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CESIUM/SODIUM SEPARATION BY NANOFILTRATION-COMPLEXATION IN AQUEOUS MEDIUM

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ABSTRACT

Some nuclear effluents contain traces of radioactive elements in sodium salt media, from which radioactive cesium must be separated. Various processes (among them liquid/liquid extraction and ion exchange) can perform such separation, but they produce additional wastes. Therefore, nanofiltration has been selected as a new

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separation process that may allow a large volume reduction without generating additional wastes. Nanofiltration is a pressure-driven membrane process, between ultrafiltration and reverse osmosis, that can separate species at the ionic scale.

To perform the separation of cesium from highly salted aqueous medium, a nanofiltration process was combined with a cesium-selective complexation step. Ligands that were known to be highly cesium-selective in organic solvents were synthesized and modified in order to make them hydrosoluble. Ligands such as resorcinarenes or calixarenes were then tested through a nanofiltration system. One of them allowed cesium removal of 90% in an aqueous medium containing 250 g/L of sodium nitrate. By combining two such nanofiltration-complexation stages, it is now possible to remove 99% of trace-level radioactive cesium.

INTRODUCTION

Among the different available separative methods (liquid-liquid and solid-liquid extractions, ion-exchange resins), nanofiltration (NF) is a new pressure-driven membrane process that generates very few additional wastes. Located between ultrafiltration (UF) and reverse osmosis (RO), NF membrane cut offs are in the molecular-weight range of 300 to 1000 g/mol [1-3]. Moreover, to increase ionic separation, NF can be combined with a selective complexation step; retention of the target element is improved because the complex with the ligand (of larger size and mass than the target ion) prevents this ion from passing through the membrane [4]. For such a purpose, water-soluble ligands with molecular weights of about 500 g/mol can be used in the case of the NF-complexation association, whereas ultrafiltration-complexation systems need larger (polymeric) ligands and cause some specific problems associated with increasing viscosity and precipitation when volumic concentration treatments are required [5]. However, it has been observed that UF needs a lower pressure than does NF and that UF systems also give higher flux rates than NF systems, even when recent NF membranes display higher hydraulic permeabilities than the membranes in use ten years ago. Moreover, UF-complexation is more than 20 years-old, and an accurate appraisal of this technique that has developed through various applications is now possible [6-9]. Meanwhile, we believe that NF-complexation should lead to much more selective separation systems because designing new, small organic selective ligands is much easier than synthesizing long, polymeric selective molecules.

Our laboratory has been developing the NF-complexation technique since 1993 [10-13]. We have shown that using EDTA with a Nanomax 50 membrane allowed 100% retention (rejection) of Sr^{2+} and other polyvalent ions and only 10% retention of Na^+ and Cs^+ ions. Then, with the use of a cesium-selective lig-



and such as resorcinarene, Cs^+/Na^+ separation was also improved - but not satisfactorily. In fact, to remove radioactive traces of cesium in highly salted aqueous medium, a minimum of 99% Cs^+ retention along with a maximum of 10% Na^+ retention would be required for industrial applications.

The present paper describes how we selected a new highly cesium-selective hydrophilic ligand, and how we determined the best conditions for using this ligand in an NF-complexation process to achieve Cs^+/Na^+ separation in an aqueous medium. The first part of this paper is an experimental section describing the NF technique and cesium analysis. The second part reviews our attempt to find a highly cesium-selective hydrophilic ligand with the use of a NF-complexation system. The third part summarizes the different aspects of the use of tetrahydroxylated bis-crown-6 calix[4]arene in an NF-complexation system.

