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SEPARATION OF LANTHANIDES(III) BY NANOFILTRATION-COMPLEXATION IN AQUEOUS MEDIUM

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ABSTRACT

At first diethylenetriaminepentaacetic acid (DTPA) was combined with a nanofiltration (NF) system in order to separate Gd^{3+} from La^{3+} ions in aqueous medium. Combined with a Sepa MG-17 NF membrane (Osmonics), it allowed Gd^{3+} retention to reach 95%, whereas La^{3+} retention only reached 50%. Then, to improve this one-step separation, new hydrophilic ligands were synthesized. Bis(diethanol)amide-DTPA 1 combined with a NF system involving a Sepa MG-17 membrane allowed Gd^{3+} retention to reach 87%, whereas La^{3+} retention remained at 5%. Finally, the same nanofiltration-complexation system was used to separate an equimolar aqueous mixture of Gd^{3+}, Pr^{3+}, and La^{3+} ions.

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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 produces very few additional wastes. Located between ultrafiltration and reverse osmosis, nanofiltration membranes "cutoffs" are in the range of 300 to 1000 g/mol molecular weight (1–3), which allows separations at ionic and small molecular scales.

In order to increase ionic separation, nanofiltration can be combined with a selective complexation step (Fig. 1): Retention of the target ion is improved because the complex with the ligand—having bigger size and mass than the target ion—prevents this ion from passing through the membrane (4). Our laboratory has been developing this promising technique since 1993 (5–8).

Water-soluble ligands with molecular weight of about 500 g/mol can be used in the case of the nanofiltration-complexation association, whereas micro- or ultrafiltration-complexation systems need larger ligands and induce some specific problems of increasing viscosity and precipitation (9).

The industrial separation of rare earths in order to increase their purity actually involves liquid-liquid extraction method (10). This process is of great importance, because highly purified lanthanides are commonly used in electronics, ceramics, optics, and catalysis. It is also of great importance for nuclear industry because actual actinides(III)-lanthanides(III) separation processes are based on such liquid-liquid extraction methods (11–12). However liquid-liquid extraction requires additional diluant, which is a problem in matter of environmental preservation. The present study demonstrates that a nanofiltration process combined with a complexation step can achieve the separation of lanthanides(III) in aqueous medium.

As it was described in our previous study (13), nanofiltration-complexation showed to be a valuable method for separating gadolinum(III) ions from lanthanum(III) ions in aqueous medium. However, the low difference (14%) between

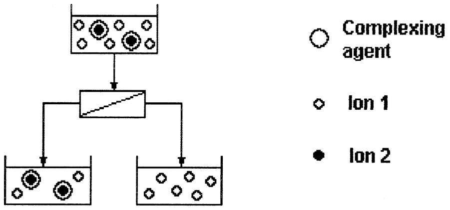


Figure 1. Principle of nanofiltration with a complexation step.

retention of Gd^{3+} and La^{3+} did not allow an easy separation of both elements with a single-stage process. In fact, retentions of Gd^{3+} and La^{3+} were respectively equal to 99% and 85%, so the retention of uncomplexed element (La) was too high for such a goal. The aim of our study was to find a nanofiltration membrane that would be completely permeable to uncomplexed trivalent ions such as Gd^{3+} and La^{3+}, while being nonpermeable to lanthanides(III)-DTPA complexes. Our goal was also to synthesize new hydrophilic ligands that would be selective towards lanthanides(III) ions and have size and mass high enough to be retained by the new chosen membrane.

The first part of this article is an experimental section describing the nanofiltration apparatus and the synthesis of new hydrophilic ligands for the separation of lanthanides(III). The second part is an attempt to choose the most suitable nanofiltration membrane and the most selective ligand for Gd^{3+}/La^{3+} separation. Finally, the most efficient system was used to separate different lanthanides(III) ions in aqueous medium.

EXPERIMENTAL

Nanofiltration Tests

Apparatus

Figure 2 is a scheme of the nanofiltration loop used in our experiments. By totally recycling permeate and retentate streams, feed remains at constant composition during the experiments. NF was carried out with Sepa-type plane membranes (Osmonics), with a surface area of 0.015 m^2, designed for tangential filtration.

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.

