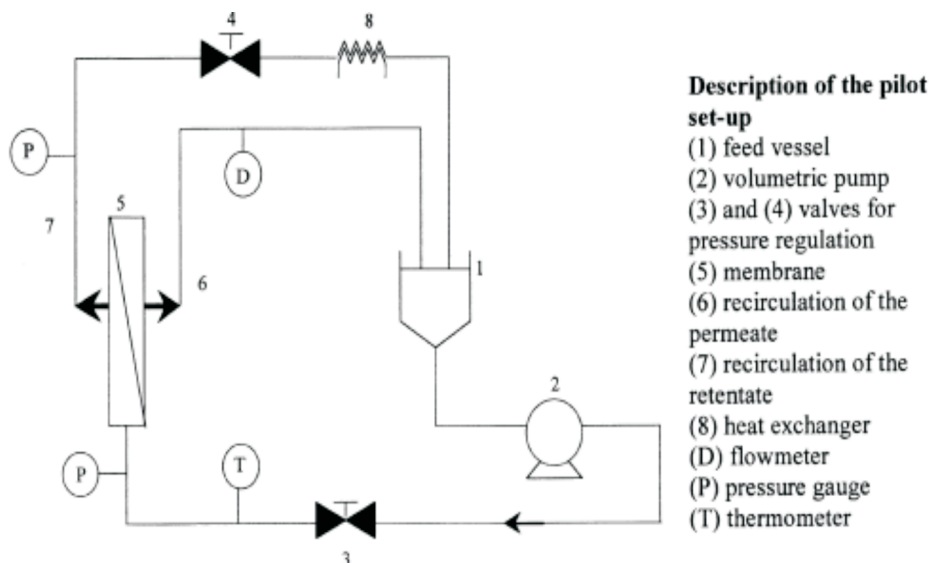


Figure 1: The Nanofiltration pilot set-up.

Procedure

The experiments were carried out on 4 L of solution containing zinc salts ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) at 10 zinc mg/L. The experiments were performed in a batch circulation mode. Both permeate and retentate were returned to the feed vessel in order to keep a constant concentration. These solutions were prepared in demineralised water (conductivity = $1\ \mu\text{S}/\text{cm}$). Permeate and concentrate were sampled after 1 h of filtration in order to measure solute concentration. After removing the feed solution from the module, the system was rinsed with acid solution (pH 2) whose anion corresponds to the salt of zinc used. It was then rinsed with water until the conductivity and the pure water fluxes were restored in order to ensure an efficient cleaning.

Analytical Methods

The Zn^{2+} concentrations were measured by atomic absorption spectrophotometer (Varian AA-1275) and the associated anions concentrations by ionic chromatography (Dionex-120). Measurements of the pH were carried out using a pH-meter, type CyberScan model 510. A conductimeter, mark MetreLab CDM 210, was used to measure conductivity of the solutions and to check the cleaning of membrane after each handling.

Analysis of the Results

The two parameters were taken into account:

The permeat flux J_v was determined by measuring the volume (ΔV) of permeate collected in a given time (Δt) interval by the relation:

$$J_v = \frac{\Delta V}{(\Delta t \times S)} \quad (1)$$

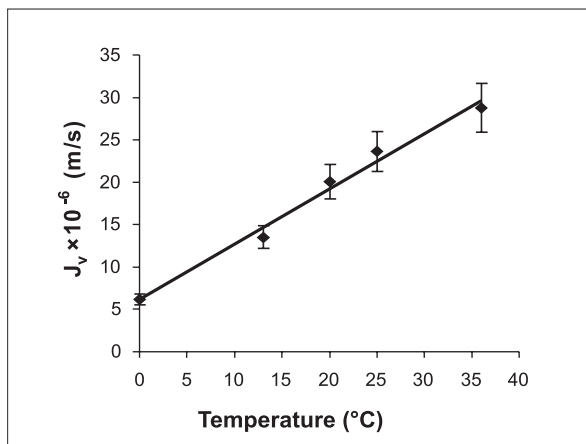


Figure 2: Effect of the temperature on permeate flux.

The observed retention (TR) was calculated by the following relation:

$$TR (\%) = \left(1 - \frac{C_p}{C_0} \right) \times 100 \quad (2)$$

Results and Discussion

Influence of the Temperature

These experiments were carried out with solution of zinc nitrate at 10 mg/L, under pressure of 8 bars, pH equal to 6 and temperature ranging from 13 to 36°C . The results obtained are presented in Figures 2 and 3.

