

# Preparation, Characterization, and Adsorption Properties of Cellulose Acetate-Polyaniline Membranes

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**ABSTRACT:** Four lots of cellulose acetate (CA) membranes, modified with polyacrylic acid, using various plasticizers, and coated with polyaniline (PANI) were prepared. The morphology of the membranes was evaluated by using scanning electron microscopy, and the membranes showed larger pore size when the plasticizers were used. The electrical conductivity of the modified membranes and coated with PANI increased by two orders of magnitude when the plasticizer triphenyl phosphate was

used. The strain at break improved by an order of magnitude and the glass transition temperature ( $T_g$ ) showed an average decrease of 36°C when the membranes were plasticized. Finally, these membranes were tested as ion-exchange materials of a gold-iodide complex. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 111: 1216–1224, 2009

**Key words:** membranes; conducting polymers; mechanical properties; ion exchangers

## INTRODUCTION

Polyaniline (PANI) is an intensively studied polymer due to its electrical, electrochemical, and optical properties and due to its high environmental stability, which confers it potential use in many applications including sensors, rechargeable batteries, anticorrosion coatings, electrochromic displays, selective membranes, etc.<sup>1–6</sup> Nowadays, a number of compound materials based on mixtures of PANI with insulating materials such as cellulose acetate (CA)<sup>7–9</sup> and poly(methyl methacrylate)<sup>10</sup> have been synthesized, resulting in good electrical conductivity. The polymeric membranes used for the processes of separation and filtration is a field of attractive study for the scientific community, which is attributed to its application in day-to-day life, a clear example of which is, water purification and desalination via reverse osmosis.<sup>11–13</sup>

Membranes formed with conjugated polymers have been studied because of their transport properties, because they can function as potential membranes for separating molecules from fluids. This property can be improved with a support matrix that provides mechanical strength and facilitates the transport of the molecules throughout the mem-

brane. This concept has been applied to different kinds of support matrices such as polycarbonate, cellulose, and CA.<sup>14</sup> With the objective of conferring flexibility to CA matrices, the mixture of plasticizers of phthalic and phosphoric acid esters has been used.<sup>15</sup> CA is commonly used to prepare membranes for different uses and various methods have been developed to control its pore diameter using polyelectrolytes.<sup>16</sup> The presence of residual polyelectrolytes in the inner wall of the pore could function as a template to fasten electroconductive polymers to the membrane and to improve the electroconductive properties. The purpose of creating electrically conductive membrane (called electromembrane) is its potential application in processes of electrodialysis in ion separation.<sup>17</sup> Togwen Xu and coworkers<sup>18,19</sup> carried out some works related to electrodialysis using bipolar membrane reporting applications in organic acid production and regenerating flue-gas desulfurizing agents.<sup>20,21</sup> Also a new route for preparing an anion exchange membrane using linear engineering plastics has been reported.<sup>22</sup> Mirmohseni and Saeedi (1998)<sup>23</sup> carried out the first report on the separation by electrodialysis of a mixture of H<sub>2</sub>SO<sub>4</sub>/H<sub>3</sub>PO<sub>4</sub> and HNO<sub>3</sub>/H<sub>2</sub>SO<sub>3</sub> using PANI membranes. Sata and Ishii (1999)<sup>24</sup> reported that the transport properties in electrodialysis of composite membranes prepared from cation exchange membranes and PANI, and these composite membranes showed sodium ion permselectivity in electrodialysis when compared with divalent cations at an

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optimum polymerization time. Nagarale et al. (2004)<sup>25</sup> developed composite membranes by polymerization of a thin layer of PANI in the presence of a high oxidant concentration on a single face of a sulfonated cation-exchange membrane and quaternary aminated anion-exchange membranes. Relative dialytic rates of Na<sub>2</sub>SO<sub>4</sub>, CaCl<sub>2</sub>, and CuCl<sub>2</sub> were estimated with reference to NaCl on the basis of electro-dialysis experiments, and it was concluded that it is possible to separate different electrolytes using PANI composite ion-exchange membranes. Tan and Belanger (2005)<sup>26</sup> modified Nafion membranes by polymerization of aniline using ammonium peroxydisulfate as the oxidant. It is demonstrated that the ionic transport of both H<sup>+</sup> and Zn<sup>2+</sup> will be dependent on the amount of PANI disrupting the ionic paths and not necessarily the total amount of PANI. Sivaraman et al. (2007)<sup>27</sup> reported the electrochemical modification of cation exchange membrane with PANI for improvement in permselectivity. Acrylic acid grafted fluorinated ethylene propylene copolymer after sulphonation (FEP-g-AA-SO<sub>3</sub>H) was used as the cation exchange membrane for modification. PANI is deposited electrochemically on the membrane. The electro-dialysis experiment shows improved permselectivity for the divalent cations. On the other hand, Montes Rojas (2007)<sup>28</sup> reported an easy method to modify the exchange membranes of electro-dialysis with electrosynthesized PANI. The polarization curves used in electro-dialysis of a nitrate solution showed some differences when using a modified membrane, possibly due to concurrent proton migration. Amado et al. (2004)<sup>29,30</sup> developed composite membrane of high-impact polystyrene/PANI membranes for acid solution treatment by electro-dialysis; in this study, it was found that a synthesized membrane can be used to recover zinc in acid media. They have been reported also the development of polyurethane/PANI membranes for zinc recovery through electro-dialysis, being that these membranes can represent an alternative to commercial membrane for the metal finishing industry.<sup>31</sup>