EXPERIMENTAL

Apparatus

Fig. 1 is a schematic diagram of the NF loop used in our experiments. By totally recycling the permeate and the retentate, the feed remains at constant composition during the experiments. NF was carried out with a Nanomax 50-plane membrane (Millipore) with a surface area of 0.015 m^2 , designed for tangential filtration. This membrane has the following specifications: a high retention of multivalent ions and neutral organic molecules with a molecular weight above 400

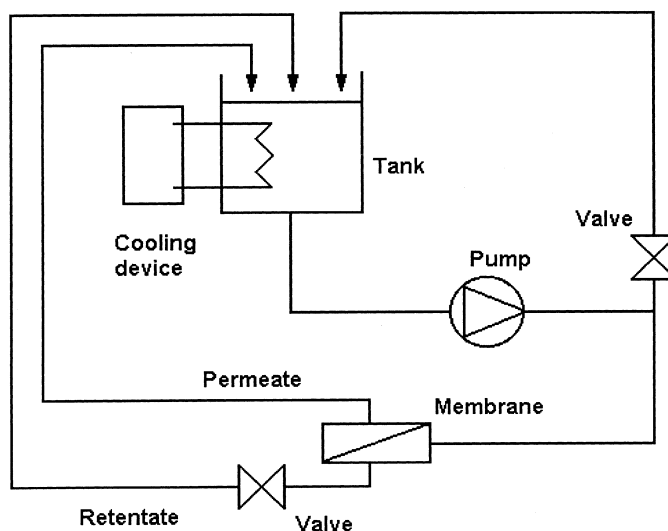


Figure 1. Schematic diagram of the nanofiltration loop.



g/mol, and a low retention of monovalent ions and neutral organic molecules with a molecular weight under 100 g/mol.

The retention (%) of a substance i was calculated as follows :

$$R_i = 100 \times (1 - C_{ip}/C_{ir}),$$

where C_{ip} is the concentration of i in the permeate and C_{ir} is the concentration of i in the retentate.

NF Test Procedure for Screening the Ligands

For each ligand to be tested, an aqueous solution containing 15 mg/L of cesium (CsNO_3) and 0.1 mol/L of sodium nitrate was filtered on an NF membrane. Different known amounts of ligand were then progressively added to the solution, and samples of permeate and retentate were taken 0.5 h after each addition of ligand. This first experiment was designed to check the ability of the ligand to improve cesium retention by the membrane in an aqueous medium containing low levels of sodium nitrate.

The second experiment was designed only for ligands having shown some cesium selectivity during the first experiment. To a solution containing 15 mg/L of cesium and a fixed [ligand]/[cesium] molar ratio, known amounts of sodium nitrate were progressively added, and the resulting solution was filtrated on an NF membrane. Samples were then taken as described above. This experiment was designed to measure the Cs^+/Na^+ selectivity of the ligand.

For all the NF results reported here, the sodium retention remained under 10%, because the Nanomax 50 membrane was chosen to be very permeable to monovalent ions. NF tests were carried out using a transmembrane pressure of 0.6 MPa and a temperature of 293 K.

Chemicals and Reagents

NaNO_3 (99%, Aldrich); CsNO_3 (99,99%, Aldrich)

Ligands: synthesized as described in references [14-16].

Analytical Methods

The cesium concentration was determined by atomic absorption spectroscopy (AAS) in an air-acetylene flame (apparatus: PU 9100X Philips atomic absorption spectrometer). Each result must be assumed to have a 5% standard deviation. Standards for AAS analysis were made of pure Cs^+ (cesium standards for ICP-AES analysis) and dissolved in the corresponding NaNO_3 and NaOH aqueous matrix.



RESULTS AND DISCUSSION

The cesium-complexing ability of resorcinarene **1** through an NF-complexation system was studied by Lemaire et al. [10, 12]. They showed that cesium retention increased with the resorcinarene **1** concentration while no significant effect on sodium retention was noticed. In fact, when used in an NF-complexation system, resorcinarene **1** helped to increase cesium retention considerably when added to a basic aqueous medium containing 0.1 mol/L of sodium nitrate (Fig. 2). However, the Cs^+/Na^+ selectivity was not high enough to allow a minimum of 90% cesium retention in a 3-mol/L NaNO_3 aqueous medium (Fig. 3). This target might be obtained by increasing the [resorcinarene]/[cesium] molar ratio, but calculations showed that a minimum of 8 g/L of ligand would have been required - a concentration too high for industrial applications.

