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Preparation of Poly(Ethylene Terephthalate) Track-Etched Membranes for the Separation of Water-Oil Emulsions

Rapid industrial growth in the petrochemical, pharmaceutical, metallurgical and food industries, as well as stormwater that accumulates pollution from the roadway and the territories of motor transport enterprises, gas stations, car washes and other municipal services have led to the formation of a large amount of oily wastewater. Oil-containing wastewater is a multicomponent, multiphase water system and, as a rule, is in a state stabilized by various factors, which greatly complicates their processing. Pollution of water sources with oil-containing compounds leads to negative consequences for both living organisms and human health. Therefore, the need to treat oily wastewater is an urgent problem. In this article, poly(ethylene terephthalate) track-etched membranes (PET TeMs) with pore diameters of ~ 5.1 μm and pore density of 1·10⁶ pore/cm² were modified by formation of polyelectrolyte complexes of PET TeMs surface with poly(allylamine) (PAAm) and tested for oil-water separation by using hexadecane/water (at pH=2) and chloroform/water (at pH=2) emulsions. Fourier-transform infrared (FTIR) spectroscopy, scanning electron microscope (SEM), energy dispersive X-ray (EDX) analysis, contact angle measurements were used for membrane characterization. The efficiency of oil-water separation was evaluated by flux measurements. Results showed separation performance of 267 mL/m²·s for hexadecane/water (pH=2) and 100 mL/m²·s for chloroform/water (pH=2) at vacuum pressure of 700 mbar.

Keywords: track-etched membranes, oily wastewater, poly(allylamine), hexadecane, chloroform, poly(ethylene terephthalate), water treatment, separation.

Introduction

At present, due to the growth of industrial production, environmental pollution is significantly increasing, as a result, there is a deterioration in the condition of objects of domestic and drinking water use [1–5]. The most common pollution of water bodies (oceans, seas, lakes, rivers, groundwater, glaciers) is oil and its products, such as gasoline, kerosene, oils, fuel oil, etc. In water bodies, oil and oil products create various forms of pollution, such as oil film floating on the water, oil products dissolved or emulsified in water, heavy fractions settled to the bottom, products adsorbed by the bottom soil or the shore of a reservoir. The negative consequences of pollution are showed in various mechanisms of exposure and damage to living organisms, including humans [6–8]. Contaminated industrial oily wastewater can also form explosive and flammable gases and mixtures or toxic substances, therefore, various methods are used to treat and neutralize them [9–11].

Such methods as coagulation [12, 13], flocculation [14, 15], flotation [16, 17], sorption [18], electromagnetic separation [19, 20] can be used to solve the problem of wastewater treatment from oil pollution. Currently, membrane separation methods (ultrafiltration, reverse and direct osmosis) are the most promising for the purification of water-oil emulsions, due to their high energy efficiency, selectivity, and economy [21–24]. Various types of membranes can be used, while track-etched membranes (TeMs) are poorly understood. However, the small thickness, non-tortuosity of pores, extremely narrow pore size distribution, and controlled pore geometry per unit area make them promising for use in the process of separating oil-in-water emulsions [25–29]. In addition to the use of TeMs in water filtration, they can be used in microelectronics, bio- and nanotechnologies (for example, as a means of delivering medicines), medicine, pharmaceutical, food and perfume industries, chemical industry, ecology and other fields [29–31]. Polytetrafluoroethylene (PTFE), polyvinylidene fluoride (PVDF), polypropylene (PP) membranes are mainly

used to separate oil-water emulsions, however, the use of polyethylene terephthalate (PET) TeMs has been little studied [32–35].

Since the surface of PET TeMs has semi-hydrophobic properties, it is necessary to increase hydrophilicity for effective separation of water-oil emulsions [36, 37]. Polyallylamine (PAAm) is a hydrophilic monomer that can be used to separate oil-in-water emulsions. In addition, polyallylamine is available and has not been previously studied for surface modification of PET TeMs. For this purpose, the hydrophilization of oxidized PET TeMs by polyallylamine was studied in this article.

Previously, we have shown that PET TeMs modified by photoinitiated graft polymerization of stearyl methacrylate [38], as well as by soaking in a trichlorooctylsilane solution [39], performed well in the separation of oil-water emulsion. In this paper, we studied a simpler and more energy efficient method of modifying PET TeMs to obtain a pH-responsive surface by soaking in a (poly)allylamine (PAAm) solution for use in the separation of chloroform/water (pH=2) and hexadecane/water (pH=2) systems in various ratios.