As it can be seen from Figure 2, the first awaited result is that an increase in the temperature also involves an increase in total flow. Figure 3 illustrates a linear correlation between solute retention and temperature. The same results were found by Wittman (1998). The slope ($\Delta TR/\Delta T$) was about 2% per Kelvin. However, according to other works that dealt with the influence of temperature on the performance of nanofiltration membranes (Maurel, 1993; Boucard, 2000), it was difficult to draw general conclusions. For some membranes, the retention decreases, whereas it seems to remain constant for other membranes. These evolutions could find an explanation in the evolution of the viscosity of solvent and in the calculation of the solute transport parameters such as diffusion coefficient (D_s) and sorption coefficient (K_s) with temperature variation. Indeed in all the models of the transport of solvent, the flux of permeate is described by an equation equal or similar to those of the solution-diffusion models:

$$J_v = L_p(\Delta P - \Delta \pi) = L_p \cdot \Delta P_{\text{eff}} \quad (3)$$

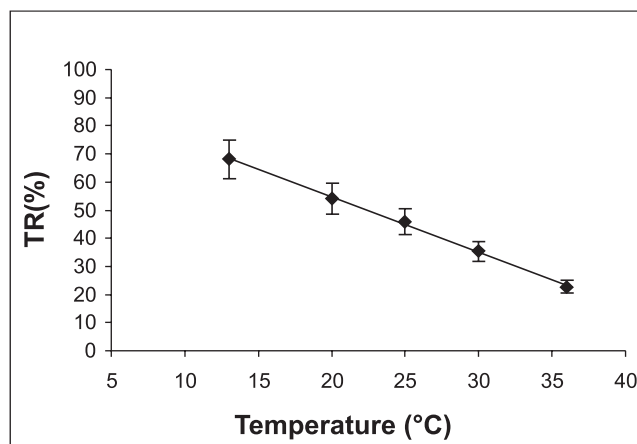


Figure 3: Effect of the temperature on the salt retention.

In all membranes separation processes driven by a pressure gradient, the permeability is generally expressed by the following equation:

$$Lp(T) = Lp(T_{ref}) \cdot T_{CF} \quad (4)$$

This gives for solvent flux:

$$J_v = Lp(T_{ref}) \cdot T_{CF} \cdot \Delta P_{eff} \quad (5)$$

In microfiltration and ultrafiltration, where the mass transfer is convective, the influence of the temperature on the permeability is determined by the relation between the viscosity and the temperature, which means for the T_{CF} :

$$T_{CF} = \frac{\eta(T_{ref})}{\eta(T)} \quad (6)$$

On the other hand, in reverse osmosis and nanofiltration, the influence of the temperature on the permeability is more important. The difference is explained by the diffusive character of the mass transfer. This influence can be described by a correlation of the type of Arrhenius equation (Mehdizahid, 1990), which gives for the T_{CF} :

$$T_{CF} = e^{-\frac{E_L}{R} \times \left(\frac{1}{T_{ref}} - \frac{1}{T} \right)} \quad (7)$$

In the literature, we can find that the values of the activation energy are varying from 20,785 kJ/mol to 29,099 kJ/mol. In our case the activation energy was found to be equal to 23,975 kJ/mol.

The viscosity of a liquid can be related to the permeability and the temperature according to the following equation (Maurel, 1993):

$$\frac{Lp(T)}{Lp(T_{ref})} = \frac{\eta(T_{ref})}{\eta(T)} = e^{-\frac{E_L}{R} \times \left(\frac{1}{T_{ref}} - \frac{1}{T} \right)} \quad (8)$$

$$\text{where } J_v = Lp(T_{ref}) \times \frac{\eta(T_{ref})}{\eta(T)} \times \Delta P_{eff} \quad (9)$$

According to equation (9) the permeate flux is inversely proportional to the solvent viscosity. The viscosity decreases when the temperature increases. This diminution generates an increase in flux and makes easier the transport of the fluid through membrane.

The solute diffusivity (D) also increases with the temperature, promoting thereby the transport and subsequently a decline in retention. This increase

generally obeys the Arrhenius's law and follows the equation below:

$$D = D_0 \exp\left(-\frac{E_a}{RT}\right) \quad (10)$$

Moreover the pore diameter of the membrane increases with the temperature thus supporting the access of the ions towards the functional groups in the pore and the reduction in their retention while decreasing the steric effects (Sharma et al., 2006).