Data from the NF-complexation tests summarized in Figs 2 and 3 allowed the calculation of distinct cesium-ligand and sodium-ligand complexation constants. These constants were found to be realistic; that is, the theoretical cesium retention calculated from the K_{CsL} and K_{NaL} values was in good agreement with the experimental cesium retention (Table 1). It appeared that the $K_{\text{CsL}}/K_{\text{NaL}}$ ratio was equal to 1300 for resorcinarene **1**, which is a measure of the Cs^+/Na^+ selectivity of this ligand.

Both of the UV-Vis spectra of resorcinarene **1** were recorded within a 4-day period (Fig. 4). The shape of the second spectrum was sufficiently different from the first one so that the resorcinarene structure could be assumed to be unstable in basic aqueous medium. Moreover, other NF experiments showed that cesium re-

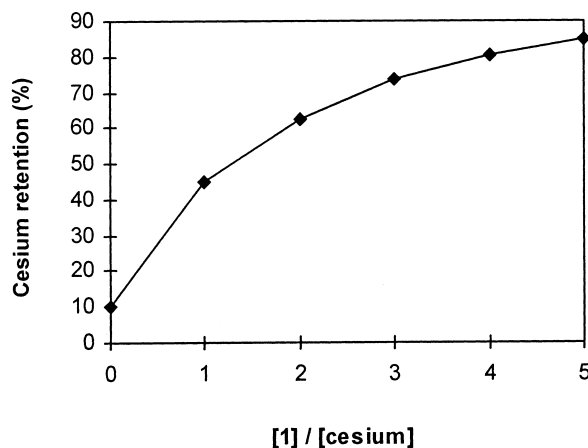


Figure 2. Cesium retention as a function of [ligand **1**]/[cesium] molar ratio. (pH = 11; T = 293 K; $\Delta P = 0.6$ MPa; [cesium] = 15 mg/L; $[\text{NaNO}_3] = 0.1$ mol/L).



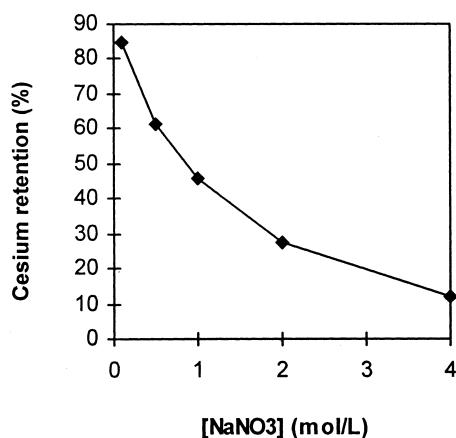


Figure 3. Cesium retention as a function of $[\text{NaNO}_3]$. ($[\text{ligand } 1]/[\text{cesium}] = 5$ (molar ratio); $\text{pH} = 11$; $\Delta P = 0.6 \text{ MPa}$; $[\text{cesium}] = 15 \text{ mg/L}$; $T = 293 \text{ K}$).

Table 1. Cesium-Ligand and Sodium-Ligand Complexation Constants of Ligand 1

Ligand	K_{CsL} (L/mol)	K_{NaL} (L/mol)
1	45,000	35

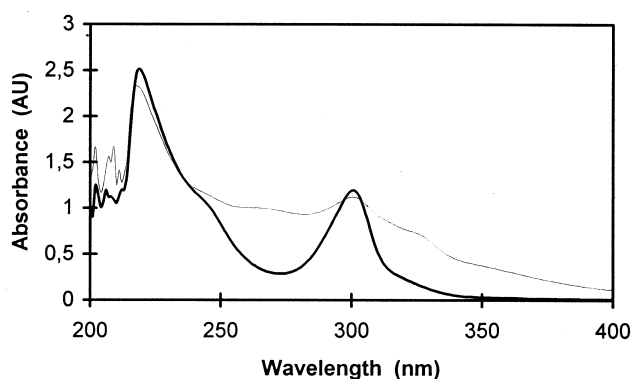
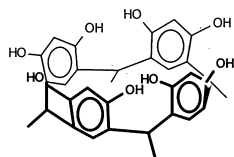
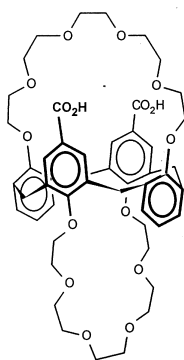


Figure 4. UV absorption spectra of ligand 1 in fresh aqueous medium (bold line) and after a 4-day period (thin line). ($[\text{ligand } 1] = 2 \times 10^{-5} \text{ mol/L}$; $\text{pH} = 13$).

