

DRAGUTIN M. NEDELJKOVIĆ¹
MARIJA P. STEVANOVIĆ²
MIRKO Z. STIJEPOVIĆ³
ALEKSANDAR P. STAJČIĆ¹
ALEKSANDAR S. GRUJIĆ¹
JASNA T. STAJIĆ-TROŠIĆ¹
JASMINA S. STEVANOVIĆ¹

¹University of Belgrade, Institute of Chemistry, Technology and Metallurgy, Belgrade, Serbia

²TENT doo TE "Morava", Svilajnac, Serbia

³Department of Chemical Engineering, Texas A&M University at Qatar, Education City, Doha, Qatar

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THE POSSIBILITY OF APPLICATION OF ZEOLYTE POWDERS FOR THE CONSTRUCTION OF MEMBRANES FOR CARBON DIOXIDE SEPARATION

Article Highlights

- Carbon dioxide separation from the waste gases
- Application of mixed matrix membranes for gas separation
- Membranes based on polymer matrix and inorganic zeolyte powder
- Polymers with PEO groups and zeolites with two-dimensional pores were tested

Abstract

The aim of this study was to construct a polymer-based mixed matrix membrane that could be used for waste gases treatment. Therefore, high permeability for the carbon dioxide and low permeability for other gases commonly present in the industrial combustion waste gases (nitrogen, oxygen, hydrogen and methane) are essential. These membranes belong to the group of dense composite membranes, whose separation is based on the solution-diffusion mechanism. In this paper, feasibility of the application of poly(ethylene oxide)-copoly(phtalamide) was tested. In order to enhance the permeability of carbon dioxide, three different zeolites with two-dimensional pores (IHW, NSI and TER) were incorporated, and in order to improve compatibility between the inorganic particles and polymer chains, n-tetradecyldimethylammonium bromide (NTAB) was added. The best results in carbon dioxide/hydrogen selectivity were obtained with the membrane constructed with PEBAX 1657 and surface treated zeolites, while better results concerning selectivity were gained with membranes based on Polyactive.

Keywords: mixed matrix membrane, zeolite, carbon dioxide separation, polymer matrix, membrane selectivity.

Global warming has emerged as one of the most serious problems in chemical and environmental engineering in the recent decades, with carbon dioxide being the main atmospheric pollutant. Carbon dioxide is emitted through various processes that include combustion (mining, power plants, transport, industrial facilities). As coal and petrol, the main sources of both energy and carbon dioxide, have no feasible alternative at the global scale, the main goal of the environmental analysis is to reduce the emission of carbon dioxide. Currently, the most common proce-

dures for CO₂ removal include adsorption and cryogenic processes [1-3]. The United Nations introduced a plan to gradually reduce CO₂ emission in the following years (United Nations Framework Convention on Climate Change (UNFCCC, colloquially known as the Kyoto protocol) [4]. Carbon dioxide membranes based on the solution-diffusion mechanism have high potential for research and development, especially in the small-to-medium scale facilities with moderate requirements concerning the purity of the products [5]. Research in this field of membrane development has rapidly expanded in last three decades, with various polymers developed as the main component of the membrane [6-10]. The suitable polymer should contain units that would enhance the solubility of the carbon dioxide, keeping at the same time low solubility of the other gasses, thus creating high permeation selectivity. Poly(ethylene oxide) (PEO) has

Correspondence: D.M. Nedeljković, Institute of Chemistry, Technology and Metallurgy (ICTM), University of Belgrade, Njegoševa 12, 11000 Belgrade, Serbia.
E-mail: dragutin@tmf.bg.ac.rs
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been proven to be potentially suitable for this application [11]. The negative property of pure PEO is its strong tendency to crystallize, which has a negative effect on the overall permeability of the membrane [12]. Instead of pure PEO, polymers that contain EO units can be used for this purpose. A polymer that is commercially available by Arkema (formerly Atotech) under the name PEBAX has the structure of poly-(amide-b-ether) and can be used as a good alternative material for this purpose [13]. PEBAX belongs to the group of thermoplastic elastomers (Figure 1a). As a second choice, the polymer under commercial name Polyactive (supplied by IsoTis OrthoBiologics) was tested (Figure 1b).