In addition, these electromembranes differs from the conventional ion-exchange membranes due to the nature of the separation mechanism. The separation in an electromembrane is based on the electrical potential and/or magnetic field "barrier" generated by passing current through the membrane, whereas the separation in a conventional ion-exchange membrane relies on the relative ionic affinity of the compounds being separated.<sup>17</sup>

Heap leaching (HL) is a geotechnological method for processing of low-grade gold-containing ores and rejects of concentration plants. Alkaline solutions of cyanides are used in enormous amounts as a lixiviant for gold, which gives rise to quite a number of ecological problems; therefore, the use of al-

ternative lixiviant has been developed. The system iodine iodide has been suggested to extract gold of several sources.<sup>32</sup> The main advantages of this process are high stability of the solution, low oxidation potential, and low possibility of reaction between iodine and sulfurous minerals.

In our study, we considered two main objectives: The first was to prepare a novel porous material with better mechanical and electroconductive properties, and our second objective was to evaluate the ion-exchange property of PANI, which has been studied only to a little extent.<sup>33</sup> To obtain an electroconductive membrane with ionic exchange capacity for anions is important because of its potential application in recovery processes in extractive metallurgy.

In the present article, we report the effect of plasticizers on the mechanical properties,  $T_g$  value, morphology, and electrical conductivity of CA membranes modified with polyacrylic acid (PAA) and coated with PANI. A preliminary evaluation of the properties of ionic interchange was made using gold iodine iodide solutions.

## EXPERIMENTAL

### Materials

Materials used were: CA powder, Aldrich. Poly (acrylic acid, sodium salt) (PAA) 35 wt %, w 15,000, Aldrich. Acetic Acid, Glacial, Sigma; Hydrochloric Acid, Merck; Ammonium persulfate, Fermont; Dimethyl phthalate 99% (DMP), Aldrich; Diethyl phthalate 99% (DEP), Aldrich; Triphenyl phosphate 99% (TPP), Aldrich; Potassium iodide, Fermont; Iodine 99.99%, Fermont; Gold, powder 99.99%, Aldrich; Anilina 99%, Merck, was distilled under vacuum in nitrogen atmosphere before use. All other reagents were used as received.

### Preparation of CA membrane

CA membranes without and with PAA (CA and CA/PAA, respectively) and four lots of CA/PAA membranes using different plasticizers was prepared. For the preparation of the lot CA, 4 g of CA was weighed and dissolved in 50 mL of acetic acid, with constant stirring for 4 h. For the preparation of the lot CA/PAA, 4 g of CA was weighed and dissolved in 50 mL of acetic acid, with constant stirring for 4 h, and subsequently, 5 mL of PAA was added under stirring for 1 h. Four lots of plasticized membranes were prepared using the solution CA/PAA as the base. The first lot contains DMP plasticizer (lot A), the second lot contains DEP (lot B), and the third lot contains TPP (lot C). For these three lots, the CA : PAA : plasticizer weight ratio was 100 : 50 : 25. The membranes of lot D were prepared with a

**TABLE I**  
**Composition of Membranes**

Components		Composition in weight parts
CA	Cellulose acetate	100
CA/PAA	Cellulose acetate : poly(acrylic acid)	100 : 50
Lot A	CA : PAA : DMF	100 : 50 : 25
Lot B	CA : PAA : DEF	100 : 50 : 25
Lot C	CA : PAA : TFF	100 : 50 : 25
Lot D	CA : PAA : DMF : DEF : TFF	100 : 50 : 25 : 25 : 2

mixture of the three plasticizers, using a DMP : DEP : TPP weight ratio of 25 : 25 : 2. This ratio is commonly used to plasticize CA.<sup>15</sup> The composition of membranes is shown in Table I.

Membranes were prepared by phase inversion in a wet process. An aliquot of the solution corresponding to each lot of membrane was poured onto a flat glass plate of  $\sim 10$  cm diameter. The excess of the solution was removed, leaving the plate covered by a fine layer. It was then placed in a coagulation bath (ice water mixture) for 30 s. The plate with the solution was then immersed in the same cold water for 15 min. Finally, the membrane was peeled off from the glass plate, washed in the same water, and dried at 25°C for 24 h on filter paper.