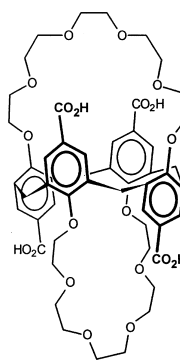




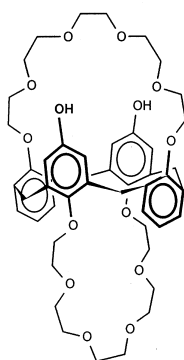
1. calix[4]resorcinarene



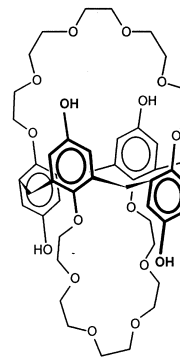
2. dicarboxylated BC6



3. tetracarboxylated BC6



4. dihydroxylated BC6



5. tetrahydroxylated BC6

tention decreased with time because of the resorcinarene structure modification during the NF experiments. In fact, ring-cupped structure of resorcinarene **1** slowly cleaves in basic aqueous medium, leading to reticulated resorcinolic oligomers that are much less cesium-selective than resorcinarene **1**. Meanwhile



resorcinarene **1** was kept as a reference in the matter of cesium-selectivity in aqueous medium. It was compared with all the other ligands in the following NF experiments.

Ungaro et al. [17] and Vicens et al. [18] showed that bis-crown-6 calix[4]arene (called BC6) had a preference for Cs^+ both in extraction and in complexation, resulting in a remarkable Cs^+/Na^+ selectivity ($S = \beta(\text{Cs}^+)/\beta(\text{Na}^+) = 2 \times 10^3$ in methanol for BC6). This selectivity was mainly due to the excellent size match between the cation and the ether loop of the ligand and to the impossibility for the crown part, attached to the calixarene, to wrap around the smaller Na^+ cation. However, they were lipophilic ligands, and the only way to use them in an aqueous medium for NF-complexation systems was to modify their structure by adding hydrophilic groups [11,15,16], hopefully keeping them cesium-selective. The resulting molecules had carboxylic (**2** and **3**) or phenolic (**4** and **5**) hydrophilic groups.

The carboxylic calix[4]arenes BC6 **2** and **3** were the first ones to be tested through NF experiments. Although the cesium retention did not increase as much with these BC6 calixarenes as with resorcinarene **1** (Fig. 5), the Cs^+/Na^+ selectivity seemed to be higher than that observed with resorcinarene. In fact, cesium retention decreased more slowly with BC6 than with resorcinarene when NaNO_3 was added as shown in Fig. 6. For ligands **2** and **3**, no K_{CsL} or K_{NaL} formation constants could be calculated from NF-complexation results because their cesium complexation effect was too low.

Both of the hydroxylated BC6 ligands **4** and **5** were then tested in NF experiments. First, dihydroxylated BC6 **4** showed good affinity toward cesium - not as high as with resorcinarene, but much higher than with carboxylic BC6 ligands.

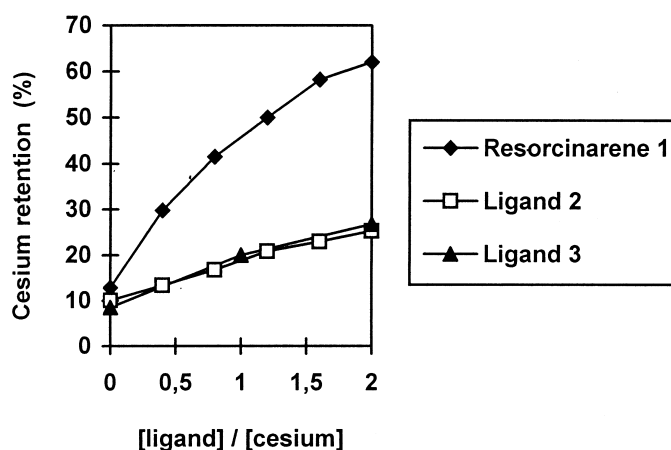


Figure 5. Cesium retention as a function of [ligand]/[cesium] molar ratio. ([cesium] = 15 mg/L; $[\text{NaNO}_3]$ = 0.1 mol/L; ΔP = 0.6 MPa; pH = 11; T = 293 K).