The coefficient of mass transfer and the permeability can be calculated by the following formula:

$$\begin{aligned} J_s &= \frac{K_s \cdot D_s}{\delta} \times (C_0 - C_p) \\ &= B \cdot (C_0 - C_p) = J_v \cdot C_p \end{aligned} \quad (11)$$

Thus, the retention can be deduced from the following equation:

$$R = 1 - \frac{B}{B + J_v} \quad (12)$$

The results are grouped in Table 1. According to Table 1, results are consistent with the previous discussion. An increase in the temperature in feed solution causes an increase in the permeability and the transfer coefficient and, thereafter, facilitates the passage of salt through the membrane and solute retention decreases.

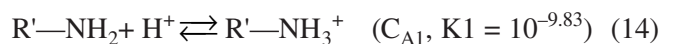
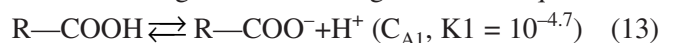
Table 1: Values of the retention, the permeability and the sorption coefficient according to the temperature variation

T (°K)	286	293	298	310
TR (%)	68.12	20.05	46	23
K_s (10 ⁶ m/s)	10.53	25.39	32.33	48.64
B (10 ⁶ m/s)	6.34	9.44	11.13	13.54

Influence of the pH

The effect of the pH on the retention has been studied by several authors. It depends specifically on the nature of the membrane material (Hagmeyer and Gimbel, 1999; Capelle et al., 2002) and the nature of the species in solution (Hafiane et al., 2000).

In our case, the active layer of the used membrane Nanomax 50 contains groups of carboxylic acid and secondary amine. These chemical function groups can react according to the following acid-basic equilibrium:



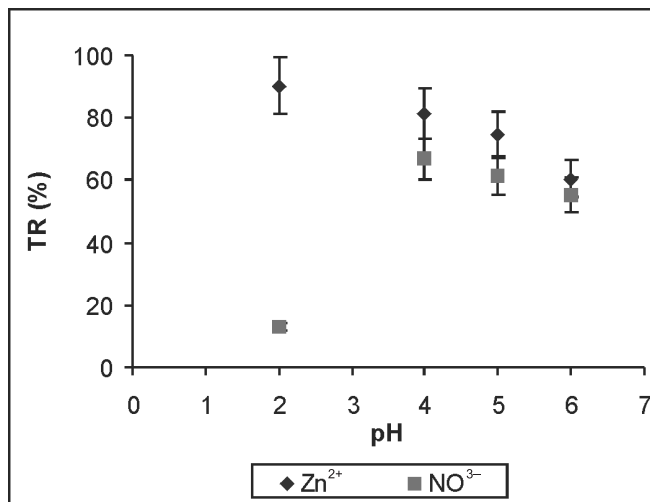


Figure 4: Retention of zinc and nitrates ions as a function of pH.

Based on the hypothesis that the adsorption of co-ions of the electrolyte is negligible compared to irradiation sites $R-COOH$ and $R'-NH_2$, the total fixed charges density $X(z)$ (mol/m³) of active layer can be written as:

$$X(z) = -[R-COO^-](z) + [R'-NH_3^+](z) \quad (15)$$

For a certain value of pH, called isoelectric point IEP, the electric charges balance reveals an overall null charge ($X = 0$). IEP depends on the nature of material membrane, possibly of the chemical treatment which it underwent during its preparation but also of the composition of the electrolytic solution in contact of material membrane. Thanks to its amphoteric character, the Nanomax 50 membrane, according to the pH, can present positive or negative charge and obviously a non-electric charge at pH equal to IEP value (Jeantet and Maubois, 1995) which was equal to 4.2 in the case of our used membrane.

Figure 4 shows that the retention of zinc ions decreases when the pH increases, meanwhile the retention of nitrate ions increases reaching a maximum and then decreases. According to the pH range from 4 to 6, the same evolution was reached for the nitrate ions retention and for zinc ions retention with a gap decrease as the pH increases until a gap equal almost zero at pH 6. The same results were found by Ahn et al. (1999) while studying the pH effect on the retention of $NiCl_2$. Results were also found for $NaCl$ salts (Lefebvre, 2002), $Cd(NO_3)_2$ salts and $Cu(NO_3)_2$ salts (Mehiguene et al., 1999). However, it was not observed that a minimal retention marked well at the isoelectric point of the membrane which is generally the case in nanofiltration (Combe et al., 1997; Chevalier, 1999; Nystöm et al., 2000; Wang, 2002; Ballet et al., 2004; Quin et al., 2004; Ozaki et al., 2002).

Indeed in acid medium, the higher value of Zn^{2+} retention and the lower value of NO_3^- retention obtained show the considerable role of the protons present in the medium. This notable difference of the retention of the two ions lets one suppose that the membrane was positively charged.