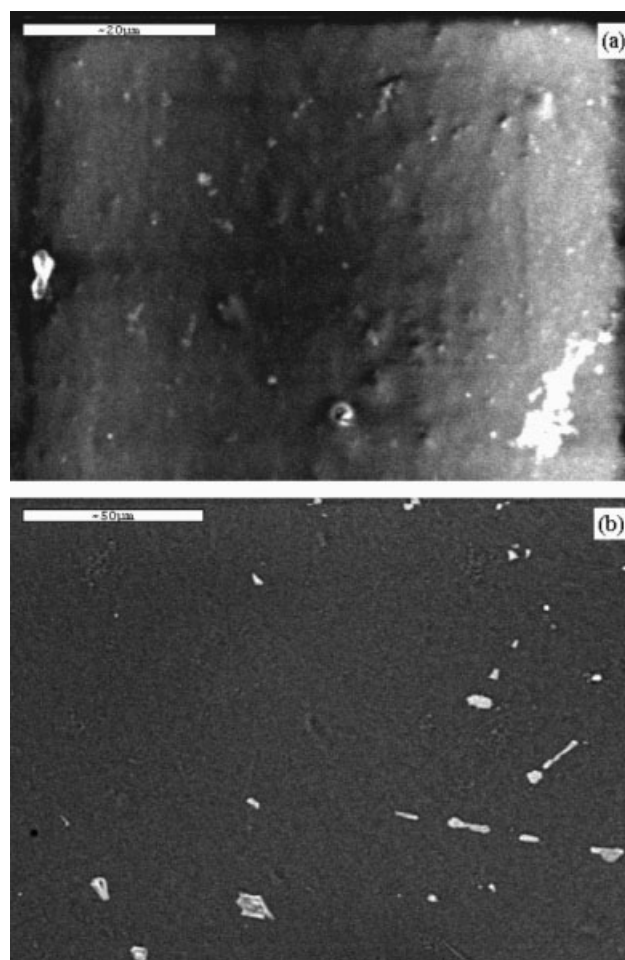
#### Coating of membranes with PANI

A solution 0.5M of aniline was prepared. Aniline was dissolved in 0.02M aqueous solution of HCl. Membranes were cut into strips to facilitate their coating and were placed in a glass vessel containing the aniline solution. The strip was immersed in the solution for 5 min. The strip was retired, drained, and placed in a glass vessel containing a solution of ammonium persulfate 0.5M, for 5 min. Subsequently, membranes were dried at 25°C for 24 h.

#### Characterization

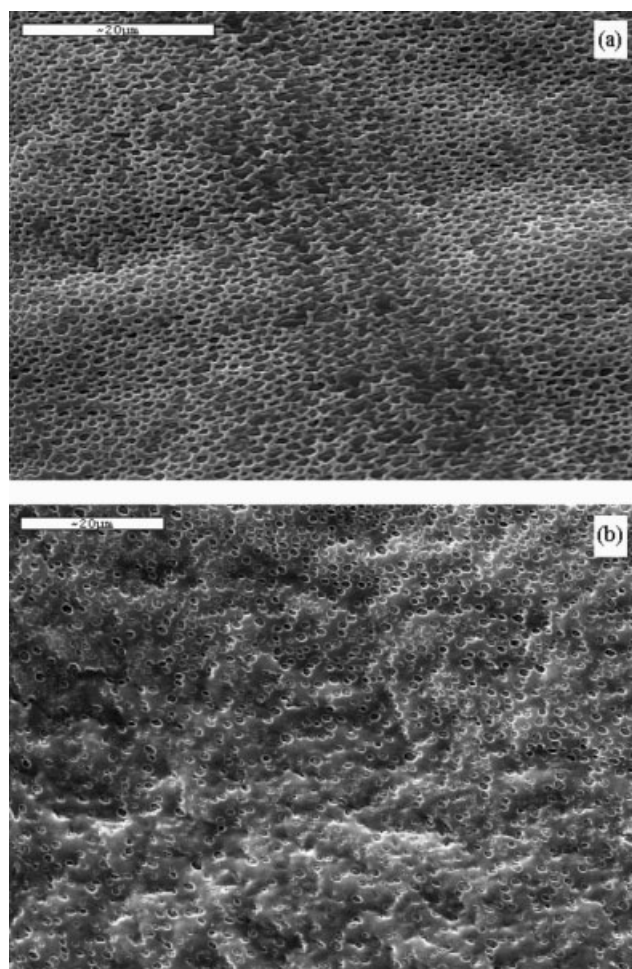
The morphology of the membranes was evaluated by using a JEOL 5410LV scanning electron microscopy (SEM), operated at 15 kV. The electrical conductivity of the membranes was measured by the standard two-point probe method. The measurements were done at 25°C with tungsten electrodes of 6 mm diameter and a Proam multimeter. The evaluation of the mechanical properties of membranes was made following norm ASTM D 1708-93 for microtensile specimens. The samples were conditioned for 1 week to 23°C and 50% of humidity. Tests of stress and strain at break were performed with a speed of 0.1 mm min<sup>-1</sup>, using a universal machine Tinius Olsen 1000. Differential thermal

analysis (DTA) was carried out on an equipment SDT 2960 simultaneous DSC-TGA TA instruments. Samples were taken approximately from 4 mg and it was warmed up until 600°C at a heating and cooling rate of 10°C min<sup>-1</sup> under a flow of 23 mL min<sup>-1</sup> of air. To test potential application as ion-exchange membrane, a leaching solution of 12 g iodine total (I<sub>2</sub> + KI) was used, with a KI to I<sub>2</sub> ratio of 2 : 1. The gold-iodide complex was prepared using the leaching solution, to which a predetermined amount of gold was added so that the Au concentration was 20 ppm. The concentration was verified by atomic absorption spectroscopy. Pieces of membranes were submerged in the gold-iodide solution under constant magnetic agitation for 12 h, after which the pieces of membranes were immediately removed from the gold-iodide solution. The concentration of Au in the solution was analyzed by atomic absorption spectroscopy. The gold concentration in the gold-iodide solutions was determined with a Perkin Elmer 3110 atomic absorption spectrometer.