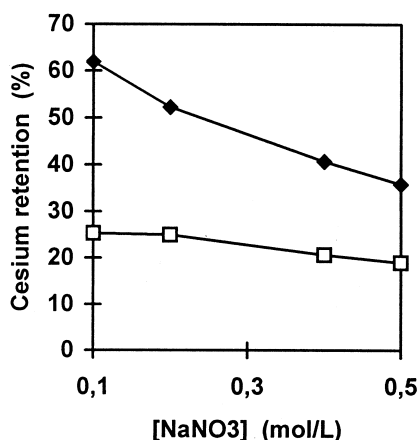


Figure 6. Cesium retention as a function of $[\text{NaNO}_3]$. ($[\text{ligand}]/[\text{cesium}] = 2$ (molar ratio); $T = 293 \text{ K}$; $[\text{cesium}] = 15 \text{ mg/L}$; $\text{pH} = 11$; $\Delta P = 0.6 \text{ MPa}$).

What was remarkable was the Cs^+/Na^+ selectivity of ligand **4**; when NaNO_3 was added, cesium retention by the dihydroxylated ligand **4** did not decrease as much as with resorcinarene **1** (Figs. 7 and 8). Finally, tetrahydroxylated BC6 **5** showed both good affinity toward cesium (similar to the resorcinarene) and an outstanding Cs^+/Na^+ selectivity that was greater than that observed for resorcinarene **1**. In fact, in a 4-mol/L NaNO_3 basic aqueous medium, the cesium retention by an NF

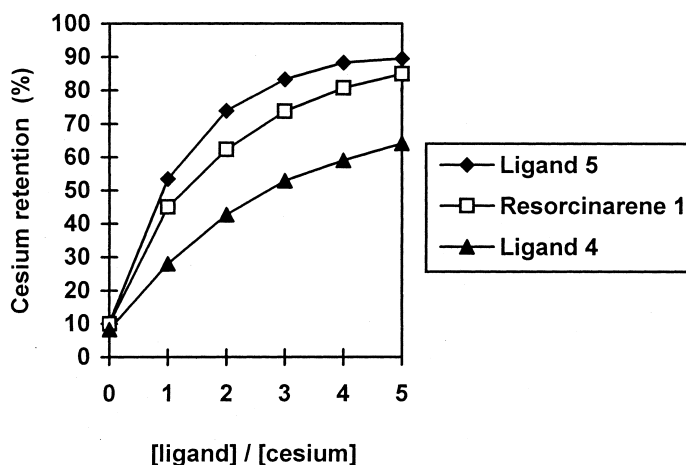


Figure 7. Cesium retention as a function of $[\text{ligand}]/[\text{cesium}]$ molar ratio. ($[\text{cesium}] = 15 \text{ mg/L}$; $[\text{NaNO}_3] = 0.1 \text{ mol/L}$; $\Delta P = 0.6 \text{ MPa}$; $\text{pH} = 11$; $T = 293 \text{ K}$).



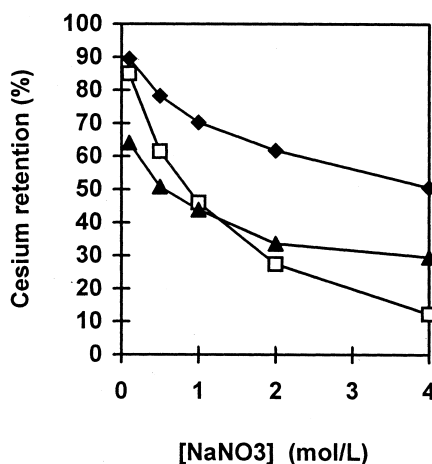


Figure 8. Cesium retention as a function of $[\text{NaNO}_3]$. ($[\text{ligand}]/[\text{cesium}] = 5$ (molar ratio); $T = 293 \text{ K}$; $[\text{cesium}] = 15 \text{ mg/L}$; $\text{pH} = 11$; $\Delta P = 0.6 \text{ MPa}$).

membrane was still as high as 50% with a $[\text{ligand}]/[\text{cesium}]$ molar ratio equal to 5 (Figs. 7 and 8).