PA stands for the polyamide hard block, and usually is nylon-6 or nylon-12, while the PE stands for the soft, amorphous polyether block (polyethylene oxide (PEO) or polytetramethylene oxide (PTMO)) [14].

As it can be seen from Figure 1b, Polyactive consists of polyethylene glycol (PEG) and polybutylene terephthalate (PBT). The ratio PEG:PBT is 77:23 (wt.%) with PEG of molecular weight of 1500 g/mol.

The properties of the polymers (chemical, physical and mechanical) can be easily modelled by the variation of the molar ratio of the blocks [15]. According to the previously reported research, both Pebax and Polyactive turned out to be the promising materials for acid gas treatment [16-19]. These polymers, when applied for the construction of the membrane, have also shown high selectivity of carbon dioxide *versus* both nitrogen and oxygen. The theoretical explanation of the high selectivity is that ester and ether groups show strong affinity to the carbon dioxide solution. The other reason for high selectivity versus nitrogen is the polarizability of the carbon dioxide (as well as the sulphur dioxide) in the presence of PEO

segments [17]. The possible solution for the increase of permeability and selectivity is the construction of a mixed matrix membrane that consists of a polymer matrix and inorganic powder. The matrix is usually made of the polymer that contains PE blocks, and the dispersed phase is inorganic particles [18-19]. The dispersed particles can be zeolites, carbon molecular sieves or other nanoparticles. The presence of these fillers improves selectivity and permeability comparing to the membranes made of the pure polymer due to their inherent separation characteristics. However, addition of the charged inorganic particles in the polymer matrix can cause problems with dispersion, agglomeration and uneven distribution of the particles. Fragility, as one of the main problems of the inorganic membrane is avoided due to the flexibility and elasticity of the polymer.

The first attempts of the mixed matrix membranes permeability improvement were reported 30 years ago, when the diffusion time lag of the carbon dioxide and methane was discovered [20]. Authors have observed that addition of the zeolite increases the time lag, but has apparently no effect on the steady-state permeation [21].

EXPERIMENTAL

The Pebax and Polyactive polymers were supplied by Arkema and IsoTis OrthoBiologics, respectively. The polymers were supplied in the form of powder and used as received. Three different zeolites with two-dimensional pores were used in this experiment. Their properties are compiled in Table 1. The average specific surface of the zeolite was 500 m²/g.

Solvents (ethanol and chloroform), zeolite and *n*-tetradecyltrimethylammonium bromide (NTAB) were