**Figure 1** SEM micrographs of the surface of cellulose acetate membranes without poly(acrylic acid), CA, with magnification of  $\times 750$  (a) uncoated (b) coated with PANI.





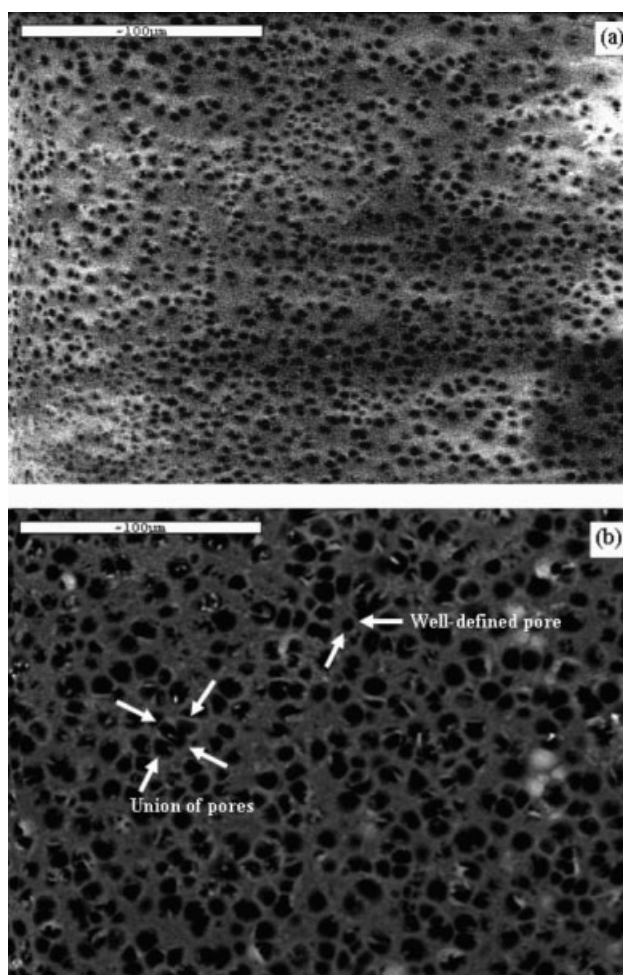
**Figure 2** SEM micrographs of the surface of cellulose acetate membranes modified with poly(acrylic acid) (CA/PAA) with magnification of  $\times 750$  (a) uncoated (b) coated with PANI.

## RESULTS AND DISCUSSION

When PAA was added to CA membranes, a material with porosity needed for its potential application in ion-exchange processes was obtained. Figures 1 and 2 show the SEM micrographs of the surface of lots of membranes before and after coating with PANI, respectively. Figure 1 shows CA membranes without PAA, (CA), and the membranes does not shown appreciable porosity. The membrane without coating presented a clear color [Fig. 1(a)] and that with one coating presented a thin, dark layer corresponding to the PANI [Fig. 1(b)]. Figure 2 corresponds to CA membranes with PAA, and it was observed that the initial porosity of membranes is due to the modification with the PAA, showing pores with diameter between 0.5 and 2  $\mu\text{m}$  [Fig. 2(a)], and with PANI the pore size was unaffected appreciably [Fig. 2(b)].

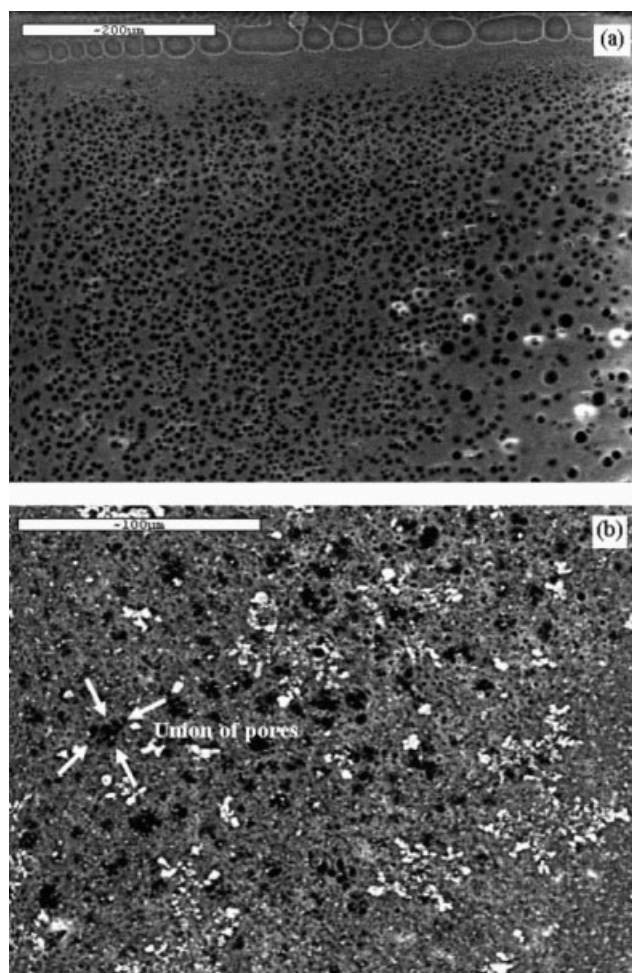
To improve the mechanical properties of CA membranes, three plasticizers were added. Figures 3–6 show the SEM micrographs of the surface of lots

