SELECTIVITY OF NF MEMBRANE FOR TREATMENT OF LIQUID WASTE CONTAINING URANIUM

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ABSTRACT

The performance of two nanofiltration membranes were investigated for treatment of liquid waste containing uranium through two conditions permeation: permeation test and concentration test of the waste. In the permeation test solution permeated returned to the feed tank after collected samples each 3 hours. In the test of concentration the permeated was collected continuously until 90% reduction of the feed volume. The liquid waste ("carbonated water") was obtained during conversion of UF\textsubscript{6} to UO\textsubscript{2} in the cycle of nuclear fuel. This waste contains uranium concentration on average 7.0 mg L\textsuperscript{-1}, and not be eliminated to the environmental. The waste was permeated using a cross-flow membrane cell in the pressure of the1.5 MPa. The selectivity of the membranes for separation of uranium was between 83% and 90% for both tests. In the concentration tests the waste was concentrated around for 5 times. The surface layer of the membranes was evaluated before and after the tests by infrared spectroscopy (ATR-FTIR), field emission microscopy (FESEM) and atomic force spectroscopy (AFM). The membrane separation process is a technique feasible to and very satisfactory for treatment the liquid waste.

1. INTRODUCTION

Nanofiltration membrane is a membrane separation process which uses the pressure driving force for separation. It presents intermediate properties between ultrafiltration (0.2-0.7 MPa) and reverse osmosis (2.0-8.0 MPa) [1-3].

The separation process takes place through a combination of two mechanisms: ion size exclusion and ion charge exclusion. Ion charge exclusion depends on the membrane surface charge, ionic strength and ion valence. The valence and ionic strength influence the charge density of the membrane and its isoelectric point (IEP). The surface of the nanofiltration membrane is generally negatively charged below the IEP and positively charged above it. In
general, the nanofiltration membrane is negatively charged in neutral and alkaline pH and positively charged at low pH [1-4].

Nanofiltration membranes are usually obtained in two stages, namely: preparation of a microporous support and deposition of a thin film, usually by interfacial polymerization, creating a film thickness between 0.1-0.5 µm, responsible for the selectivity of the membrane [3].

The knowledge of the physicochemical properties of this film is crucial to determine the behavior of nanofiltration membranes, including long-term performance [5-6]. In general, several techniques are used together to characterize virgin and used membranes. Zeta potential, attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR), atomic force microscopy (AFM), scanning electron microscopy (SEM) are among the most cited ones [7].

Nanofiltration processes are widely used in seawater desalinization, purification of enzymes, concentration of fruit juices, among others, because they are able to reject negative multivalent ions, such as \( \text{SO}_4^{2-} \) and \( \text{PO}_4^{3-} \), whereas monovalent ions like \( \text{Cl}^- \) are less rejected [3]. The application of membrane technology to treat low and intermediate level liquid radioactive wastes (LRWs) is relatively new [7-13]. Some articles have reported that reverse osmosis and nanofiltration gave uranium rejection in the range 80-99.5%.

The use of membrane processes to treat radioactive waste requires an initial set of experiments before implementation of such systems in a nuclear installation [14]. For membranes to be competitive with conventional technologies, a membrane process needs to operate with a high rate of flux, present a high degree of selectivity and a high resistance to fouling. The main requirement is to choose a nanofiltration membrane presenting chemical resistance and stability to radiation, as the membrane will be continuously exposed to the radioactive waste. Polyamide membranes meet these requirements because they present many intermolecular crosslinkings [14-16]. The pore size of the NF membrane is in the range 1-10 nm and is very stable between pH 2-11. In the nuclear area, depending on the composition, nature of the ion and activity of the waste to be treated, the performance of the membrane may change, resulting in loss of its properties.

Some papers focused on the treatment of LRWs containing radionuclides such as \(^{137}\)Cs, \(^{241}\)Am, \(^{238}\)U and \(^{235}\)U using nanofiltration membranes [13,17]. Rejection factors over 80% were obtained. However, membrane characterization before and after waste treatment has been scarcely studied. Long-term tests are also essential to decide the viability of nanofiltration as a separation technique to recover radionuclides from their wastes. Such studies are useful to determine possible changes in the membrane structure. Therefore, it is necessary to study various parameters related to the operation before applying a membrane separation process to treat a given radioactive waste.