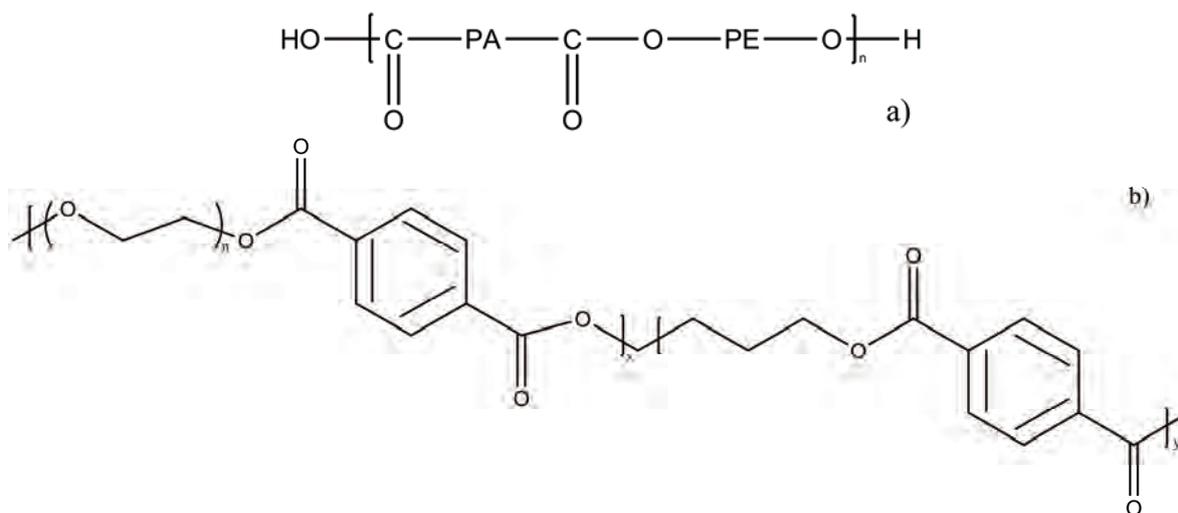


Figure 1. Structural formula of a) Pebax co-polymer; b) Polyactive co-polymer.

supplied by ABCR. All of the chemicals and used as received. The aim of the NTAB addition was to provide good contact between electrically charged zeolyte particles, and highly hydrophobic polymer matrix. The electrically charged end and long, normal hydrocarbon chain should interact with the aforementioned components of the composite membrane, respectively.

Table 1. Properties of different types of zeolite used for the construction of the membrane; 2D channel system dimension

Framework type code	Pore size, nm
IHW	0.37 X 0.35
NSI	0.26 X 0.24
TER	0.52 X 0.47

The first step in the membrane preparation procedure was the solution of the polymer in the suitable solvent. For the Pebax membranes, the solvent was mixture ethanol/distilled water (70/30 mass ratio). The solvent for the Polyactive membranes was chloroform. The Pebax was dissolved at the 80 °C under reflux, while Polyactive was dissolved at the room temperature, both of them for two hours. The zeolyte powder was at the same time dissolved in the same solvent as the polymer, and the additive was added (if the samples were made with additive). The homogenization of the zeolyte solution was done by ultrasound mixing with a titanium head. The duration of ultrasound mixing was five minutes in order to avoid contamination of the solution by titanium nanoparticles detached from the head. The zeolyte solution and polymer solution were mixed and stirred overnight at the same temperature as the respective polymers. Overnight stirring was necessary in order to eliminate possible clusters and agglomerates of the zeolyte powder formed in the solution. This procedure resulted in viscous solution that was casted to the Teflon surface, covered with non-woven textile in order to protect sample from dust and any other unwanted particles and left overnight at room temperature and ambient pressure to dry. Teflon was used in order to avoid stitching of the membrane to the drying surface. If the drying process was too fast, it would result in the formation of bubbles and thus, bad permeation properties of the membrane. The viscosity of the solution had to be kept at an optimal value, which was determined empirically. If the viscosity is too low, the sedimentation velocity is too high, and the resulting membrane will have an uneven distribution of the particles through the volume. On the other hand, if the viscosity is too high, casting and drying processes are dominated by the surface tension, resulting in a mem-

brane with uneven thickness. After drying at room temperature, the material was placed on a high vacuum line in order to remove any traces of the residual solvent.

The gas permeability measurements were carried out by the time lag method. The, diffusivity (D), permeability (P) and selectivity (α) were determined by the equations [22-24]:

$$P = DS = \frac{V_p I (p_{p2} - p_{p1})}{ART \Delta t (p_f - (P_{p2} + p_{p1}) / 2)} \quad (1)$$

$$d = \frac{l^2}{6\theta} \quad (2)$$

$$\alpha_{A/B} = \frac{P_A}{P_B} = \frac{D_A S_A}{D_B S_B} \quad (3)$$

where V_p stands for the constant permeate volume, l for the thickness of the membrane, A for the area of the membrane, R for the universal gas constant, Δt for the time that permeate pressure needs to increase from value p_{p1} to value p_{p2} , p_f for the feed pressure and D for the diffusion coefficient. In Eq. (2), d stands for the intercept on the time axis when pressure on the permeate side is presented versus time. This curve has a parabolic shape which turns into a straight line once the steady state is obtained. Extrapolation of the steady-state line to the x -axis gives the parameter d . The solubility can be calculated as the ratio between permeability and diffusivity. The solution-diffusion model was used for the analysis of the gas transport properties of the membranes [25]. The selectivity of the membrane for the gas A versus gas B was defined as the ratio of their permeabilities.