of plasticized membranes before and after coating with PANI. The pore size in all lots using any plasticizer was greater than that in unplasticized membranes. This increase is attributed to the partial dissolution of plasticizer in water during the membrane formation in the coagulation bath. Figure 3(a,b) shows the SEM micrograph of a membrane lot A before and after coating with PANI, respectively. The pore size before coating ranges from 2 to 6  $\mu\text{m}$  [Fig. 3(a)]. Two types of pores can be observed in the membrane coated with PANI [Fig. 3(b)], well-defined pore with diameter between 3 and 11  $\mu\text{m}$  and larger pores formed by the union of two or more pores. This union of pore surface is attributed to a small degradation of the membrane, due to contact with the solution of aniline dissolved in aqueous HCl. In the case of lot B, membranes without coating have a pore size ranging from 3 to 14  $\mu\text{m}$  [Fig. 4(a)] and after coating with PANI there occurs union of pores, creating larger pores and irregular diameter,



**Figure 3** SEM micrographs of the surface of membranes of lot A with magnification of  $\times 500$  (a) uncoated (b) coated with PANI.





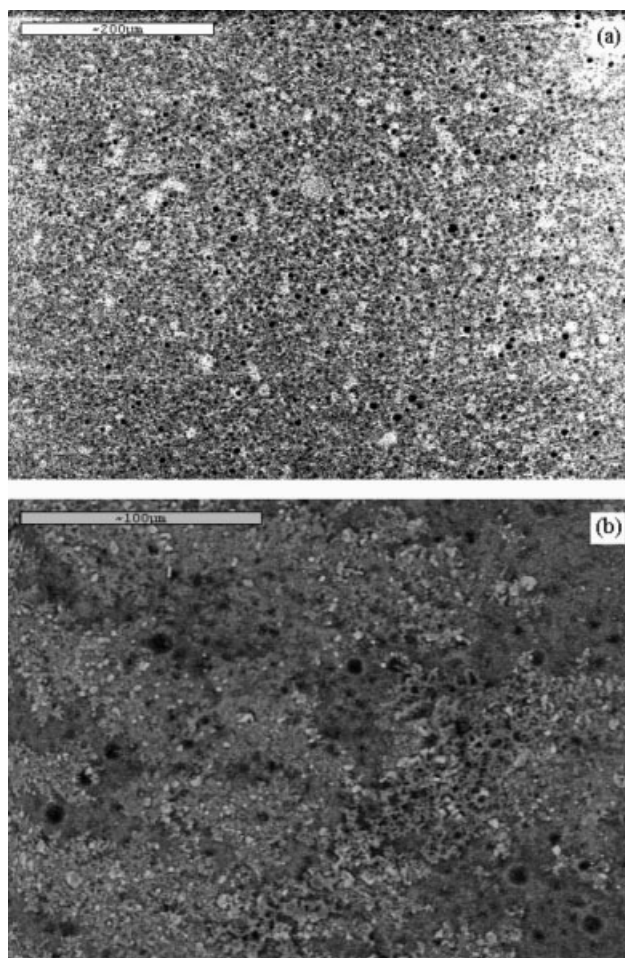
**Figure 4** SEM micrographs of the surface of membranes of lot B with magnification of  $\times 500$  (a) uncoated (b) coated with PANI.

which varies from 6 to 15  $\mu\text{m}$  [Fig. 4(b)]. In the case of lot C, no significant difference was observed in the pore size before and after coating with PANI. The pore size of this lot without coating was 3–9  $\mu\text{m}$  [Fig. 5(a)] and after coating the pore size was 3–10  $\mu\text{m}$  [Fig. 5(b)]. Membranes lot D without coating shows a very large pore size of up to 154  $\mu\text{m}$  [Fig. 6(a)]. We can clearly observe smaller pores within the larger pores, making it obvious that the membrane presents an asymmetric structure. The superficial pore size is unaffected appreciably by coating with PANI [Fig. 6(b)].