Nanofiltration Test Procedure

For each nanofiltration test, an aqueous solution of 1 L containing 902.5 mg/L (2 mmol/L) of $Gd(NO_3)_3$, $6H_2O$ and 865.8 mg/L (2 mmol/L) of $La(NO_3)_3$, $6H_2O$ was filtrated on a NF membrane. Different known amounts of ligand were then progressively added to the solution, and 10 mL samples of permeate and retentate were taken 30 min after each ligand addition.

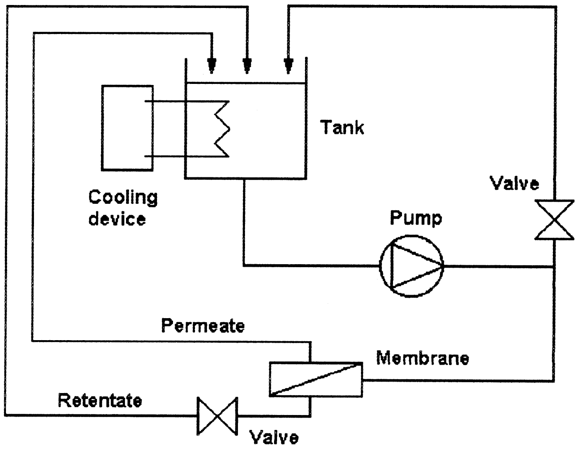


Figure 2. Scheme of the nanofiltration loop.

Transmembrane pressure ΔP was kept at 0.6 MPa during each experiment. The flow ratio of permeate to retentate was equal to 0.01.

Chemicals and Reagents

$Gd(NO_3)_3$, $6H_2O$ (99.9%, Aldrich), $Pr(NO_3)_3$, $6H_2O$ (99.9%, Aldrich), $La(NO_3)_3$, $6H_2O$ (99.9%, Aldrich), EDTA (98%, Aldrich), and DTPA (97%, Aldrich).

Analytical

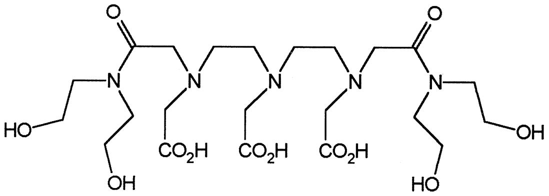
Gadolinium, praseodymium and lanthanum concentrations were determined by ICP-AES (Inductively Coupled Plasma, Atomic Absorption-Emission Spectroscopy) with a SPECTRO D spectrophotometer.

Synthesis of New Hydrophilic Ligands

Synthesis of Bis(diethanol)amide DTPA **1**

To a solution of diethylenetriaminepentaceticacid (DTPA) dianhydride (10g, 27.98 mmol) in 150 mL of dry DMF at 80°C under nitrogen were slowly added 17 g of diethanolamine (167.9 mmol) in 50 mL of DMF. The mixture was stirred for 48 h. The DMF was decanted from the oily residue and the residue diluted in water. Addition of acetone led to a viscous precipitate, which was ground and isolated by decantation. Purification on amberlite IR-120 with water as eluant gave pur 1 (7.98g, 50%) as a white hygroscopic powder after evaporation and drying under vacuum.

^{1}H NMR (D_2O): 3.1 (t, J = 6.25, 4H); 3.48 − 3.52 (t + s, 10H); 3.59 (t, J = 6.2, 4H); 3.76 (t, J = 5.2, 8H); 3.91 (s, 4H); 4.49 (s, 4H). ^{13}C NMR (D_2O): 50.59, 55.88, 56.62, 58.84, 59.95 (CH_2CO_2H and NCH_2CH_2N); 51.75, 52.14, 60.85, 61.20 ($N(CH_2CH_2OH)_2$); 169.0, 172.6, 176.8 (CO_2H and CO). ES-MS (ES^-): 566.3 ($[M-H]^-$); 282.7 ($[M-2H]^{2-}/2$). Elemental analysis cal. for $C_{22}H_{41}O_{12}N_5,2H_2O$ (603.59): C 43.78, H 7.51, O 37.11, N 11.60. found: C 43.91, H 7.41, O 37.24, N 11.17.