For this reason, this work aimed at evaluating the behavior of the two NF membrane to treat a radioactive liquid waste in order to recover the radionuclides present (\(^{235}\)U + \(^{238}\)U). The transport properties (permeate flux, hydraulic permeability and rejection) of the nanofiltration membrane were evaluated in two types of experiments: permeation test and concentration test. After these experiments the transport properties, chemical composition and morphology of the selective layer of the membranes were evaluated.
2. EXPERIMENTAL

2.1. Radioactive Waste Sample

The sample of LRW was kindly provided by the Nuclear Fuel Factory (INB), which is responsible for the production of nuclear fuel for PWR (Pressurized Water Reactor), adopted in Brazil for electricity generation. This waste is generated after conversion of uranium hexafluoride gas ($\text{UF}_6$) into uranium dioxide ($\text{UO}_2$). The reaction of uranium hexafluoride with carbon dioxide ($\text{CO}_2$) and ammonia ($\text{NH}_3$) produces ammonium tricarbonatouranate(VI) known as TCAU [17-19]. TCAU is dried in filters, generating a liquid waste containing uranium, known as "carbonated water", because of its high concentration of $\text{CO}_3^{2-}$ ions. This waste can contain up to 50 mg L$^{-1}$ of soluble uranium. Uranium was determined by arsenazo(III) method [20] using a FEMTO 800 XI spectrophotometer ($\lambda = 650$ nm). Carbonate was determined by acid-base titration [21]. Ammonia ($\text{NH}_3$) was determined by the Nessler method [22]. Fluoride ion was determined using an ion-selective electrode. The pH was determined using a Digmed DM-22 digital pHmeter.

2.2. Membranes Samples

The nanofiltration membranes used in this study ("Developmental NF" and Developmental SWNF) was kindly supplied by Dow/Brazil. It presents a selective layer of polyamide on a poly (ether sulfone) support and was identified in this work as NF0 and SW0.

2.2.1. Transport properties of the membranes

2.2.1.1. Permeate flux, hydraulic permeability and rejection

For the experiments were used a cell filtration system CF II Membrane Element Cell, Osmonics, with a membrane active area of 0.0140 m$^2$. The permeation experiments were conducted at a constant trans-membrane pressure of 1.5 MPa. Initially, the membrane was placed in water for an hour before being placed in the cell. The cell membrane was filled with distilled water at 1.5 MPa to compact the membrane. The compactation is necessary for accommodation of the structure at the operating pressure. Compactation is achieved when three readings of the permeate flux at 20 min intervals are identical. The same procedure was carried at pressures of 1.0 and 0.5 MPa. The value of permeated flux ($J_p$) of the membrane was calculated through Eq. (1) [2].

\[ J_p (L m^{-2} h^{-1}) = \frac{\text{Flow}(L s^{-1}) \times 3600(s/h)}{\text{membrane area} (m^2)} \] (1)

Data obtained were used to build a curve of permeate flux versus pressure to determine the hydraulic permeability of the membrane, which is the slope of the line.
2.2.1.2. Rejection for sulfate ions

Before starting the concentration test, the membranes were characterized for rejection of sulfate ion to monitor their integrity. The rejection was determined using sodium sulfate (1000 mg L\(^{-1}\)) solution at pH 9.4 and 1.5 MPa. After determining the hydraulic permeability, water was removed and filled with sulfate solution and aliquot of 50 mL was taken away and reserved together with a sample of the feed solution. The concentrations of sulfate ions in the feed and permeates were determined by conductivity (Digmed DM-23 digital conductivity meter). The rejection of the membranes was estimated by the rejection factor (R). It is defined as the fraction of solute retained by the membrane for a given concentration of feed solution, according to Eq. (2) [2].

\[
R(\%) = \frac{C_f - C_p}{C_f} \times 100
\]  

(2)

where \(C_f\) is the sulfate concentration in the feed and \(C_p\) is the concentration in the permeate.

2.3. Permeation and Concentration Tests of the Waste

2.3.1. Permeation tests of the waste

The aim of this test was to evaluate the performance of membranes NF0 and SW0 as the rejection of uranium and permeated flux with permeation time. This test was carried out with permeated return to the feed tank. After compaction, section 2.2.1.1, the water was removed and added 2.5 L of waste in the feed tank. The system was turned on and 5 min awaited to circulate the waste before to apply pressure. Then the pressure was slowly applied in the system until 1.5 MPa. After stabilization of the permeated flux was measured and waited 1 h to make new flux measurement. Permeated was collected each 3 h and the flux measured and the output of permeated returned to the feed tank. The aliquots of permeated collected were identified and reserved for determination of the uranium concentration. The same procedure for the SW0 membrane was repeated.