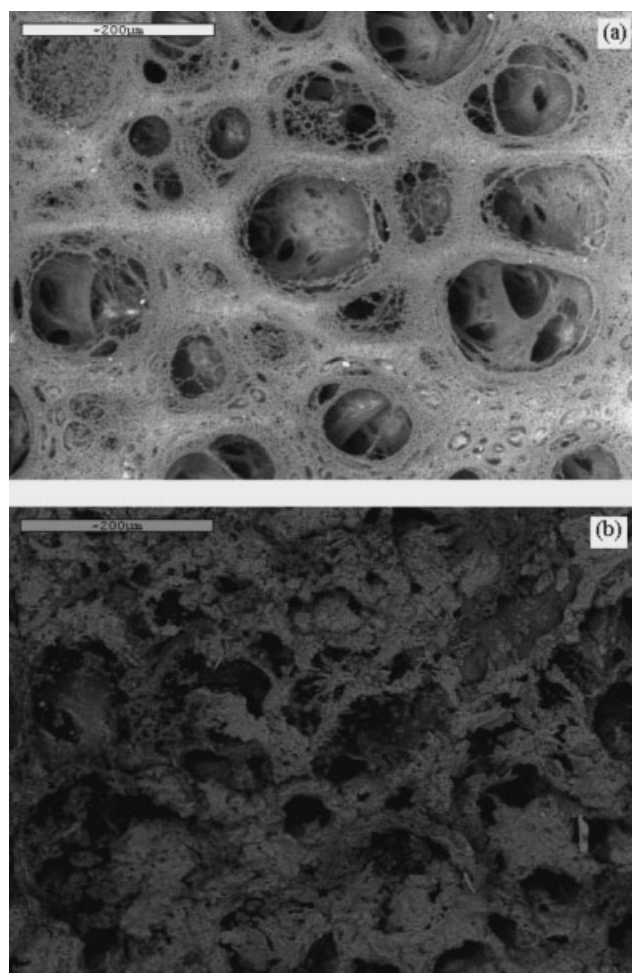
The increased pore size in membranes of lots A and B containing plasticizers DMF and DEF, Figure 3(b) and 4(b) respectively, coated with PANI is attributed possibly to an acid hydrolysis of the plasticizers on the surface of membrane, and this hydrolysis could be generated by the aqueous HCl, which functions as a solvent for aniline during the coating of the membrane. To confirm this hypothesis, the simulation of these reactions was realized using the

software HiperChem. The values for Gibbs free energy change ( $\Delta G$ ) of reaction of membrane lots A and B were  $-2618 \text{ Kcal mol}^{-1}$  and  $-3358 \text{ Kcal mol}^{-1}$ , respectively. Whereas the value in membranes of lot C was  $115,987 \text{ Kcal mol}^{-1}$ ; these results show that in lots A and B a hydrolysis reaction of plasticizer can take place, whereas for the lot C this reaction is impossible because of the high value for  $\Delta G$  of reaction. The hydrolysis reactions proposed are shown in Figure 7.

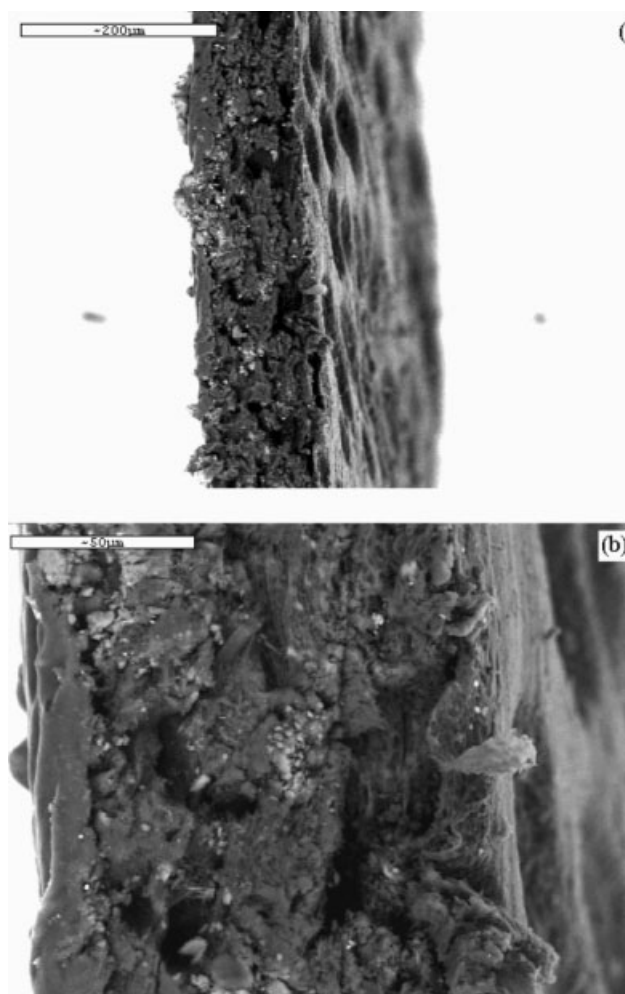
Figure 8 shows the SEM images of the cross-section of a membrane of lot A. With an increase of magnification to  $200\times$ , the pores of the surface and cross-section can be observed [Fig. 8(a)], which clearly shows that the pores of the surface are larger than the pores on the cross-section, and with an increase of magnification to  $750\times$  [Fig. 8(b)], it can be observed that the pore on surface can follow several paths to cross to the other side of the membrane, and this is a proof that exist asymmetric structure on the membrane.



**Figure 5** SEM micrographs of the surface of membranes of lot C with magnification of  $\times 500$  (a) uncoated (b) coated with PANI.