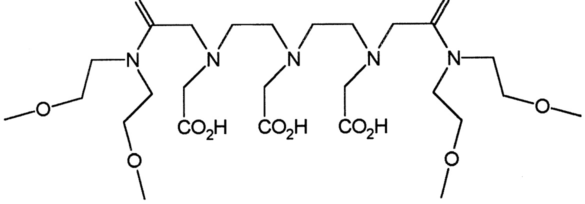


Ligand 1.

Synthesis of Bis-(di(2-methoxyethyl)amide) DTPA **2**

DTPA dianhydride (3 g, 8.4 mmol) was dissolved in dry DMF (80 mL) with Et_3N (10 mL, 72 mmol) at room temperature under nitrogen. 7.5 mL of bis(2-methoxyethyl)amine (51.2 mmol) in 40 mL of dry DMF were slowly added and the mixture was stirred for 24 h. Et_2O was then added and the viscous oil was separated from the solvents by decantation. The residue was purified twice by dissolution in MeOH, precipitated in Et_2O and ground. Drying under vacuum gave **2** as a colorless hygroscopic foam (5.18 g, 60%), which was used without additional purification.

^{1}H NMR (D_2O + NaOD): 2.50 (t, 4H); 2.52 (t, 4H); 2.98 (s, 2H); 3.11 (s, 4H); 3.31 (s, 6H); 3.32 (s, 6H); 3.51 (s, 4H); 3.53 − 3.56 (m, 16H). ^{13}C NMR (D_2O): 45.94, 47.03, 47.81, 49.75, 53.69, 56.29, 57.78, 67.16, 69.56, 69.69 (CH_2); 58.63, 59.07 (OCH_3); 166.9, 170.5, 175.7 (CO_2H and CO). ES−MS (ES^-): 622.1 ($[M-H]^-$); ES−MS (ES^+): 624.3 ($[M + H]^+$); 646.2 ($[M + Na]^+$).

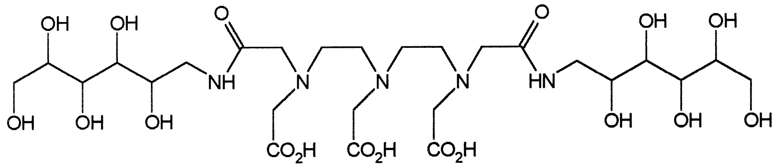


Ligand 2.

Synthesis of Bis(1-deoxy-1-amidosorbitol) DTPA **3**

DTPA dianhydride (5 g, 14 mmol) was dissolved in dry DMSO (80 mL) with Et_3N (15 mL) at 80°C under nitrogen. 5.32 g of 1-amino-1-deoxy-D-sorbitol (29.4 mmol) in DMSO (40 mL) were slowly added and the mixture was stirred for 24 h. After cooling, 300 mL of acetone were added and the resulting white precipitate filtered off. Drying under vacuum gave **3** (9.9 g, 98%) as a white powder, which was used without additional purification.

^{13}C NMR (D_2O): 42.17, 47.14, 49.61, 53.41, 56.73, 57.48, 63.16 (CH_2); 69.44, 71.08, 71.18, 71.38 (CH); 171.0, 178.86 (CO_2H and CO). ES-MS (ES^+): 741.2 ($[M + Na]^+$).

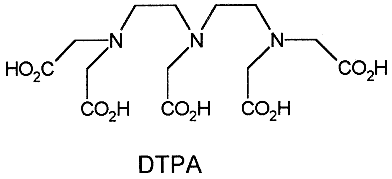


Ligand 3.

RESULTS OF THE NANOFILTRATION-COMPLEXATION TESTS

Discussion

In our previous study (13), DTPA (diethylenetriaminepentaaceticacid) was used as a selective ligand for the separation of gadolinium(III) and lanthanum(III) ions because literature presented DTPA as a very selective ligand for lanthanides(III) ions (14). However, the Nanomax 50 membrane that was used for that study had a too low permeability for trivalent ions such as Gd^{3+} and La^{3+}, and it did not allow a good separation of Gd^{3+} and La^{3+} ions.



DTPA

Scheme 1.

The present study, therefore, started with a screening of different commercially available NF membranes. They were chosen among the OSMONICS catalog that supplies a wide range of NF and UF membranes. The first tests of ion permeability showed that commercial data concerning the tested membranes did not match exactly with the results obtained in our experiments. Therefore, every kind of NF membrane had its ion permeability and its "cutoff" tested before being used in nanofiltration-complexation separation tests.