2.3.2. Concentration test of the waste

At this experiment was evaluated as rejection membrane permeation is performed continuously, i.e. without return the solution permeated to the feed tank. This condition simulates an industrial operation. In this trial NF0 and SW0 membranes were tested using the same procedure mentioned in section 2.3.1, however the permeated was continuously taken until approximately 80% volume with respect to feed solution. A minimum volume, approximately 0.5 L, it is necessary to equipment is not automatically turn off. The permeated were collected aliquots of the 500 ml each. Flux was measured along permeation until minimum volume. The aliquots of permeated and concentrate were identified and reserved for determination of the uranium concentration. The same procedure for the SW0 membrane was repeated.
2.3.3. Rejection for uranium

The rejection for uranium of the membranes was determined after permeation and concentration tests of the waste; aliquots of 50 mL were taken away and reserved together with a sample of the feed solution. The rejection of the membranes was estimated by the rejection factor (R), according to Eq. (2). The uranium was determined by arsenazo(III) method ($\lambda = 650$ nm). The readings were inserted in the respective analytical curves for then calculation of rejection (R).

2.4. Morphological and Chemical Structure of the Surface Layer

2.4.1. Attenuated total reflection Fourier transform infrared spectroscopy (ATR - FTIR)

The surfaces layer from the NF0 and SW0 membranes after tests were washing with Milli-Q water followed was washed with acetone and dried at 25°C. ATR-FTIR spectra were obtained in a Nicolet 6700-Thermo Scientific instrument with a ZnSe crystal at an incidence angle of 45° (0.4–0.6 µm depth) [6,23]. Each spectrum results from 64 scans collected from 500-4000 cm$^{-1}$ at 2 cm$^{-1}$ resolution at room temperature.

2.4.2. Scanning electron microscopy by field emission (FESEM)

The membrane was immersed in ethanol for 24 h followed by 24 h in n-hexane, dried at 25°C for 30 min, followed by drying at 60°C for 30 min. This procedure intends to avoid the collapse of the porous surface due to the high surface tension of water. The treated sample was placed on the sample holder and coated with gold to provide electrical conductivity to the membrane. The top surface morphology of the membrane was studied using a JEOL JSM6710F microscope.

2.4.3. Atomic force microscopy (AFM)

AFM images were taken on a WiTec Raman Sensitivity instrument. The silicon cantilever with nominal tip apex radius of 10 nm was used to scan the surface morphology of membranes at the contact mode. This technique allows the estimation of the pore size and the roughness of the nanofiltration membrane [24,25]. The images were obtained over an area of $2 \times 2$ µm for three different surfaces for both before and after concentration tests of the concentration of the waste. The images were analyzed with an AFM image program for determine the roughness.
3. RESULTS AND DISCUSSION

3.1. Transport Properties of the Membranes

3.1.1. Permeate flux, hydraulic permeability and sulfate rejection

Figure 1 shows the graph of permeate flux versus pressure of the NF0 and SW0 membrane samples. The slope is the hydraulic permeability of each sample, and the values found were around of the 50 and 42 L m\(^{-2}\) h\(^{-1}\) MPa, respectively. These values fall in the range found in the literature for nanofiltration membranes (10-66 L m\(^{-2}\) h\(^{-1}\) MPa) [3]. The hydraulic permeability can be correlated with the hydrophobic/hydrophilic character (affinity with water) of the monomer used in the selective membrane layer. NF membranes usually have a selective layer of polyamide, which is hydrophilic [2,3].