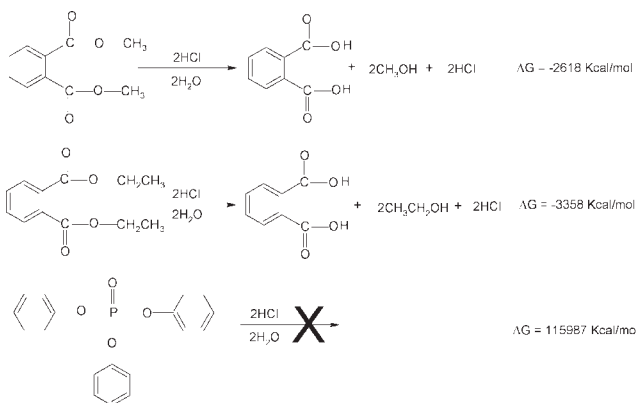


**Figure 6** SEM micrographs of the surface of membranes of lot D with magnification of  $\times 500$  (a) uncoated (b) coated with PANI.



**Figure 8** SEM micrographs of the cross-section of a membrane of lot A coated with PANI (a) with magnification of  $\times 200$  (b) with magnification of  $\times 750$ .

When CA/PAA/plasticizer membranes were coated with PANI, an electroconductive material with potential application in electro dialysis processes and ion exchange were obtained. Table II summarizes the electrical conductivity determined for each lot of membrane coated with PANI. The electrical conductivities of the membranes ranged from  $10^{-8}$  to  $10^{-3} \text{ S cm}^{-1}$  depending on the conditions of preparation. The electrical conductivity of the membranes prepared with polyelectrolyte PAA is three orders of magnitude greater than that of the



**Figure 7** Proposed acid-hydrolysis reaction for plasticizers.

marizes the electrical conductivity determined for each lot of membrane coated with PANI. The electrical conductivities of the membranes ranged from  $10^{-8}$  to  $10^{-3} \text{ S cm}^{-1}$  depending on the conditions of preparation. The electrical conductivity of the membranes prepared with polyelectrolyte PAA is three orders of magnitude greater than that of the

**TABLE II**  
Electrical Conductivity Values for Each Lot of Membrane Coated with Polyaniline

	Electrical conductivity ( $\text{S cm}^{-1}$ )
AC-PANI	$1.03 \times 10^{-8}$
AC/PAA/PANI	$1.51 \times 10^{-5}$
Lot A-PANI	$3.68 \times 10^{-4}$
Lot B-PANI	$2.21 \times 10^{-4}$
Lot C-PANI	$1.37 \times 10^{-3}$
Lot D-PANI	$5.57 \times 10^{-4}$



**TABLE III**  
Mechanical Properties of Unplasticized and Plasticized Membranes

Membrane	Stress at break (MPa)	Strain at break (%)
AC	25.23 ± 4.81	3.8 ± 0.2
AC/PAA	9.38 ± 1.29	0.2 ± 0
Lot A	1.81 ± 0.11	2.6 ± 0.5
Lot B	2.02 ± 0.18	3.8 ± 0.4
Lot C	1.86 ± 0.04	3.5 ± 0.3
Lot D	1.74 ± 0.13	3.2 ± 0.4
<sup>a</sup> Lot C	0.75 ± 0.11	4.4 ± 1.6

<sup>a</sup> Coated with PANI.

membranes without PAA. This result could be correlated to the pore formation. The nonporous membranes without PAA (AC-PANI) have no electrical path percolation, whereas membranes with pores provide the path percolation through PANI on the cross-section of the membrane, and in this way, an electrically conductive membrane is obtained. In addition, the PAA serves as a template for the polymerization of aniline, PANI, thus aniline is polymerized in a more orderly manner, resulting in longer chains of PANI with fewer ramifications, and therefore a better electrical conductivity value is obtained.<sup>34</sup> The incorporation of plasticizers enhances the electrical conduction of the membranes because the plasticizers bring greater order to chains of PAA. We can establish that the plasticizer indirectly helps to have a more ordered structure of PANI facilitating the transport charge carrier, increasing the electrical conductivity. The lot C has the maximal value in electrical conductivity because the plasticizer in this lot, TPP, is a molecule with greater volume than the other plasticizers used, providing higher order to PAA chains and consequently to PANI. These same observations have been reported also by Doufor and supported by WAXD studies.<sup>35,36</sup>

The mechanical properties of the unplasticized and plasticized membranes were studied using stress-strain measurements (Table III), and by means

**TABLE IV**  
 $T_g$  Values Determined by DTA of Unplasticized and Plasticized Membranes

Membrane	$T_g$ (°C)
AC	135.92
AC/PAA	155.97
Lot A	118.99
Lot B	110.71
Lot C	122.80
Lot D	127.88
<sup>a</sup> Lot C	113.86

<sup>a</sup> Coated with PANI.

of DTA, the value of the  $T_g$  for each membrane was determined (Table IV).