Indeed when the pH decreased, the dissociation of the carboxylic groups was removed and replaced simultaneously by the protonation of the amines groups. Consequently, there was a change of the charge sign and an increase in the positive charge of the pores of the surface membrane; thus, increasing the Zn^{2+} retention and decreasing the NO_3^- retention.

The reduction in this retention can be explained by the fact that under these conditions, separation depends only on the species size (r) and their diffusion coefficient (D).

Protons which are characterized by D equal to $9.34 \cdot 10^{-9} \text{ m}^2 \cdot \text{s}^{-1}$ and r equal to 0.053 nm, that cross the membrane more easily than Zn^{2+} ions which are characterized by D equal to $0.72 \cdot 10^{-9} \text{ m}^2 \cdot \text{s}^{-1}$ and r equal to 0.074 nm. Then, these protons take with them the nitrate ions to restore the electroneutrality.

In contrast, the increase in pH promotes the dissociation of carboxylic groups and the deprotonation of amine groups thus encouraging an increase in the negative charge of the membrane and subsequently a decreasing of Zn^{2+} ions retention. In this case, membrane attracts more charged cations, contributing to fast partial neutralization surface charges responsible for the electrostatic exclusion, thus promoting their transportation.

In comparison to our study on the pH effect, Tanninen and Nyström (2002) studied the change in the retention of $NaNO_3$ as a function of pH ranging from 0.5 to 7 for membrane Desal 5 DK and have found that the retention of Na^+ reaches a maximum at pH close to 2.5 (IEP for the membrane Desal 5 DK equal to 4) and then decreases. They explained this maximum by the double layer phenomena.

In contrast, during the study of the pH effect on the retention of KCl , $CaCl_2$ and $MgSO_4$, Teixeira et al. (2005) noted that the retention increases with pH without passing by a minimum at IEP. These authors have explained this behaviour by mechanism of membrane pore size and conformation pore variation. This same report was found and published by Childress and Elimelech (2000), Schaep et al. (2001), Oak et al. (1997) and Qin et al. (2003).

Nevertheless, Labbez et al. (2002) have found during their work on salts (KCl , $LiCl$, $MgSO_4$, $MgCl_2$ and

K₂SO₄) by the membrane TiO₂ that retention for symmetrical salts pass through a minimum corresponding to IEP. (The same explanation was given by the authors cited above.) But for asymmetrical salts the retention varies slightly according to the pH (a reduction for MgCl₂ and an increase for K₂SO₄). These authors have explained this effect by the fact that the membrane is slightly positive in the vicinity of IEP (6.2) for K₂SO₄ and slightly negative for MgCl₂. The low charge of the membrane in the case of these two salts was attributed to adsorption of ions Mg²⁺ and SO₄²⁻.

Based on the above we cannot generalize the behaviour of pH on the retention of salts beyond the isoelectric point; however, according to the work mentioned above, the variation in pH plays a very important role for membrane charge and subsequent salts retention.

Conclusion

The retention of zinc nitrate decreases as a function of temperature. Two hypotheses to explain this decrease were given. The first is that the ions diffusion coefficient increases with the temperature thus promoting their transport and subsequently their retention. The second hypothesis was attributed to the fact that the radius of the pores of the membrane increases with the temperature, which facilitates the passage of ions through it, thus increasing their concentration in the permeate flow and reducing their rejection. We noticed also that the Zn²⁺ retention decreases when pH increases from 2 to 6. This result was attributed to the membrane charge. Indeed, the interaction between the membrane and zinc ions is weak above the IEP allowing their transport through the membrane.

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Asian Journal of Water, Environment and Pollution



Aims and Scope

Asia, as a whole region, faces severe stress on water availability, primarily due to high population density. Many regions of the continent face severe problems of water pollution on local as well as regional scale and these have to be tackled with a pan-Asian approach. However, the available literature on the subject is generally based on research done in Europe and North America. Therefore, there is an urgent and strong need for an Asian journal with its focus on the region and wherein the region specific problems are addressed in an intelligent manner. In Asia, besides water, there are several other issues related to environment, such as; global warming and its impact; intense land/use and shifting pattern of agriculture; issues related to fertilizer applications and pesticide residues in soil and water; and solid and liquid waste management particularly in industrial and urban areas.

Asia is also a region with intense mining activities whereby serious environmental problems related to land/use, loss of top soil, water pollution and acid mine drainage are faced by various communities.

Essentially, Asians are confronted with environmental problems on many fronts. Many pressing issues in the region interlink various aspects of environmental problems faced by population in this densely habited region in the world. Pollution is one such serious issue for many countries since there are many transnational water bodies that spread the pollutants across the entire region. Water, environment and pollution together constitute a three axial problem that all concerned people in the region would like to focus on.

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