Before the permeability measurements, the membrane was kept at high vacuum for 30 min in order to remove any traces of humidity that could have penetrated at the ambient conditions. After the drying, the gas that was measured was applied at one side of the membrane. The other side of the membrane was evacuated, causing the pressure gradient as the driving force for the diffusion and gas permeation. The pressure as the function of time was measured at the low pressure side of the membrane, and the permeation properties were calculated by equations (1)-(3). Due to security measures in order to avoid mixing of the flammable gases, the order of gases for the measurement was: hydrogen, nitrogen, oxygen, carbon dioxide. Between measurements of different gases, the membrane and the whole equipment was kept under high vacuum for 15 min [26].

RESULTS AND DISCUSSION

Four different series of the membranes were constructed, two with each polymer. Two series were made of pure polymer and zeolyte, without the addition of the filler. In the second series, NTAB was added to the polymer and zeolyte. The weight ratios were calculated as the mass ratio *versus* overall mass of the membrane. The compositions of the membranes are given in Table 2.

Table 2. The composition and appearance of the membranes made without the additive

Series	Polymer	Additive
I	Pebax	-
II	Pebax	NTAB
III	Polyactive	-
IV	Polyactive	NTAB

Initially, the appearance of the membranes was assessed visually. If the membrane is made properly, it should be smooth, transparent or pale, flat without pinholes or visible damages. If the membrane is not transparent, that indicates that the light transmitted at the polymer-zeolyte surface and the contact between them is bad. Rough surface indicates uneven distribution of the particles through the volume of the membrane, while the self-rolling of the membrane indicates the sedimentation of the particles, and thus uneven distribution of the zeolyte.

The composition and evaluation of the membranes from the series I and series II are given in Table 3.

Table 3. The composition and appearance of the membranes of Series I and II

Membrane No.	Zeolyte filler	Filler, %	Additive, %	Appearance
I-1	IHW	22	-	White
I-2	IHW	22	-	White
I-3	NSI	22	-	Transparent
I-4	NSI	22	-	White
I-5	TER	22	-	White areas
I-6	TER	22	-	Transparent
II-1	-	-	3.3	Transparent
II-2	IHW	22	3.3	White
II-3	IHW	22	3.3	White
II-4	NSI	22	3.3	Transparent
II-5	NSI	22	3.3	Transparent
II-6	TER	22.5	2.2	Transparent
II-7	TER	23	1.1	Transparent

As it is obvious from Table 3, both NSI and TER types of zeolyte could be used for the construction of the membrane. Although transparent membranes were not obtained in all of the samples, their construction with NTAB was attempted. The explanation of the white spots in the membranes constructed with IHW can be that the zeolyte particles agglomerate. This agglomeration comes as the consequence of strong electrostatic forces between the zeolyte particles, which are stronger than the viscosity of the polymer solution. Areas of the different colour indicate a non-stationary drying process that causes rapid local variations in viscosity of the solution, and therefore, the agglomeration was allowed in some areas of the membrane. To check the possible agglomeration and distribution of the zeolytes in the polymer, an SEM image of the sample I-6 was obtained (Figure 2). In general, good distribution of the zeolyte particles is present in this system. Agglomeration is still visible as the white cluster in the upper right part of the figure.

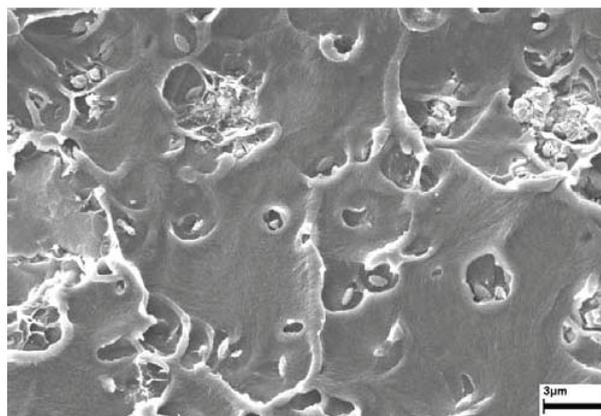


Figure 2. SEM image of the sample I-6.