![Figure 1. Graph of permeate flux versus pressure of the membranes NF0 and SW0](image)

The Tables 1 and 2 show the values sulfate rejection and permeated flux before and after permeation and concentration tests of the waste in the system tangential flux of the membranes NF0 and SW0. This characterization was carried out with the aim of monitor the performance of the membranes after concentration test of the waste.
Table 1. Permeated flux and rejection sulfate before and after permeation test of the waste system tangential flux to the membrane NF0 and SW0.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Pressure (MPa)</th>
<th>Initial flux* (L m(^{-2}) h(^{-1}))</th>
<th>Rejection SO(_4^{2-}) (%)</th>
<th>Final flux* (L m(^{-2}) h(^{-1}))</th>
<th>Rejection SO(_4^{2-}) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NF</td>
<td>1.5</td>
<td>79.2</td>
<td>84.0</td>
<td>82.7</td>
<td>81.7</td>
</tr>
<tr>
<td>SW</td>
<td>1.5</td>
<td>63.5</td>
<td>84.0</td>
<td>96.0</td>
<td>50.0</td>
</tr>
</tbody>
</table>

* Flux determined with water before and after concentration of the waste, pH 9.4

Table 2. Permeated flux and rejection sulfate before and after concentration tests of the waste system tangential flux to the membrane NF0 and SW0.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Pressure (MPa)</th>
<th>Initial flux* (L m(^{-2}) h(^{-1}))</th>
<th>Rejection SO(_4^{2-}) (%)</th>
<th>Final flux* (L m(^{-2}) h(^{-1}))</th>
<th>Rejection SO(_4^{2-}) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NF</td>
<td>1.5</td>
<td>79.0</td>
<td>84.0</td>
<td>78.0</td>
<td>82.3</td>
</tr>
<tr>
<td>SW</td>
<td>1.5</td>
<td>82.0</td>
<td>83.0</td>
<td>80.0</td>
<td>67.4</td>
</tr>
</tbody>
</table>

* Flux determined with water before and after concentration of the waste, pH 9.4

3.2 Permeation Test of the Waste

The permeated flux and rejection of the uranium of the NF0 membrane is shown on Table 3. The uranium average rejection was 92%. The results showed a 10% variation in the rejection of uranium (85.7-93.9%) in a total time of permeation of the 260 h. This value is satisfactory, considering that this test the permeated returned to the feed tank after permeated sample withdrawal.

P0 refers to first measure, flux circulation of the waste after a few minutes, with flow of 16.6 L m\(^{-2}\) h\(^{-1}\). Compared to the initial flux, 79.2 L m\(^{-2}\) h\(^{-1}\) (Table 1), there was a considerable reduction in the flux, approximately 80%. This reduction may be due to the effect of concentration polarization phenomenon inherent to all membrane separation process [26]. After circulation of 1 h, flux was taken (P1h), reduction of the 24% until time 100 h.

After 172 h almost the flux did not vary, stabilizing at 9.3 L m\(^{-2}\) h\(^{-1}\) until the end of the 260 h permeation. After permeation, the waste was removed and a new sulfate rejection was measured showing that the rejection was slightly lower (81.7%), suggesting that the charge layer selective was not significantly altered [2, 3].
Table 3. Rejection of uranium and permeated flux in the permeation test system tangential flux to the membrane NF0

<table>
<thead>
<tr>
<th>Permeated</th>
<th>Permeation time (h)</th>
<th>Flux (L m(^{-2}) h(^{-1}))</th>
<th>Rejection U (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P0</td>
<td>-</td>
<td>16.6</td>
<td>-</td>
</tr>
<tr>
<td>P1h</td>
<td>1</td>
<td>12.6</td>
<td>-</td>
</tr>
<tr>
<td>P1</td>
<td>4</td>
<td>9.3</td>
<td>85.7</td>
</tr>
<tr>
<td>P2 - P6</td>
<td>102</td>
<td>7.5</td>
<td>89.8</td>
</tr>
<tr>
<td>P7 - P10</td>
<td>147</td>
<td>8.3</td>
<td>93.2</td>
</tr>
<tr>
<td>P11 – P13</td>
<td>172</td>
<td>9.3</td>
<td>92.3</td>
</tr>
<tr>
<td>P14 – P18</td>
<td>260</td>
<td>9.3</td>
<td>93.9</td>
</tr>
</tbody>
</table>

Average = 92.0 ± 2.9 %

The Table 4 shows the values of retention of uranium and permeated flux obtained in the test for membrane permeation SW0. The membrane SW0 results showed slightly smaller than NF0 membrane with uranium average rejection of 90% in a period around 500 h. This result was satisfactory, showing that the membrane is stable to reject the conditions studied.

P0 flux showed 15.9 L m\(^{-2}\) h\(^{-1}\), compared to the initial flux of 63.5 L m\(^{-2}\) h\(^{-1}\) (Table 1) observing a 75% reduction in the flux, similar behavior was observed with the NF0 membrane showing the same effect of concentration polarization. After 1 h of circulation (P1h), 40% reduction in flux was observed. After 3 h the permeation flux was still low, and the permeation from that time until around 400 h the flux increased.

