

# CAS 2011 PROCEEDINGS

VOLUME 1

## 2011 INTERNATIONAL SEMICONDUCTOR CONFERENCE

October 17-19, Sinaia, ROMANIA

NATIONAL INSTITUTE FOR RESEARCH AND DEVELOPMENT  
IN MICROTehnOLOGIES (IMT-Bucharest)



under the aegis of  
Romanian Academy  
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VOLUME 2

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**2011**  
**INTERNATIONAL**  
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34<sup>th</sup> Edition  
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# POLYSULFONE-POLYANILINE BLEND COMPOSITE MEMBRANE FOR FUEL CELLS APPLICATIONS

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**Abstract**—This paper presents the synthesis and characterization of a composite polysulfone-polyaniline membrane for electrochemical applications. The polysulfone membrane was obtained by the phase inversion process and polyaniline, obtained from the polymerization of *p*-phenylenediamine in the presence of hydroquinone, was filtered through this membrane. The material was characterized by thermal analysis, Scanning Electron Microscopy and FT-IR spectroscopy.

**Keywords:** composite membrane, phase inversion process, polysulfone, polyaniline, *p*-phenylenediamine, hydroquinone

## 1. INTRODUCTION

In the past few years, there has been a growing interest for the conducting polymers. They combine the electrical properties similar to those of metals and the characteristics of organic polymers such as light weight, resistance to corrosion, flexibility, lower cost and on the other hand the advantage that they can be tailor-made to the requirements of the application through modifications in the polymer structure [1]. Conducting polymers are remarkable materials with a potent application in a number of growing new technologies, such as energy storage, optoelectronic devices, and for the fabrication of chemical sensors [2]. Among the conducting polymers, polyaniline (PANI) is the most prospective due to its wide range of conductivity from insulating to metallic regime, unique redox tunability associated with the chain nitrogen, good environmental stability, low cost, easy synthesis route, and promising applications in various fields, such as metallic corrosion protection, electromagnetic interference shielding, electrostatic discharge, gas, chemical and pH sensors, actuators, separation membranes etc. [1, 3].

A relatively new approach is to synthesize polyaniline from aromatic diamines, like *p*-

phenylenediamine, due to the fact that the new polymer exhibit better chemical functionality, mechanical and thermal properties than the classically obtained polyaniline [4].

In spite of all these advantages, conducting polymers, including polyaniline, present a poor mechanical strength which limits their applications in some areas including membranes [5]. A solution to this problem is to introduce polyaniline into composite materials. In the present paper polysulfone is used as a blend material for composite membranes due to its remarkable properties.

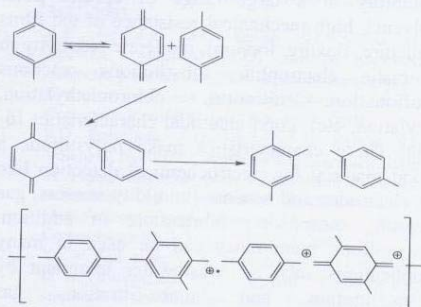
Polysulfone is widely studied as a polymeric material due to its properties such as: high thermal resistance, chemical resistance on entire pH range, resistance in oxidative medium, solubility in a large range of aprotic polar solvents, high mechanical resistance of the films (fracture, flexure, torsion), moderate reactivity in aromatic electrophile substitutions reactions (sulfonation, nitration, chloromethylation, acylation, etc), good electrical characteristics [6-11]. These characteristics make polysulfone a good material for electrochemical detection like in electrodes and sensors (humidity sensors, gas sensors, biosensors) fabrication, in addition, polysulfone membranes can be used in many applications such as: wastewater treatment by ultra-filtration and micro-filtration, gas separation, pervaporation, hemodialysis, fuel cell applications [12-15].

According to the above advantages of the tow materials, this paper reports the synthesis and characterization of a polysulfone-polyaniline composite membrane obtained by the phase inversion process followed by filtration. The new obtained membrane was characterized by thermal

analysis, FT-IR spectroscopy and Scanning Electron Microscopy.