The compatibility of the polymer and filler was tested by the construction of the membrane that was solely made of polymer and additive. A transparent membrane was obtained, and therefore this filler was taken as the compatibilization additive.

Analyzing the appearance of the membranes from the series II, it is obvious (Table 3) that both NSI and TER have shown good compatibility with Pebax in the presence of NTAB as the additive. Therefore, these two zeolytes were used for the construction and measurement of the permeation properties of gases. The IHW zeolyte could not be used for this purpose due to the bad polymer-zeolyte contact (sample II-2) and agglomeration of the zeolyte particles (sample II-3). An SEM image was obtained (Figure 3) in order to check the microstructure of the membrane. Comparison of the results for the series I and II shows significant improvement in the zeolyte-polymer contact.

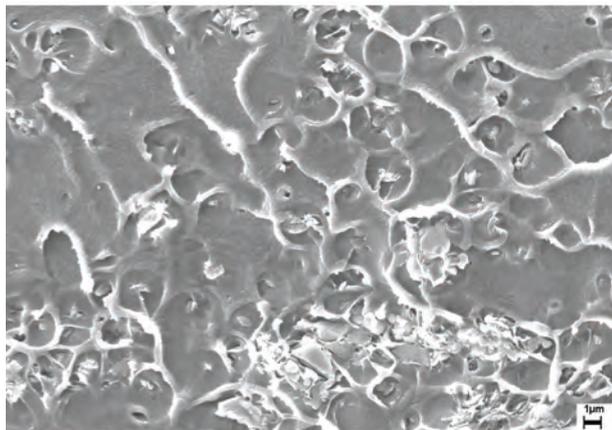


Figure 3. SEM image of the sample II-6.

Comparing Figures 2 and 3, it can be observed that the latter shows improved distribution of the zeolyte. Both of the samples show good particle distribution, but in the case of sample II-6, agglomeration is nearly avoided. Only partial formation of the cluster is visible (bottom part of the Figure 3 in the middle). However, it can be seen that the polymer is present between the particles of the cluster.

The membranes based on the Polyactive based polymer (series III and IV) were constructed in the manner analogous to the Pebax based membranes (series I and II). The main difference in the procedure is that chloroform was used rather than ethanol/water mixture for Polyactive and Pebax polymers, respectively. Due to the low boiling point of the chloroform, the removal of residual solvent from the membrane was easier. Tetrahydrofuran (THF) can be used for this purpose as an alternative. The amounts of polymers, zeolytes and additives were analogous as for the membranes of the series I and II. The data of

composition and appearance of the membranes are presented in Table 4.

The sample II-1 was constructed solely from the Polyactive polymer. The purpose of this sample was to compare its properties with the properties specified by the supplier. The measured results slightly differ from the specification. This difference can be attributed to the eventual error in the measurement, or to the variations in the different polymer batches. Similarly to the Pebax based membranes, the IHW filler is not compatible in the series without additive (samples III-2 and III-3) and with additive (samples IV-2 and IV-3). As in the case of the Pebax based membranes, both NSI and TER have been proved as good fillers, resulting in smooth, transparent membranes without visible pinholes or other damage. However, comparing the appearance of the membranes based on Pebax and Polyactive containing NSI and TER, it might be seen that results obtained with the Polyactive are not as good as the results obtained with Pebax. Although the IHW cannot be used for the construction of the membrane with any of tested polymers, different behaviour with two different polymers was observed. While the samples with IHW from series I and II (prepared with Pebax) are white, the samples from series III and IV contain white spots. This behaviour indicates different types of the behaviour of the zeolyte particles in the presence of different polymers, and thus, difference in the structure of the samples made with the same powder, but different polymer. The samples with white spots show the agglomeration of zeolyte particles, and white membrane or white areas on the membrane show bad contact between the zeolyte particles and polymer chains.