Table 1 describes the commercial values for monovalent ions retention and cutoffs. It also shows the experimental values for lanthanide(III) ion retention and the DTPA-lanthanides(III) complex maximum retention. It appeared that MQ-09 and MX-07 membrane retention of Gd^{3+} and La^{3+} was too high to obtain a good separation by nanofiltration-complexation. Only the five remaining membranes showed a low retention ($<5\%$) of trivalent lanthanide ions. Among these five membranes, BP-02 and HP-09 showed a too low retention of DTPA-lanthanide(III) complexes ($<90\%$). We therefore kept the three remaining membranes (MG-17, MW-15, and BQ-01) to investigate their performance in the separation of Gd^{3+} from La^{3+} by using nanofiltration-complexation.

A 2 mmol/L of Gd^{3+} and La^{3+} aqueous solution with different amounts of DTPA was filtrated on the three chosen membranes (Figs. 3, 4, and 5).

The difference between Gd^{3+} and La^{3+} retentions was at a maximum for a ligand concentration of 2 mmol/L (i.e., when [DTPA]/[Gd] = 1). This difference was 45% with the MG-17 membrane, 17% with the MW-15 membrane, and 19% with the BQ-01 membrane. So the MG-17 membrane was chosen as the most

Table 1. Comparison between Theoretical Cutoffs and Monovalent Ion Retention versus Experimental Trivalent Lanthanide Retention and DTPA-Lanthanide Complexe Retention for Seven Different NF Membranes (Experimental Runs were Carried Out with $[Gd^{3+}] = [La^{3+}] = 2$ mmol/L; $\Delta P = 0.6$ MPa; $T = 293$ K; pH Ranged from 2 to 6; [DTPA]/[Gd] Ratio Ranged from 0 to 2)

	Commercial Data		Experimental Results	
NF Membranes	Cutoff (g/mol)	Monovalent Ions Retention (%)	Gd^{3+} and La^{3+} Retention (%)	Max. Retention of DTPA-Ln(III) Complexes (%)
Sepa MQ-90	200–400	85	80–95	100
Sepa MX-07	300–600	50–70	98–100	100
Sepa MG-17	1000–3000	0	0–5	99
Sepa MW-15	2000–4000	0	0–5	94
Sepa BQ-01	400–900	30–50	0	96
Sepa BP-02	1000–4000	30–50	0	62
Sepa HP-09	1000–4000	0	5	25

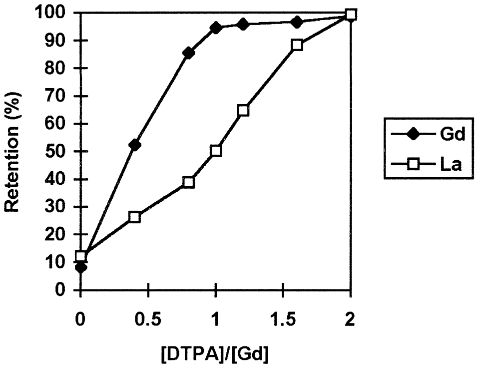


Figure 3. Retention of Gd^{3+} and La^{3+} as a function of [DTPA]/[Gd] ratio (Sepa MG-17 membrane; pH = 3.8; $\Delta P = 0.6$ MPa; T = 293 K; [Gd] = [La] = 2 mmol/L).

suitable NF membrane for a Gd^{3+}/La^{3+} separation by nanofiltration-complexation with ligands having the size and mass of DTPA.

However Fig. 3 also showed that with a ratio of [ligand]/[Gd] = 1, roughly 100% of Gd^{3+} but also 50% of La^{3+}, was retained by the membrane, hence DTPA had complexed more lanthanides(III) ions than it was supposed to. It was suggested that DTPA, an L^{5-} ligand in its basic form, formed a $[DTPA(Gd)]^{2-}$ complex that was able to complex other lanthanides(III) ions, but not selectively.