Experimental

Materials

Sodium hydroxide, benzophenone, N,N-dimethylformamide, 2-propanol, poly(allylamine hydrochloride), methanol, ethanol, chloroform, hexadecane were purchased from Sigma-Aldrich. Deionized water (18.2 M Ω) was used in all experiments.

Preparation of track-etched membranes (TeMs)

The PET TeMs were prepared as described below. Ion tracks on PET films were generated by irradiation with Kr ions using the accelerator DC-60 (Astana branch of Institute of Nuclear Physics) with an energy of 1.75 MeV/nucleon and ion fluences $1 \cdot 10^6$ ion/cm². Then the membranes were processed by photosensitization for 30 min on both sides and were chemically treated in 2.2M NaOH solution at certain periods of time. After chemical etching in a 2.2 M NaOH solution at 85 °C, membranes with a pore diameter of ~5.1 μ m were obtained.

Pre-oxidation of PET track-etched membranes in H₂O₂/UV system

PET TeMs were oxidized in a solution of 0.3 M H₂O₂ at pH=3 (HCl). Oxidation was carried out for 180 min under UV lamps (190W). After the oxidation, samples were washed in deionized water and dried [25].

Preparation of poly(allylamine) solution

0.6 g of polyallylamine hydrochloride was neutralized by reaction with 0.224 g of NaOH in 10 ml of methanol. The reaction kept for 24 h at 60 °C. Then solution was separated from the precipitate and 40 ml of ethanol was added. Then, oxidized PET TeMs were kept in a 6 % poly(allylamine) solution for 6h. After that, before determining the contact angle (CA), the samples were additionally kept in a solution of pH=2 and pH=9 for 30 min.

Methods of characterization

For identification of chemical groups before and after modification, FTIR spectra were recorded using FTIR spectrometer InfraLUM® FT-08 with an ATR accessory. The measurements were carried out in the range from 400 to 4000 cm⁻¹, 32 scans with 2 cm⁻¹ resolution at a temperature of 21–25 °C.

The water and hexadecane contact angles were measured by using Digital Microscope with 1000 \times magnification by the sessile drop method at room temperature. Before measuring the contact angle, the samples were soaked for 30 minutes at a certain value of pH. The average drop volumes of water at different pH was 15 μ l, the average value of the CA was obtained by measuring the sample in a few different positions.

Hitachi TM3030 scanning electron microscope with a Bruker XFlash MIN SVE microanalysis system at an accelerating voltage of 15 kV was used to study the membrane morphology and elemental composition of the surface before and after modification.

The gas permeability test was used to evaluate the effective pore sizes of the membranes at a pressure drop of 20 kPa according to the method described in [40].

A burst strength procedure was performed to evaluate the mechanical properties of oxidized and modified PET TeMs. Burst strength was evaluated at pressure that damages a circular sample of 1 cm² surface area. Burst strength is 0.28 MPa for pristine PET TeMs with pore diameter of ~5.1 μ m, and there is a slight decrease to 0.20 MPa after modification with PAAm.

Performance of modified membranes in oil-water separation

The separation of water-oil mixtures with modified PET TeMs was carried out by filtration according to the scheme presented in our previous works [38, 39].

The performance of the obtained membranes was tested out as follows. First, the modified PET TeMs were kept in a pH=2 solution for 30 minutes. Then, the samples were dried and fixed in a vacuum filtration unit. Chloroform and hexadecane at pH=2 were dispersed in a volume of 20 ml in the different ratios (chloroform/water (pH2) = 1:50 and 1:100 (vol.), hexadecane/water (pH2) = 1:50 and 1:100 (vol.)) using an IKA T18 digital ULTRA-TURRAX disperser at a speed of ~ 22000 rpm. Next, the mixture was poured into a glass funnel, a hose was connected from the IKA VACSTAR Control vacuum pump, setting the desired pressure value. Next, the filtration of the mixture was observed by recording the time at which the liquid passes through the pores of the membrane.

The performance (Q) of the filtered water-oil mixture was calculated by Equation (1):

$$Q = \frac{V}{S \cdot T} \quad (1)$$

where Q — the performance (ml/s·cm²); S — the filtration area of sample (cm²); V — the volume of solution (ml); T — the filtration time (s).

The separation efficiency (R , %) was calculated using Equation (2):

$$R = \frac{V_2}{V_1} \cdot 100 \% \quad (2)$$

where V_2 — the volume of water collected after separation; V_1 — the volume of water in water-in-oil emulsion before separation.