As with resorcinarene **1**, both of the NF-complexation tests carried out with ligands **4** and **5** allowed the calculation of distinct cesium-ligand and sodium-ligand complexation constants (Table 2). The theoretical cesium retention rates calculated from the following K_{CsL} and K_{NaL} values were in good agreement with the experimental retention rates.

It appeared that K_{CsL} was ten times higher for ligand **1** than for ligand **4**, which explained why ligand **1** was more cesium-selective than ligand **4** in low-salted aqueous medium (Fig. 7). However, the $K_{\text{CsL}}/K_{\text{NaL}}$ ratio was equal to 1290 for **1** and to 1950 for **4**; therefore, the Cs^+/Na^+ selectivity was higher for **4** than for **1** (Fig. 8).

It also appeared that K_{CsL} was only twice as high for ligand **1** as for ligand **5**, which explains why retention curves were quite similar for both ligands in low-salted aqueous medium (Fig. 7). However, the $K_{\text{CsL}}/K_{\text{NaL}}$ ratio was 1290 for lig-

Table 2. Cesium-Ligand and Sodium-Ligand Complexation Constants of Ligands **4** and **5**

Ligand	K_{CsL} (L/mol)	K_{NaL} (L/mol)	$K_{\text{CsL}} / K_{\text{NaL}}$
4	3,900	2	1,950
5	23,000	3.5	6,570



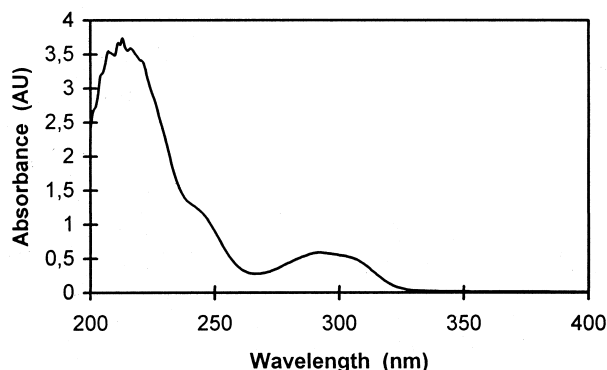


Figure 9. UV absorption spectrum of ligand **5** in aqueous medium. (pH = 12; [ligand **5**] = 10^{-4} mol/L).

and **1** and 6570 for ligand **5**, so the Cs^+/Na^+ selectivity was five times higher for **5** than for **1** (Fig. 8).

The Cs^+/Na^+ selectivity of a ligand usually depends on the nature and the number of its hydrophilic groups. Both ligand **4** and ligand **5** bear phenolic groups that are known to be very favorable for Cs^+ complexation [19]. Because ligand **5** also bears four phenolic groups (i.e., twice as many as for ligand **4**), ligand **5** was found to be more cesium selective than ligand **4**.

Ligand **5** was much more stable in the basic aqueous medium than was resorcinarene **1**. For instance, both of the spectra of tetrahydroxylated BC6 **5** were recorded within a 7-day period. The shape of the second spectrum was similar to the first one (Fig. 9 only represents one of both spectra). Therefore, it could be assumed that this ligand remained stable in basic aqueous medium.

USE OF TETRAHYDROXYLATED BIS-CROWN-6 CALIX[4]ARENE FOR THE SEPARATION OF Cs^+ IN HIGHLY SALTED AQUEOUS MEDIUM

Separating traces of cesium from a highly concentrated sodium salt medium is a challenging research subject because there is an important demand for an efficient, reliable, and clean process for such a separation. For instance, the removal of traces of radioactive cesium in nuclear effluents containing highly concentrated sodium salts requires a process that could remove 99% of the cesium and less than 10% of the sodium salts in one stream (for long-term storage) and less than 1% of the cesium and much of the sodium salts in the other stream (for eventual discharge).