It was decided to choose L^{3-} type ligands to improve the Gd^{3+}/La^{3+} separation. In order to keep good complexing properties and to enhance the Gd^{3+}/La^{3+} selectivity, synthesis of new hydrophilic ligands having similar structures as

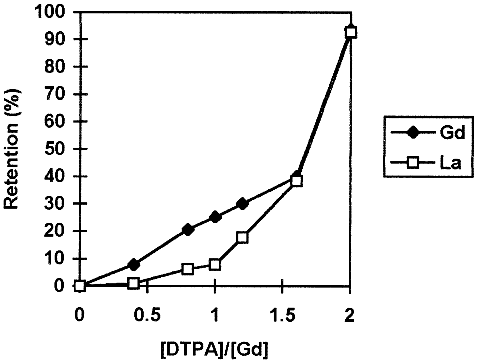


Figure 4. Retention of Gd^{3+} and La^{3+} as a function of [DTPA]/[Gd] ratio (Sepa MW-15 membrane; pH = 3.8; $\Delta P = 0.6$ MPa; T = 293 K; [Gd] = [LA] = 2 mmol/L).

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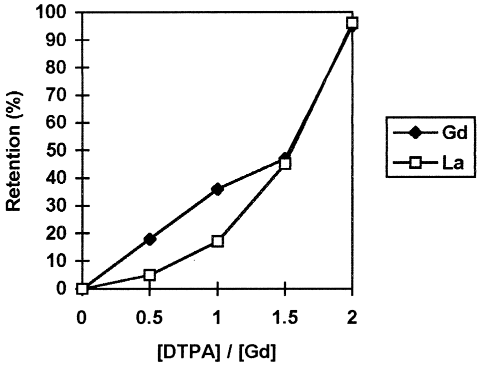
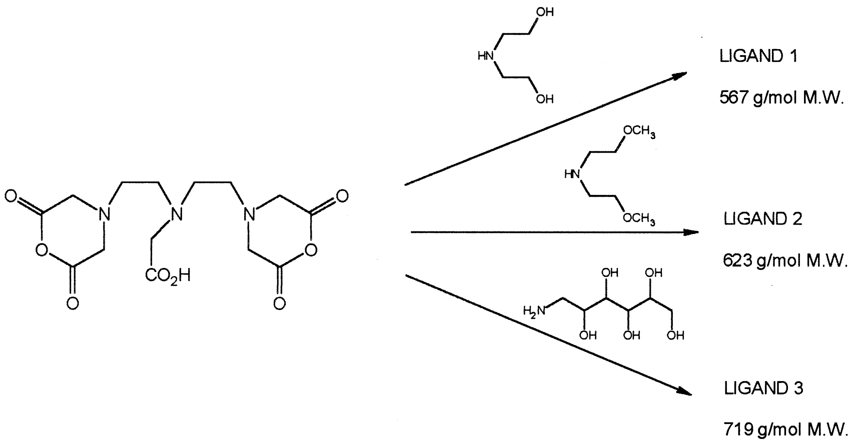


Figure 5. Retention of Gd^{3+} and La^{3+} as a function of [DTPA]/[Gd] ratio (Sepa BQ-01 membrane; pH = 3.8; ΔP = 0.6 MPa; T = 293 K; [Gd] = [La] = 2 mmol/L).

DTPA was carried out. We started from DTPA dianhydride, which reacted with hydrophilic amines in order to obtain ligands with three carboxylic and two amino alcohol functions.



Scheme 2.

Ligands **1**, **2**, and **3** were obtained (see "Experimental" section), each one having a different size and mass from the other ones.

All subsequent experiments were carried out in aqueous medium using a Sepa MG-17 membrane.

Compared with ligands **1** and **2**, ligand **3** appeared to be the less-selective ligand for the Gd^{3+}/La^{3+} separation, with a maximum of 71% between the retention of Gd^{3+} and La^{3+} (Fig. 6). The Gd^{3+}/La^{3+} selectivity of ligand **3** may have

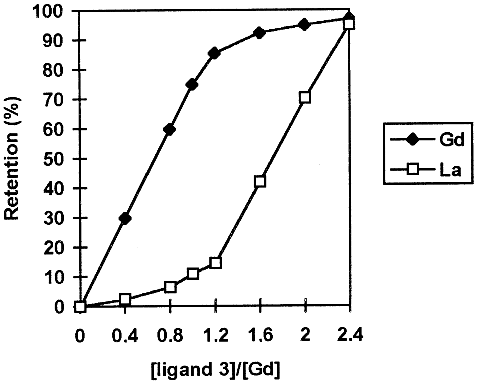


Figure 6. Retention of Gd^{3+} and La^{3+} as a function of [ligand **3**]/[Gd] ratio (Sepa MG-17 membrane; pH = 3.8; ΔP = 0.6 MPa; T = 293 K; [Gd] = [La] = 2 mmol/L).

been lowered by the steric hindrance of hydroxyl groups. Meanwhile, Gd^{3+}/La^{3+} separation was greatly improved compared to previous experiments.