Table 4. The composition and appearance of the membranes of Series III and Series IV

Membrane number	Porous filler	Filler, %	Additive, %	Appearance
III-1	-	-	-	Transparent
III-2	IHW	22	-	White spots
III-3	IHW	22	-	White spots
III-4	NSI	22	-	Transparent
III-5	NSI	22	-	White areas
III-6	TER	22	-	Transparent
III-7	TER	22	-	White spots
IV-1	-	-	3.3	Transparent
IV-2	IHW	22	3.3	White spots
IV-3	IHW	22	3.3	White areas
IV-4	NSI	22	3.3	White spots
IV-5	NSI	22	3.3	Transparent
IV-6	TER	22	3.3	Transparent
IV-7	TER	22	3.3	White

The sample IV-1 was made, in the analogue manner as the sample II-1, in order to check the possibility of the application of the NTAB in the Polyactive matrix. As the experiment resulted in a transparent membrane without visible spots, it was concluded that NTAB could be used as an additive for the membranes made with Polyactive. Similarly to the Pebax membranes, both NSI and TER have shown good results, and again, better results were gained in the presence of NTAB. The amount of filler was determined as the optimal value of 22 wt.% *versus* overall mass of the membrane. If the concentration of the filler is lower, the permeation properties of the membrane are negatively affected. If the concentration is higher, the agglomeration is enhanced due to the electrostatic forces between the particles of the zeolyte powder. The amount of the additive is determined in the similar manner. If the concentration of the additive is too low, not all of the particles of the zeolyte could be covered. On the other hand, if the concentration of the additive is too high, the zeolyte particles precipitate, and that precipitation could be attributed to the electrostatic forces. The SEM image

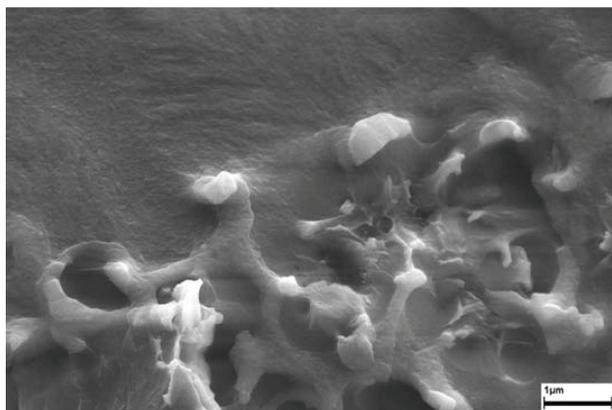


Figure 4. SEM image of the sample IV-6.

of sample IV-6 is shown in Figure 4. Comparing this sample with the previous, it can be seen that the polymer completely surrounds the zeolyte particles and that no agglomeration is present.

For the permeability measurements, all of the transparent samples without visible damage were used, regardless of whether they were made with or without additive. Prior to the measurement of the membranes, the permeability of samples II-1 and IV-1 was measured in order to compare permeability of the pure polymer and permeability of the polymer with additive. The obtained results clearly indicate that dispersion of the NTAB does not influence the permeability of the pure polymer. The results of permeability and selectivity measurements are given in Table 5.

It should be noted that the usual unit for the gas permeability of the membrane in the membrane research community is a Barrer. One Barrer is the permeability of 1 cm³ of a gas under the standard pressure and temperature conditions, through the 1 cm² of the area and 1 cm of the thickness driven by the pressure gradient of 1 cmHg in 1 s divided by the factor of 10⁻¹⁰.