When PAA was added to CA membranes, the % strain at break is reduced remarkably from 3.8% to 0.2%, accompanied with an increase in the  $T_g$  value of CA, which indicates that a crossover could exist between the chains of CA and PAA, reducing mobility of polymeric chains and causing a rigidity in the material. In membranes CA/PAA with plasticizers, an increase in % of strain accompanied by a diminution of stress at break and  $T_g$  value was observed. This phenomenon is attributed to the fact that the plasticizers can locate between the chains of polymers, decreasing intermolecular bonding forces where the free sliding of polymeric chains is permitted.<sup>37</sup> We can observe that the membranes to which the mixture of the three plasticizers was added to plasticize CA; according to the concentration recommended in literature,<sup>15</sup> presented values of stress and strain at break are very similar to the values for membranes that involve a single plasticizer. We can observe that the improvement of the mechanical properties obtained with the use of the plasticizer is not significantly affected by the coating with PANI.

A potential application of membranes obtained is as ion-exchange material, due to PANI chains are doped with chloride ions, which could be interchanged with other anions, for example, the gold-iodide complex. Taking advantage of this property, tests of ionic interchange for the gold recovery using as lixiviant solution complex iodine-iodide was realized.

To test the potential application as ion-exchange material, membranes of lot C were used. The result obtained in this experiment is presented in Table V. We can observe that the amount of gold adsorbed by unit of adsorbent mass diminishes with the increase of the solid/liquid ratio. This observation apparently indicates that a low proportion of membrane promotes the most efficient use of the same, possibly due to smaller amount of adsorbent material; the active sites of membranes present a lower

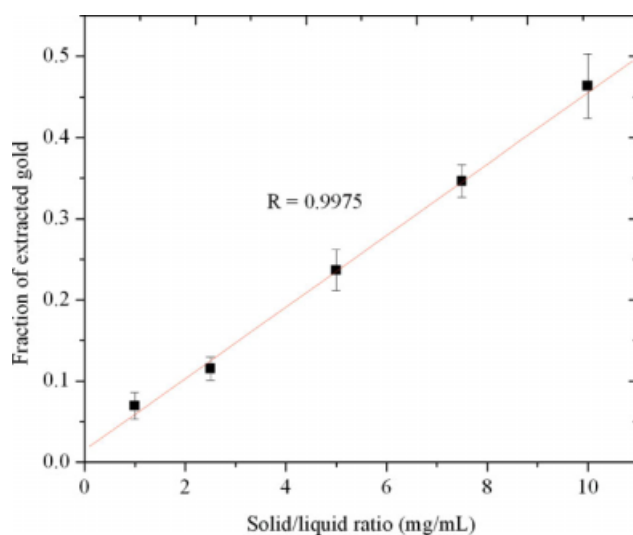
**TABLE V**  
Values Obtained in the Tests of Potential Application as Ion-Exchange Membrane for Gold Recovery from Gold-Iodide Solutions

Solid/liquid ratio (mg mL <sup>-1</sup> )	Gold loading on membrane (mg g <sup>-1</sup> )		Fraction of extracted gold	
	Value	±	Value	±
1	1.2666	±0.1154	0.0629	±0.0051
2.5	0.9466	±0.1222	0.1153	±0.0141
5	0.9466	±0.1006	0.2366	±0.0251
7.5	0.9422	±0.0630	0.3462	±0.0201
10	0.9366	±0.0862	0.4635	±0.0397

competition among them for the process of interchange of chloride ions by the Au-Iodide complex, ( $\text{AuI}_2^-$ ). Nevertheless, to obtain a suitable gold recovery, it is necessary to obtain good results in terms of the fraction of extracted gold. Figure 9 shows the fraction of extracted gold vs. the solid/liquid ratio (mg of membrane/mL solution), it is possible to observe a linear increase of the fraction of extracted gold as the amount of adsorbent material is also increased; this can be explained in terms of a greater capacity of adsorption available in the case of a high value of the solid/liquid ratio. A blank experiment (membrane uncoated with PANI) was realized for each solid/liquid ratio to know ion exchange properties of membrane coated with PANI, (Table VI), for all solid/liquid ratio the fraction of extracted gold is increased with the PANI coating. Finally, we can say that the best gold recovery is obtained for the prepared samples at a solid/liquid ratio of 10.

## CONCLUSIONS

SEM analysis shows that the porosity of CA membranes is due to the modification with the PAA. This porosity is accompanied by an increase in electrical conductivity of  $10^{-8} \text{ S cm}^{-1}$  (nonporous membranes) to  $10^{-5} \text{ S cm}^{-1}$  for membranes with PAA. The use of plasticizers indirectly gives a more ordered PANI, with highest conductivity values,  $10^{-3} \text{ S cm}^{-1}$ , membrane lot C. With the increase in % strain at break and the diminution in  $T_{g'}$ , it can be concluded that the plasticizers improve the mechanical properties of membranes. Finally, good results were obtained



**Figure 9** Effect of the solid/liquid ratio on the fraction of extracted gold by membranes of lot C, using a gold-iodide solution, with an initial gold concentration of 20 ppm.

**TABLE VI**  
**Fraction of Extracted Gold of Membranes without PANI (Blank) and Coated with PANI**

Solid/liquid ratio (mg mL <sup>-1</sup> )	Fraction of extracted gold (blank)	Fraction of extracted gold (with PANI)
1	0.0253	0.0629
2.5	0.0456	0.1153
5	0.0837	0.2366
7.5	0.1294	0.3462
10	0.1852	0.4635

with the use of these membranes in the gold recovery from gold-iodide solutions.

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