



Magnetic properties of the magnetic hybrid membranes based on various polymer matrices and inorganic fillers



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ABSTRACT

Magnetic hybrid membranes based on ethylcellulose (EC), poly(2,6-dimethyl-1,4-phenylene oxide) (PPO) and various magnetic praseodymium and neodymium powder microparticles as fillers were obtained. Permeability, diffusion and sorption coefficients of O₂, N₂ and synthetic air components were estimated for homogeneous and heterogeneous membranes using the Time Lag method based on constant pressure permeation technique. The microstructure studies and the phase analysis of magnetic membranes were also performed using SEM and XRD. The influence of magnetic parameters, like coercivity, remanence and saturation magnetization of created membranes on the gas transport properties was studied. The results showed that their coercivity depended on composition and microstructure of the magnetic powder. On the other hand, remanence and saturation magnetization increased with the increase of the powder addition in the membrane. It was found that the magnetic membrane's gas transport properties were improved with the increase of membrane's remanence, saturation magnetization and magnetic particle filling. The decrease in powder particle size and associated increase of the membrane's coercivity also positively influenced the gas transport and separation properties of investigated membranes. It was observed that the magnetic ethylcellulose and poly(2,6-dimethyl-1,4-phenylene oxide) membranes had higher gas permeability, while their permselectivity and solubility coefficient values were rather maintained or slightly increased. The results also showed that the magnetic powder content enhanced significantly gas diffusivity in EC and PPO membranes. It was also analyzed the dependence of the drift coefficient *w* on the magnetic parameters of investigated membranes. The correlation between the membrane selectivity, permeability and magnetic properties with their XRD characteristics was stated.

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1. Introduction

Polymer composites are defined as polymers in which some amounts of micro- or nanometer size fillers are homogeneously dispersed. The main idea of such composites is to integrate several component materials and their properties in a single material. These composites represent the very attractive class of materials, which have become a center of many research projects throughout the world. Their physical and chemical properties show sensitivity to changes and have the ability to adapt to the environmental parameters like temperature, pressure, magnetic field, optical wavelength, etc. Composites with polymer matrix are materials of great interest for researchers because their properties can be adjusted by

controlling the composition, content and morphology of the particle addition, different processing techniques or by modification of the polymer matrix. Material and type of filler (particles, short or elongated fibers) as well as of their amount in the composite have a significant influence on the physical and mechanical properties (thermal stability, optical and thermal properties as well as mechanical properties) [1,2].

Over the last decade, many researchers focused on a new type of hybrid material based on polymers–matrix composites containing dielectric or/and magnetic fillers. There are polymers with extremely good thermal and chemical stability, an easy tenability of their electronic properties and high levels of electromagnetic shielding performances at microwave frequencies with a low mass by unit of surface. The electromagnetic properties of polymers can be modified by the addition of inorganic fillers. The inclusion of magnetic particles may improve the magnetic and dielectric properties of host materials. Therefore, polymers combined with

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magnetic particles provides materials exhibiting novel functionalities [3].

Combination of polymers with other organic or inorganic materials provides new materials with tailored properties, suitable for various electrical and electromagnetic applications in aeronautic or aerospace areas, various sensors, microelectronics, photocatalysis, magnetic devices, batteries, electrochemical display devices, electrical-magnetic shields and microwave absorption materials, coatings, biomedical, and powder metallurgy [2,4].

Polymer composites have the benefit of easy process and preparation, several species of polymer with low price make the composite materials to be used extensively [5]. There have been used many polymer composites with enhanced electrical and magnetic properties, like, for instance: non-conducting and conducting polymers PANI with Fe₃O₄, nano-colloidal iron oxides, γ-Fe₂O₃ and Fe₃O₄, NiZn ferrite, BaFe₁₂O₁₉, hollow Fe₃O₄-PANI spheres, PANI nanotubes containing Fe₃O₄ nanoparticles, the SiO₂/Nd-Fe-B magnet, ferrosinels of the type MFe₂O₄ (M is a divalent metal cation), Fe₃O₄/rubber, MnFe₂O₄/polyaniline, NiFe₂O₄/polystyrene and hexaferrite/polymer [3,4,6–10]. Authors focused not only on ferrites, but they extended their research to other materials such as titanium dioxide, zinc oxide, manganese oxide, vanadium pentaoxide, clinoptilolite, carbon nanotubes and silicon PANI/Co_{1-x}Mg_xFe₂O₄ nano-composites (x = 0, 0.5, 1) [11]. It could be also used magnetic core-shell nanocomposites using Zn_{0.5}Co_{0.5}Al_{0.5}Fe_{1.46}La_{0.04}O₄ as a core being surfactant with four different polymeric shells (PVP, PVA, PVAc and PEG) [12].