Ligands **1** and **2** gave roughly the same result, i.e., a maximum of 82% between the retention of Gd^{3+} and La^{3+} with a 1:1 [ligand]/[metal] ratio (Figs. 7 and 8). Compared with the performance of DTPA in the same conditions, ligands **1** and **2** proved to be much more selective probably because their L^{3-} type structure better fitted to trivalent lanthanides(III) ions than L^{5-} type ligands such as DTPA did.

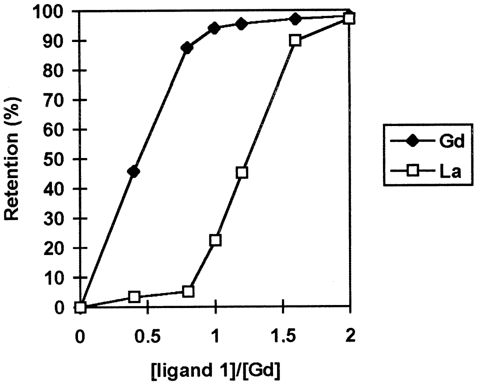


Figure 7. Retention of Gd^{3+} and La^{3+} as a function of [ligand **1**]/[Gd] ratio (Sepa MG-17 membrane; pH = 3.8; ΔP = 0.6 MPa; T = 293 K; [Gd] = [La] = (2 mmol/L)).

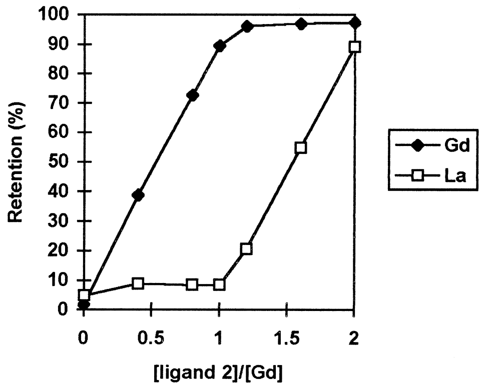


Figure 8. Retention of Gd^{3+} and La^{3+} as a function of [ligand **2**]/[Gd] ratio (Sepa MG-17 membrane; pH = 3.8; $\Delta P = 0.6$ MPa; T = (293 K; [Gd] = [La] = (2 mmol/L)).

For separation methods, an α factor is generally used to evaluate the separation efficiency (11). Similarly, an α factor could be defined as follows:

$$\alpha_{Gd/La} = ([Gd]_{retentate}/[Gd]_{permeate})/([La]_{retentate}/[La]_{permeate})$$

This factor was calculated for each ligand at the maximum difference between retentions of Gd^{3+} and La^{3+}, i.e., when [ligand]/[Gd] = 1, because it allowed a greater precision on analytical results. It was found that α = 35 for ligand **3**, α = 93 for ligand **2**, and α = 128 for ligand 1. It must be noticed that α results presented here were underestimated, because ligands' real complexing properties were limited by the membrane performances: for instance, when La^{3+} complexing ratio was 0% (i.e., when there was no ligand), La^{3+} retention was always higher than 0%; and when Gd^{3+} complexing ratio was 100% (i.e., when [ligand]/[Gd^{3+}] = 1), Gd^{3+} retention was always lower than 100%. However, even underestimated, the nanofiltration-complexation process with ligand **1** ranks among the best separation processes for Ln(III)/Ln(III) separation.

We could also notice a strong pH dependance for the Gd^{3+} and La^{3+} retentions when ligand **1** was used (Fig. 9). The maximum difference between retentions of Gd^{3+} and La^{3+} was observed when pH = 3.8.

Ligand **1** efficiency was also tested with Gd^{3+}, Pr^{3+}, and La^{3+} ions. A 1 mmol/L gadolinium(III), praseodymium(III) and lanthanum(III) nitrate ion aqueous solution was filtrated on a Sepa MG-17 NF membrane. Then ligand **1** was progressively added to the solution (Fig. 10).