Analyzing the permeability data presented in Table 5, it is obvious that all of the membranes that appeared transparent showed good and comparable permeability and diffusivity properties. Comparison of the obtained results indicates that permeability for carbon dioxide is slightly better in cases of membranes constructed with Pebax (series I and II), but the selectivity is better in the case of Polyactive (series III and IV). Therefore, the choice of polymer for the construction of the membrane is determined by the requirements concerning the capacity of equipment (higher flux requires higher permeability, and therefore Pebax is the preferred polymer) and purity of the products (higher requirements for purity need

Table 5. The results of the permeability measurement of the membranes

Membrane No.	Thickness, μm	P (CO ₂), Barrer	α (CO ₂ /H ₂)	α (CO ₂ /O ₂)	α (CO ₂ /N ₂)
I-3	187	70	8.1	20	52
I-6	152	110	8.7	19	48
II-3	134	112	8.4	17	55
II-4	205	120	9.1	18.6	60
II-5	268	95	8.8	19	59
II-6	254	105	9.6	21	57
III-4	189	92	8.2	19.5	61
III-6	165	89	8.5	21	63
III-7	176	97	9.9	20	56
IV-5	171	91	10.8	21.5	60.4
IV-6	231	99	9.9	19.7	58.4

higher selectivity, and Polyactive is in that case the better choice).

CONCLUSIONS

In this paper, the possibility of the construction of a mixed matrix membrane based on a polymer matrix and surface treated inorganic powder was examined. Two different types of polymers, three different types of two-dimensional zeolites and one additive were used for the construction of the membrane. Preliminary optical testing showed that not all of the combinations are suitable for the construction of the membrane. In the case of Pebax-based membranes, NSI and TER have shown good compatibility between the inorganic powder and polymer chains. Good contact and distribution of the particles could not be provided in the case of the IHW zeolyte. The NTAB has improved the compatibility between the inorganic zeolyte particles and polymer chains without affecting the permeability properties of the membrane. Therefore, it is reasonable to conclude that NTAB is a good foundation for future research in the field of gas separation membranes. The main challenge in future research would therefore be the downsizing of the membrane thickness to values lower than 100 μm . The other goal of the research is to test the possibility of application, permeability and selectivity in wet conditions.

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DRAGUTIN M.NEDELJKOVIĆ¹
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 ALEKSANDAR S. GRUJIĆ¹
 JASNA T. STAJIĆ-TROŠIĆ¹
 JASMINA S. STEVANOVIĆ¹

¹Institut za hemiju, tehnologiju i metalurgiju (IHTM) - Centar za mikroelektronske tehnologije, Univerzitet u Beogradu, Njegoševa 12, 11000 Beograd, Srbija

²TENT doo TE "Morava", Svilajnac, Srbija

³Department of Chemical Engineering, Texas A&M University at Qatar, Education City, Doha, Qatar

NAUČNI RAD

MOGUĆNOST PRIMENE ZEOLITNIH PRAHOVA ZA KONSTRUKCIJU MEMBRANA ZA IZDVAJANJE UGLJEN-DIOKSIDA

Cilj ovog rada je bio da se konstruiše neporozna kompozitna membrana bazirana na polimernom matriksu koja može da bude upotrebljena za tretman otpadnih gasova. Za ovu svrhu, neophodno je da membrana ima visoku permeabilnost za ugljen-dioksid i nisku permeabilnost za druge gasove koji ne najčešće sreću u produktima sagorevanja (azot, kiseonik, vodonik, metan). Ove membrane pripadaju grupi neporoznih membrana i mehanizam separacije gasova je baziran na rastvorljivosti i difuziji. U ovom radu, testirana je mogućnost primene poli(etilenoksida)-kopolimeri(ftalamida). Da bi se povećala permeabilnost ugljen-dioksida, dodavana su tri različita zeolitna praha, a da bi se poboljšala kompatibilnost neorganskih čestica i polimernih lanaca, dodat je n-tetradeciltrimetilamonijum-bromid (NTAB). Ispitivani zeoliti pripadaju grupi sa dvodimenzionalnim porama (IHW, NSI i TER). Najbolji rezultati u separaciji ugljen-dioksida i vodonika su postignuti kod membrana baziranih na polimeru PEBAK 1657 i zeolitima uz dodatak aditiva. Sa druge strane, bolja selektivnost je postignuta kod membrana baziranih na Polyactive polimeru.

Ključne reči: neporozne membrane, zeolitni prahovi, separacija ugljen-dioksida, polimerni matriks, selektivnost membrane.