## 2. METHODS

The polysulfone-PSf polymer was supplied by BASF (Ultrason S3010). The polymer was further purified by re-precipitation from chloroform with methanol and dried in vacuum at 60°C. As solvents for polysulfone N, N'-dimethylformamide (DMF), and chloroform (Fluka) were used. As non solvents for membrane phase inversion process, water, methanol and propanol were used. For the synthesis of polyaniline, p-phenylenediamine, hydroquinone, hydrochloric acid (HCl), potassium persulfate (K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) and water were used. The polysulfone solution was prepared by introducing the required amount of solvent (DMF) into an Erlenmeyer glass and adding small portions of polymer, under magnetic stirring, until the desired concentration was achieved (15%). The air was then removed from solution into a desiccator under vacuum for 30 minutes. The polysulfone membrane was formed by depositing a small amount of polymer solution onto a spectral glass with a standard thickness of the membrane fixed at 250µm for. The polymer film deposited on glass was immersed into the coagulation bath which contains 200 mL mixture water : propanol 1 : 1.



**Scheme 1.** Schematic representation of the probable reactions and polyaniline structure.

The polyaniline was obtained by dissolving p-phenylenediamine in a solution of water and HCl, then hydroquinone was added to this solution and in order to induce the polymerization of polyaniline, potassium persulfate was added. The

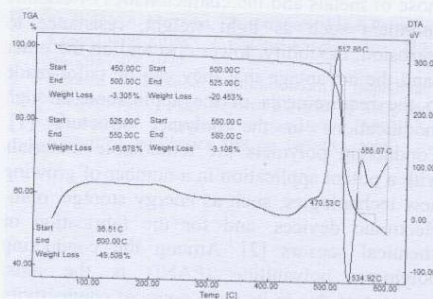
polyaniline presence can be observed due to the fact that a black precipitate is formed.

From the oxidative polymerization of p-phenylenediamine in the presence of small amounts of hydroquinone results a polymer with a doped polyaniline-like structure with semi-quinoid and quinoid rings [4] and probably with hydroquinone bridges intercalated, as it can be seen in scheme 1.

The polyaniline is introduced into the polysulfone membrane by filtering the polymer solution obtained above through the membrane.

## 3. RESULTS

From the thermal analysis of the obtained composite membrane, presented in figure 1, it can be observed a maximum loss point at 512,8°C specific for polysulfone and a maximum loss point at 555,07°C specific for polyaniline. The shape of the thermo-gravimetric graph indicate that no chemical interaction appear between those two polymers. The advantage of using polysulfone as a matrix for polyaniline is given by the highly thermal and chemical resistance. Conductive polyaniline based devices like sensors and microcapacitors can be manufactured for harsh work conditions (oxidative media, high temperature).



**Fig. 1.** Thermal analysis diagram of obtained composite membrane.

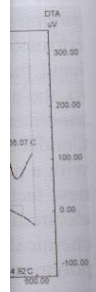
The SEM analysis images, presented in figure 2, reveal a homogenous material with polyaniline fibres intercalated with polysulfone macromolecular fibres (fig. 2. a, b, c). An interesting morphology presents the membrane in cross-section (fig. 2. d), unusual for a membrane obtained by phase inversion (not very symmetrically).

**Fig. 2**

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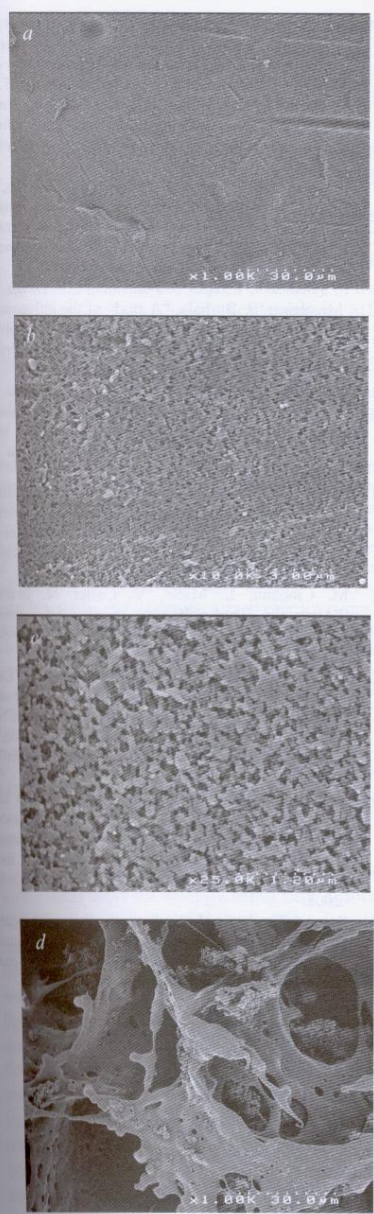


Fig. 2. SEM images of obtained composite membrane, top surface (a-x1000, b-x10000, c-x25000) and cross section (d-x1000).