However, is noted that the uranium rejection remained nearly constant proving again that the charge of the selective layer of the membrane was not significantly altered since the rejection of multivalent ions, in this case the [UO\(_2\) (CO\(_3\))\(_3\)]\(^{4-}\) preferentially occurs via charge exclusion [27]. After 500 h permeation uranium rejection was reduced and the flux continued to increase. After permeation, the waste was taken out and fresh rejection was determined sulphate (50.0%). Removing the membrane module it was observed that the same showed damaged areas, probably caused by the constant pressure relief due to the long breakthrough time.
Table 4. Rejection of uranium and permeated flux in the permeation test system tangential flux to the membrane SW0

<table>
<thead>
<tr>
<th>Permeated</th>
<th>Permeation time (h)</th>
<th>Flux (L m(^{-2}) h(^{-1}))</th>
<th>Rejection U (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P0</td>
<td>-</td>
<td>15.9</td>
<td>-</td>
</tr>
<tr>
<td>P1h</td>
<td>1</td>
<td>9.5</td>
<td>-</td>
</tr>
<tr>
<td>P1</td>
<td>4</td>
<td>8.2</td>
<td>85.2</td>
</tr>
<tr>
<td>P2 – P7</td>
<td>102</td>
<td>11.0</td>
<td>89.5</td>
</tr>
<tr>
<td>P8 - P15</td>
<td>386</td>
<td>15.4</td>
<td>92.1</td>
</tr>
<tr>
<td>P16 – P17</td>
<td>410</td>
<td>19.5</td>
<td>91.1</td>
</tr>
<tr>
<td>P18</td>
<td>500</td>
<td>22.3</td>
<td>77.2</td>
</tr>
</tbody>
</table>

Average = 90.6 ± 3.0%

P0 - beginning of the permeation
P1h - after 1 h of the permeation

3.3 Concentration Test of the Waste

The concentration of the waste test was carried out in continuous mode permeation, i.e. without returning the solution permeated for the feed tank. The waste "carbonated water" contains uranium 235 \(^{235}\)U and concentration around 7 mg L\(^{-1}\), which has high value. Thus, their recovery could return to the productive process of uranium pellets, as well as reducing the volume of waste to be stored. The rejection values of uranium and permeate flux obtained in the concentration test of the waste with the NF0 and SW0 membranes are shown in Tables 5 and 6. The rejection values no showed large changes during the whole time of the tests, with 97% of the average for both membranes. This value is higher compared to values obtained from the permeation tests (with return permeate to the feed tank) with the NF0 and SW0 membranes, of the 90%.

It can be observed that the flux from both membranes is smaller than the permeation test. This fact can be explained based on the characteristics of the feed solution. Increasing the concentration of a specific ion in the feed solution leads to increased in rejection. This increase depends of the valence and the diffusion coefficient of the cations and the degree of hydration [28,29].

The concentration tests lasted for 170 hr. Permeate was collected in a 500 ml each concentrated and about 400 mL. In this test the volume reduction of the waste was 82%, which is very important in the case of storage. The permeated contains still around 0.60 mg L\(^{-1}\) uranium, above the limit established to discharge to the environment \((2 \times 10^{-2} \text{ mg L}^{-1})\) [30], which may undergo another process for membrane separation, reverse osmosis, which due to porosity low allows only the passage of water (solvent) [3,26].
Table 5. Rejection of uranium and permeated flux in the concentration test system tangential flux to the membrane NF0

<table>
<thead>
<tr>
<th>Permeated</th>
<th>Rejection of the U (%)</th>
<th>Flux (L m$^{-2}$ h$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P0</td>
<td>-</td>
<td>6.3</td>
</tr>
<tr>
<td>P1h</td>
<td>-</td>
<td>6.3</td>
</tr>
<tr>
<td>P1</td>
<td>96.8</td>
<td>6.2</td>
</tr>
<tr>
<td>P2</td>
<td>96.5</td>
<td>6.7</td>
</tr>
<tr>
<td>P3</td>
<td>97.5</td>
<td>6.7</td>
</tr>
<tr>
<td>P4</td>
<td>97.7</td>
<td>7.5</td>
</tr>
<tr>
<td>Average</td>
<td>97.0 ± 1.0</td>
<td>6.8 ± 0.6</td>
</tr>
</tbody>
</table>