Gadolinium was the first one to be complexed, then praesodymium and finally lanthanum. At 1 mmol/L of ligand **1**, Gd^{3+} retention was up to 72% whereas Pr^{3+} and La^{3+} retention were respectively equal to 21% and 6%. At 2

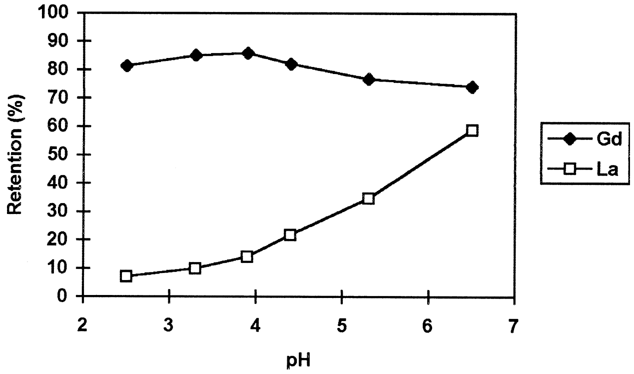


Figure 9. Retention of Gd^{3+} and La^{3+} as a function of pH (Sepa MG-17 membrane; [ligand **1**] = 2 mmol/L; $\Delta P = 0.6$ MPa; T = 293 K).

mmol/L of ligand **1**, Gd^{3+} and Pr^{3+} retentions reached 93% and 75% while La^{3+} retention was only equal to 23%. With such retention profiles, it could be possible to separate each one of the three lanthanides. For instance, with a 0.5 [ligand **1**]/[Gd] ratio, Gd^{3+} retention reached 43%, whereas La^{3+} and Pr^{3+} retentions were respectively equal to 3% and 6%. So with a five-stage process involving such separation factors, it would be possible to remove 94% of Gd^{3+}, while only removing 27% of Pr^{3+} and 14% of La^{3+}.

Ligand **1** proved to be highly selective towards trivalent ions of some lanthanides. More precisely, ligand **1** behavior was the same as DTPA, i.e., the higher the lanthanides(III) mass, the higher the ligand **1** affinity towards lanthanides(III).

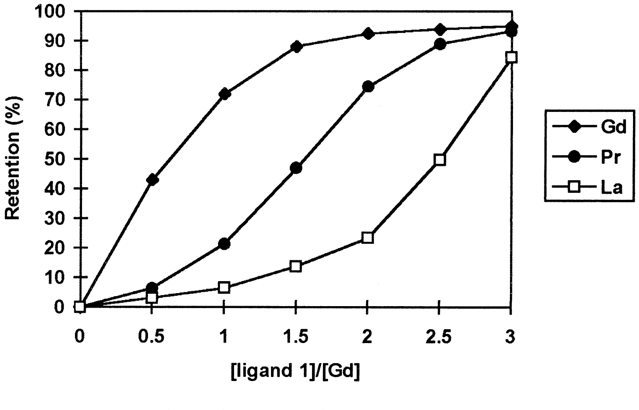


Figure 10. Retention of Gd^{3+}, Pr^{3+}, and La^{3+} as a function of [ligand **1**]/[Gd] ratio (Sepa MG-17 membrane; pH = 3.8; $\Delta P = 0.6$ MPa; T = 293 K).

Other experiments pointed out the fact that lanthanides ions in their pure form could be recovered from aqueous solutions containing lanthanides(III)-ligand **1** complexes. Such solutions were passed through specific ion exchanging resins, and ligand **1** and lanthanides(III) ions were removed separately.

This nanofiltration-complexation process could also be efficient for actinides(III)/lanthanides(III) separation, should ligand **1** be proved to be selective towards minor actinides(III) such as Am^{3+} and Cm^{3+}. Experiments are still in progress.

CONCLUSION

This article shows that even if nanofiltration-complexation is a new process, it is already a valuable process for lanthanides(III)/lanthanides(III) industrial separations. It is a particularly easy separative method that does not require solvent addition, which is not the case of liquid-liquid extraction and which is a great advantage in matter of environment preservation. In this way, nanofiltration should be predicted a promising development in many industrial applications.

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