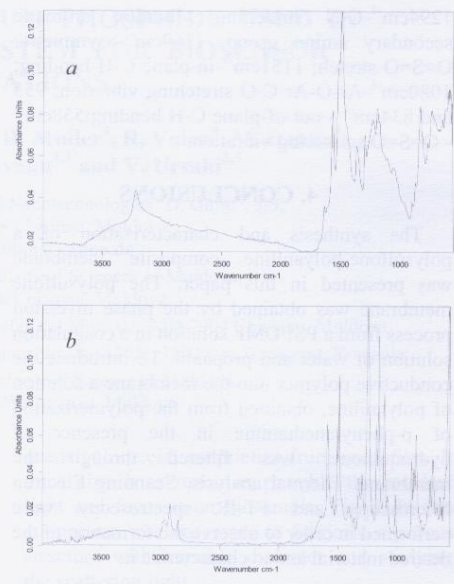


Fig. 3. FT-IR spectrum of polyaniline (a) and polysulfone-polyaniline composite membrane (b).

In the FT-IR spectrum presented in figure 3 the specific peaks and bands for polyaniline and polysulfone-polyaniline membrane can be observed. The assigned vibrational signals for polyaniline obtained by the polymerization of p-phenylenediamine in the presence of hydroquinone (fig. 3. a) are as follows: 3215cm<sup>-1</sup> (3000-3250cm<sup>-1</sup>)-O-H stretching vibration attributed to the O-H group from hydroquinone; 1560cm<sup>-1</sup>-asymmetric C<sub>6</sub> ring stretching attributed to the quinoid ring; 1487cm<sup>-1</sup>-asymmetric C<sub>6</sub> ring stretching attributed to the benzoid ring; 1358cm<sup>-1</sup>-O-H deformation C-O stretching vibration interaction; 1289cm<sup>-1</sup>-C-N stretching vibration aromatic secondary amino group; 824cm<sup>-1</sup>-C-H aromatic deformation. The assigned vibrational signals for the composite membrane (fig. 3. b) are as follows: the band from 3000 to 3250cm<sup>-1</sup>-O-H stretching vibration attributed to the O-H group from hydroquinone; 2969cm<sup>-1</sup>-C-H asymmetric stretch for CH<sub>3</sub> group; 2850cm<sup>-1</sup>-C-H symmetric stretch for CH<sub>3</sub> group; 1585cm<sup>-1</sup>-asymmetric C<sub>6</sub> ring stretching (quinoid ring); 1504cm<sup>-1</sup>-asymmetric C<sub>6</sub> ring stretching (benzoid ring); 1323cm<sup>-1</sup>-symmetric O=S=O stretching vibration from sulfone group;

1294 $\text{cm}^{-1}$ -C-N stretching vibration aromatic secondary amino group; 1169 $\text{cm}^{-1}$ -asymmetric O=S=O stretch; 1151 $\text{cm}^{-1}$ -in-plane C-H bending; 1080 $\text{cm}^{-1}$ -Ar-O-Ar C-O stretching vibration; 955 and 834 $\text{cm}^{-1}$  - out-of-plane C-H bending; 558 $\text{cm}^{-1}$  - O=S=O scissoring vibration.

#### 4. CONCLUSIONS

The synthesis and characterization of a polysulfone-polyaniline composite membrane was presented in this paper. The polysulfone membrane was obtained by the phase inversion process from a PSf/DMF solution in a coagulation solution of water and propanol. To introduce the conductive polymer into the membrane a solution of polyaniline, obtained from the polymerization of p-phenylenediamine in the presence of hydroquinone, was filtered through the membrane. Thermal analysis, Scanning Electron Microscopy and FT-IR spectroscopy were performed in order to observe the formation of the desired material and to characterize it.

**Acknowledgement**—The work has been funded by the Sectoral Operational Programme Human Resources Development 2007-2013 of the Romanian Ministry of Labour, Family and Social Protection through the Financial Agreement POSDRU/88/1.5/S/60203.

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