Nanofiltration-complexation could meet these requirements only through the use of a highly cesium-selective ligand. In the following experiments, tetrahy-



Table 3. Cesium Retention as a Function of Cesium Concentration

([NaNO₃] = 0.1 mol/L; [ligand **5**] = 0.5 g/L; pH = 12; T = 293 K; ΔP = 0.6 MPa).

[Cesium] (mg/L)	Cesium retention (%)	Permeate flow (L.h ⁻¹ .m ⁻² .bar ⁻¹)
1.5	90	4
3	87	4
7.5	87	3.8
15	86	3.8

droxylated BC6 **5** was the selected ligand for Cs⁺/Na⁺ separation by NF-complexation, and the experimental conditions were supposed to simulate industrial conditions.

1. When [ligand **5**] was > 1 g/L, ligand **5** was soluble in aqueous medium only when the pH was > 11.5. This ligand could be recovered after use by acidification of the aqueous medium. When the pH was 5, ligand **5** totally precipitated.

2. It seemed that the [ligand **5**]/[cesium] molar ratio was not the key factor for cesium retention by NF-complexation. Table 3 shows the results obtained when the cesium concentration was increased during an NF test involving ligand **5** and a 0.1-mol/L sodium nitrate solution. However, the cesium retention did not appear to significantly after cesium addition. Calculations confirmed that the main factor was the total concentration of the ligand itself.

3. All the previous NF experiments were carried out at a temperature of 293 K. In this experiment, the temperature was increased to determine the effect of temperature on cesium retention.

Table 4. Cesium Retention as a Function of Temperature

([NaNO₃] = 3 mol/L; [ligand **5**] = 3.4 g/L; [cesium] = 15 mg/L; pH = 12; ΔP = 0.6 MPa).

Temperature (K)	Cs retention	Permeate flow (%)(L.h ⁻¹ .m ⁻² .bar ⁻¹)
293	91.2	3.4
298	90.4	3.8
303	88.4	4.2
313	85.0	5.1



Table 5. Cesium Retention as a Function of Sodium Nitrate Concentration

([cesium] = 12.5 mg/L; [ligand **5**]/[cesium] = 40; pH = 12; T = 293 K; ΔP = 0.6 MPa).

[NaNO ₃] (mol/L)	Cesium retention (%)	Permeate flow (L.h ⁻¹ .m ⁻² .bar ⁻¹)
0.1	98.5	4
1	96.1	3.6
2	93.6	3.2
3	90.6	2.8

At a constant ligand concentration, the cesium retention decreased with increasing temperature (Table 4). This phenomenon, which proved to be reversible, was due to a partial opening of the polymeric membrane pores rather than to a de-complexation of the cesium-ligand **5** complex.

4. Results of another experiment revealed that achieving 90% cesium retention by NF in a 3-mol/L sodium nitrate aqueous medium was possible with ligand **5**. With a basic aqueous solution containing 12.5 mg/L of cesium, 3.4 g/L of ligand was required to reach 90% cesium retention (Table 5).

This experiment proved that tetrahydroxylated bis-crown-6 calix[4]arene **5** was a high-performance ligand for an eventual Cs⁺/Na⁺ separation by NF-complexation in aqueous medium. It allowed 90% cesium retention in a 3-mol/L NaNO₃ aqueous medium. By combining two stages of such a process, it should then be possible to remove 99% of traces of cesium and not more than 10% of the initial sodium salts.

CONCLUSIONS

Tetrahydroxylated bis-crown-6 calix[4]arene **5** showed a high Cs⁺/Na⁺ selectivity in the experiments reported here. Through an NF-complexation process, it allowed separation of traces of cesium from a highly salted aqueous medium. Such efficient separation has never been achieved with other filtration methods. As compared with liquid-liquid extraction, NF does not require solvent addition, which is a great advantage from the standpoint of environmental consideration. Nanofiltration applications are currently used in industrial chemical processes for trace compound removal, valuable metal recovery, and products/catalysts separation in homogeneous catalytic processes. Because of its wide industrial applications, NF has strong potential for further development as a process that would be useful for cleaning of nuclear effluents.



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