Concentrated 31.0 mg L$^{-1}$

Expected 36.1 mg L$^{-1}$

P0 - beginning of the permeation
P1h - after 1 h of the permeation

Table 6. Rejection of uranium and permeated flux in the concentration test system tangential flux to the membrane SW0

<table>
<thead>
<tr>
<th>Permeated</th>
<th>Rejection of the U (%)</th>
<th>Flux (L m$^{-2}$ h$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P0</td>
<td>-</td>
<td>9.3</td>
</tr>
<tr>
<td>P1h</td>
<td>-</td>
<td>8.2</td>
</tr>
<tr>
<td>P1</td>
<td>94.3</td>
<td>8.6</td>
</tr>
<tr>
<td>P2</td>
<td>95.2</td>
<td>8.8</td>
</tr>
<tr>
<td>P3</td>
<td>95.5</td>
<td>9.2</td>
</tr>
<tr>
<td>P4</td>
<td>97.6</td>
<td>9.2</td>
</tr>
<tr>
<td>Average</td>
<td>96.0 ± 1.5</td>
<td>9.0 ± 0.3</td>
</tr>
</tbody>
</table>

Concentrated 35.4 mg L$^{-1}$

Expected 40.6 mg L$^{-1}$

3.4 Characterization of the Membrane Surface

3.4.1 ATR-FTIR data

The infrared spectra of the selective layer recovered before and after the concentration test are presented in Figure 2 (a) and (b), respectively. The profiles are very comparable themselves since the spectra are overlapped. This suggests that the integrity of the membranes was not affected by the waste. The characteristic N–H polyamide bands (3500-3200 cm$^{-1}$), C–H aliphatic (~2900 cm$^{-1}$), C=O and aromatic rings (1700-1550 cm$^{-1}$) are well noticeable. The spectrum of the Figure 2 (b), membranes after concentration of the waste is different from the previous ones (Figure 2 (a), in particular, the band in the region 3500-3200 cm$^{-1}$ show almost overlapping, probably due to tangential flux system.
Figure 2. ATR-FTIR spectra of the membranes NF0 and SW0: (a) before concentration of the waste and (b) after concentration of the waste.

3.4.2. FESEM images

The morphological aspects of the NF0 and SW0 membrane surface before of the concentration of the waste are presented in Figure 3 (a) and (b), respectively. Figure 3 (a) shows the original (NF0) membrane surface, which appears to be completely smooth without apparent porosity, even after magnification of 100,000x. The SW0 membrane, Figure 3 (b), shows a granular structure can be distinguished. The membrane surface after concentration of the waste was observed by AFM.
3.4.3. AFM data

The AFM images of the surface topography of the membrane originals (NF0 and SW0) and after concentration test of the waste (NF conc. and SW conc.) are shown in Figure 4. It is observed that a surface of the NF0 and SW0 membranes contains many thin peaks, which are responsible for the roughness, the membranes showed roughness value (Rms) of the 2.67 nm and 2.00 nm respectively. This result suggests peaks height very similar. This roughness of these membranes refers to the measurements performed in the area 4 µm² as a reference to compare the values with the membranes after concentration of the waste. Comparing the images, it is clear that the surface roughness was reduced after test concentration. It can be observed that both images showed smoother surfaces, homogeneous. The membranes showed roughness value (Rms) of the 1.38 nm and 1.23 nm respectively. This may be related to the fact that the system used tangential flux, which it fluxes parallel to the membrane surface.
4. CONCLUSIONS

The concentration of the waste was satisfactory for separation of uranium from the waste to both membranes, showed rejections practically constant during the tests. The membranes showed 97% of rejections and an average concentrate containing 33.0 mg L$^{-1}$ uranium.

The ATR-FTIR spectra before and after concentration tests presented the same band profile, suggesting that the integrity was not affected by the test.

The scanning electron microscope by field emission (SEM-FE) was very useful to examine the surface morphology of the membranes before tests concentration, allowing to visualize clearly the difference between the two membranes studied.

The roughness value of the NF conc. and SW conc. membranes was lower than that of the NF0 and SW0. This may be related to the fact that the system used tangential flux, which it fluxes parallel to the membrane surface.
ACKNOWLEDGMENTS

We acknowledge INB for providing the waste and Dow/Brazil for furnishing the membrane employed this work.

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