Because nowadays the interest in polymer composites is still growing, they have found also the application in membrane separation techniques. They have been implemented as so called mixed matrix membranes (MMMs). This is a new class of membrane materials, that offers the significant potential in membrane separation technology. The successful application of MMMs depends mainly on the polymer matrix selection, the inorganic filler as well as the interaction between these two phases. These membranes combine the selectivity of fillers and the simplicity of polymer membrane processing [13,14]. It was observed that dispersion of impermeable particles in polymers could improve the gas separation properties of membranes [15–19].

Over the last few years, our research had concentrated on magnetic hybrid membranes used for the air enrichment in oxygen. We based on the difference between magnetic properties of paramagnetic oxygen and diamagnetic nitrogen. That has provided the differentiated conditions (other than sorption-diffusion) in mass transport of the separated gases and gives the opportunity for their separation.

This paper is the continuation of our earlier work [20–22], where we have found that incorporation of magnetic micropowders (with granulation 20–32 μm and 32–50 μm) into the polymer matrix (EC, PPO) improved the gas transport properties of membranes. In this work, we are going to exam the magnetic parameters (coercivity, remanence and saturation magnetization) of various types of hybrid membranes (EC and PPO with dispersed magnetic powders, like commercial MQP-14-12, MQP-16-7 and MQP-B with various particle sizes).

2. Theoretical

2.1. The evaluation of gas transport coefficients

We have collected experimental permeation data for the oxygen and nitrogen, both for individual, pure gases, as well as components of air. We have analyzed the system, i.e. membrane and permeating gas using appropriate description [20–22], supported by the percentage of the air enrichment.

During the gas permeation experiments was measured flow rate Q , which in standard conditions is defined as [20].

$$Q_{STP} = Q \frac{T_{STP} p}{T p_{STP}} \quad (1)$$

where:

Q – measured flow rate [cm³/s],
 p – atmospheric pressure [Pa],
 T – temperature [K],
 p_{STP} – standard pressure, $p_{STP} = 1.013 \cdot 10^5$ Pa,
 T_{STP} – standard temperature, $T_{STP} = 293.15$ K.

To avoid the necessity of taking into account the impact of possible changes of temperature and pressure on the measurement results, they were converted to standard conditions.

This flow rate Q_{STP} was then recalculated into diffusive mass flux in a stationary state J_s :

$$J_s = \frac{Q_{STP}}{A} \quad (2)$$

where:

Q_{STP} – flow rate at standard condition $\left[\frac{\text{cm}^3}{\text{s}}\right]$,
 A – membrane active area [cm²].

Using this J_s , permeation coefficient P was evaluated according to the given equation [20,22]:

$$P = \frac{J_s l}{\Delta p} \quad (3)$$

where:

P – permeation coefficient [Barrer], $\text{Barrer} = \frac{\text{cm}^3_{STP} \cdot \text{cm}}{\text{cm}^2 \cdot \text{s} \cdot \text{cmHg}} \cdot 10^{-10}$,
 l – membrane thickness [cm],
 Δp – gas pressure difference at both sides of the membrane [cmHg],
 J_s – diffusive mass flux in a stationary state $\left[\frac{\text{cm}^3_{STP}}{\text{cm}^2 \cdot \text{s}}\right]$.

An average diffusion coefficient \bar{D} was calculated from the stationary state of permeation according to the formula:

$$\bar{D} = \frac{J_s \cdot l}{\Delta c} \quad (4)$$

where

J_s – diffusive mass flux in stationary state (cm³_{STP}/cm²·s),
 l – thickness of membrane (cm),
 Δc – concentration difference.

After integration of J_s with respect to time, we have got a downstream absorption permeation $Q^a(l, t)$ (total flow of penetrant). Concentration difference Δc and Time Lag L^a were obtained using the Time Lag method. The first one was obtained from an intercept of the asymptote to the stationary permeation curve with the $Q^a(l, t)$ axis. While the second one L^a was determined as an intercept of the asymptote to the stationary permeation curve with the time axis [20–22].

Comparing \bar{D} with the value of diffusion coefficient D_l calculated from the downstream absorption Time Lag L^a [20–22] according to the following equation:

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