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Preparation of imide-siloxane/silica nanocomposite membrane

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Preparation of imide-siloxane/silica nanocomposite membrane

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Abstract
In this work, supported polyimide-siloxane hybrid membranes containing different percentages of silica were successfully prepared from pyromellitic dianhydride (PMDA), aminopropyltrimethoxysilane (APrTMOS) and tetramethoxysilane (TMOS) by polycondensation, imidisation and sol-gel reaction. Membranes were coated on supports by spin coating method and the film thickness was 1-3 μm. The molecular structure and thermal performance of these membranes were characterized by using Fourier transform-infrared spectrometry (FT-IR) and thermogravimetric analysis (TGA). Scanning electron microscopy (SEM) was used to study the quality of coatings. The results indicated the presence of imide and Si-O-Si bonds in the sample. Weight loss of the hybrid polyimide membrane was reduced 29% compared to the pure polyimide. No defects or cracks were observed on the substrate/coating interface.

Keywords: Polyimide-Silica-Nanocomposite-Membrane-spin coating

Introduction
In the last two decades significant improvements in the performance of polymeric membranes for gas separation have been made. Polymer-inorganic nanocomposite materials have been investigated for gas separation, and have the potential to provide a solution to the trade-off problem of polymeric membranes. The nanocomposite materials may combine the advantages of each material: for instance, the flexibility and processability of polymers, and the selectivity and thermal stability of the inorganic fillers [1].Regarding gas separation properties of polyimides, their low permeability coefficients, high selectivities, good thin film formability and heat stability make them attractive for this application. In other respects, siloxane polymers are known to have high permeability coefficients, but low selectivities and poor film formability [4].Park and co-workers studied the gas transportation properties of imide-siloxane block copolymer/silica hybrid membranes[2]. Zhong and co-workers fabricated two types of supported polyimide–silica hybrid membranes were prepared from polyamic acid and different silicon sources by sol–gel technique [3]. Homogeneous nanocomposite membranes of polyimide–siloxane copolymers containing different silica contents were prepared by Smaihi et al. [4].

The purpose of this study was to examine the properties of polyimide-siloxane/silica hybrid membranes prepared by polycondensation, imidisation and sol-gel reaction of PMDA, APrTMOS and TMOS. The method used is similar to the method applied by Smaihi et. al. [4]. The difference is in that the spin coating method has been used instead of casting. The molecular structure, thermal performance and coating quality of these hybrid membranes were characterized by methods of FT-IR, TGA and SEM.

Experimental
At first polyamic acid solution was prepared by condensation of 5 mmol PMDA with 10 mmol of aminoalkoxysilane in 12.5 ml of N,N-dimethylacetamide (DMAC). Then, the desired amount of TMOS and water in proportion of 4 mol of water for 1 mol of amic acid were added to solution. The resulting mixture was stirred for few hours. The membrane prepared by spin coating of the solution on α-alumina substrate (An excess amount of solution is placed on the substrate, which is then rotated at certain speed and time) and heating at 40ºC for 12h, 60ºC for 6h, 100ºC for 2h, 230ºC for 10min and 300ºC for 3h.

Results and discussion
The infrared spectra of polyimide-siloxane are presented in fig.1. The sample presents the imide characteristic peaks at 720cm^{-1}, 1380cm^{-1} and 1779cm^{-1}. Characteristics of Si-O-Si bonds are present at 1000-1100cm^{-1} in sample spectra. The absorption in the range of 3200-3700 cm^{-1} is characterized by OH bonds. The results show clearly the formation of desired bonds in sample.

Figs. 2 and 3 show TGA thermograms of polyimide [4] and polyimide-siloxane made in this study,

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respectively. Comparing these graphs shows that weight loss of hybrid polyimide membrane is reduced 29% compared to pure polyimide.
The SEM images of polyimide-siloxane cross section are shown in fig. 4. The coating thickness is 1-3µm. The film is homogeneous and free of defects, indicating that spin coating is a suitable method for preparation of this membrane.

Conclusion

In this work, supported polyimide-siloxane hybrid membranes containing different percents of silica were prepared. The infrared result showed the formation of desired bonds in the sample. Thermogravimetric analysis showed that the weight loss of hybrid polyimide membrane was reduced compared to pure polyimide. SEM results showed good quality of coating.

References

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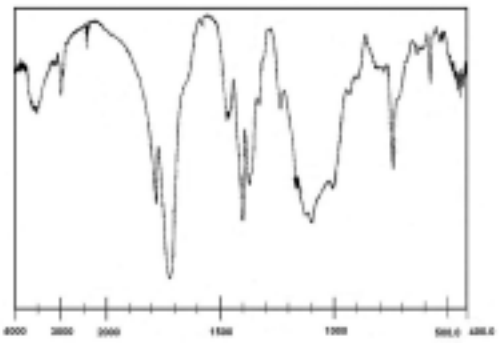

Fig. 1: Infrared spectra of polyimide-siloxane

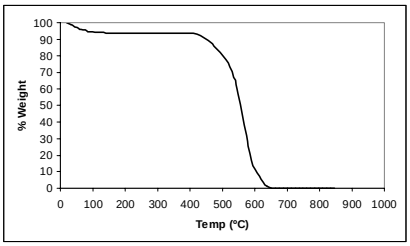

Fig. 2: Thermogravimetric analysis of polyimide

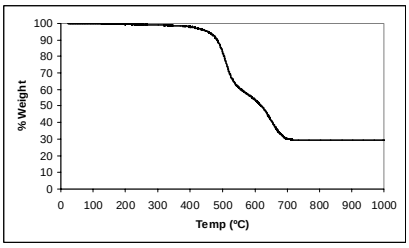

Fig. 3: Thermogravimetric analysis of polyimide-siloxane

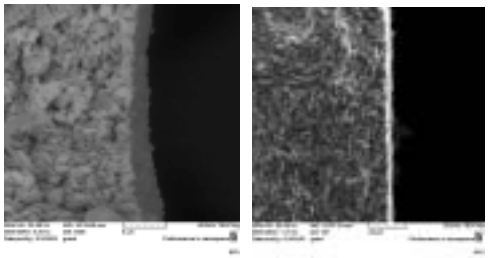

Fig. 4: SEM images of polyimide-siloxane