Results and Discussion

To separate “oil-in-water” type of emulsion, it is necessary to obtain membranes with a surface that can pass water and retain an organic medium. With this aim, we proposed a simple method for the formation of a polyelectrolyte complex by negatively charged pre-oxidized PET membrane surface contained carboxylic and hydroxylic groups and polyallylamine. One part of the amino groups of polyallylamine interacts with the membrane surface, and the other part can be ionized by soaking in solutions with acidic pH. For example, after soaking in pH=2, NH₂ groups are converted into charged NH₃⁺ groups, which make the membrane surface hydrophilic, since the charged groups have a dipole.

Figure 1 shows the contact angle (CA) values for water and hexadecane, measured for oxidized PET TeMs and modified PET TeMs-PAAM at different pH value (before measurement, the membranes were soaked in water with the appropriate pH).

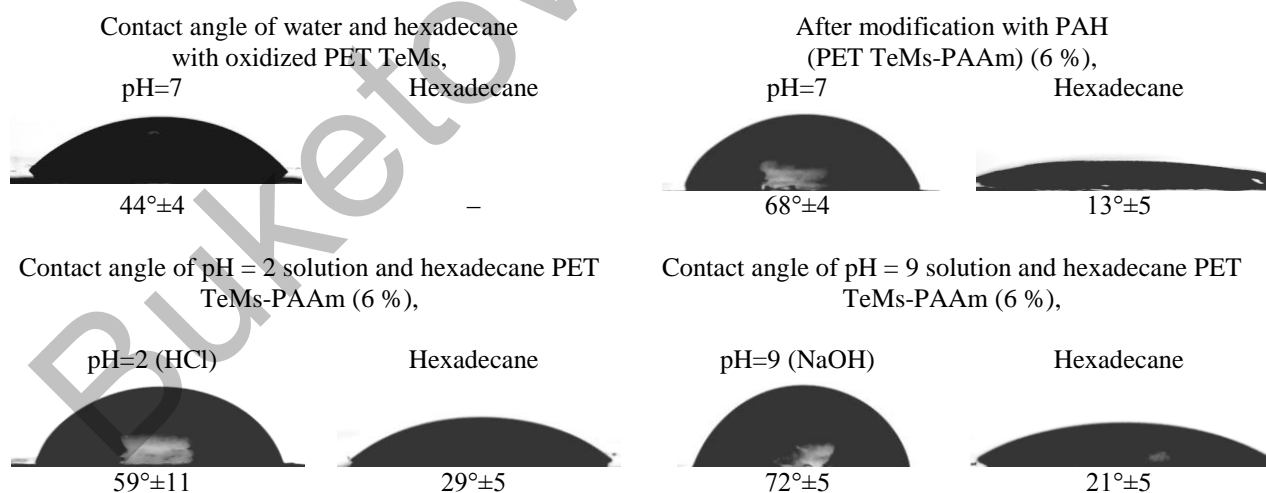


Figure 1 Contact angle of water, hexadecane, solutions pH = 2 and pH = 9 of the oxidized and modified PET TeMs-PAAM

As can be seen in Figure 1, the oxidized PET TeMs have water CA of 44°, however, hexadecane immediately passed through the pores of the membranes and this membrane cannot be used for water-oil separation, while PET TeMs-PAAM at pH=7 showed water CA of 68° and hexadecane of 13°, at pH=2 water CA is 59° and hexadecane of 29°. In this case, a drop of water passes through the pores of the membranes, while

a drop of hexadecane does not pass. This indicates that such membranes can be used to separate water-oil mixtures.

The FTIR spectra of the pristine PET TeMs contain absorption peaks at 2972 cm^{-1} (aromatic C-H), 2910 cm^{-1} (aliphatic C-H), 1716 cm^{-1} (C=O), 1471 cm^{-1} (CH_2 vibr.), 1410 cm^{-1} (CH vibr.), 1341 cm^{-1} (CH_2), 1246 cm^{-1} (stretching vibrations of C(O)-O bonds), 1019 cm^{-1} (CCC ring), 970 cm^{-1} (O- CH_2) [41]. The presence of polyallylamine on the surface causes the appearance of peaks at $\sim 2924\text{ cm}^{-1}$ and $\sim 980\text{ cm}^{-1}$ related to the -CH group, at $\sim 3400\text{ cm}^{-1}$ and 1624 cm^{-1} related to the amino groups - NH_2 and -NH (Fig. 2).

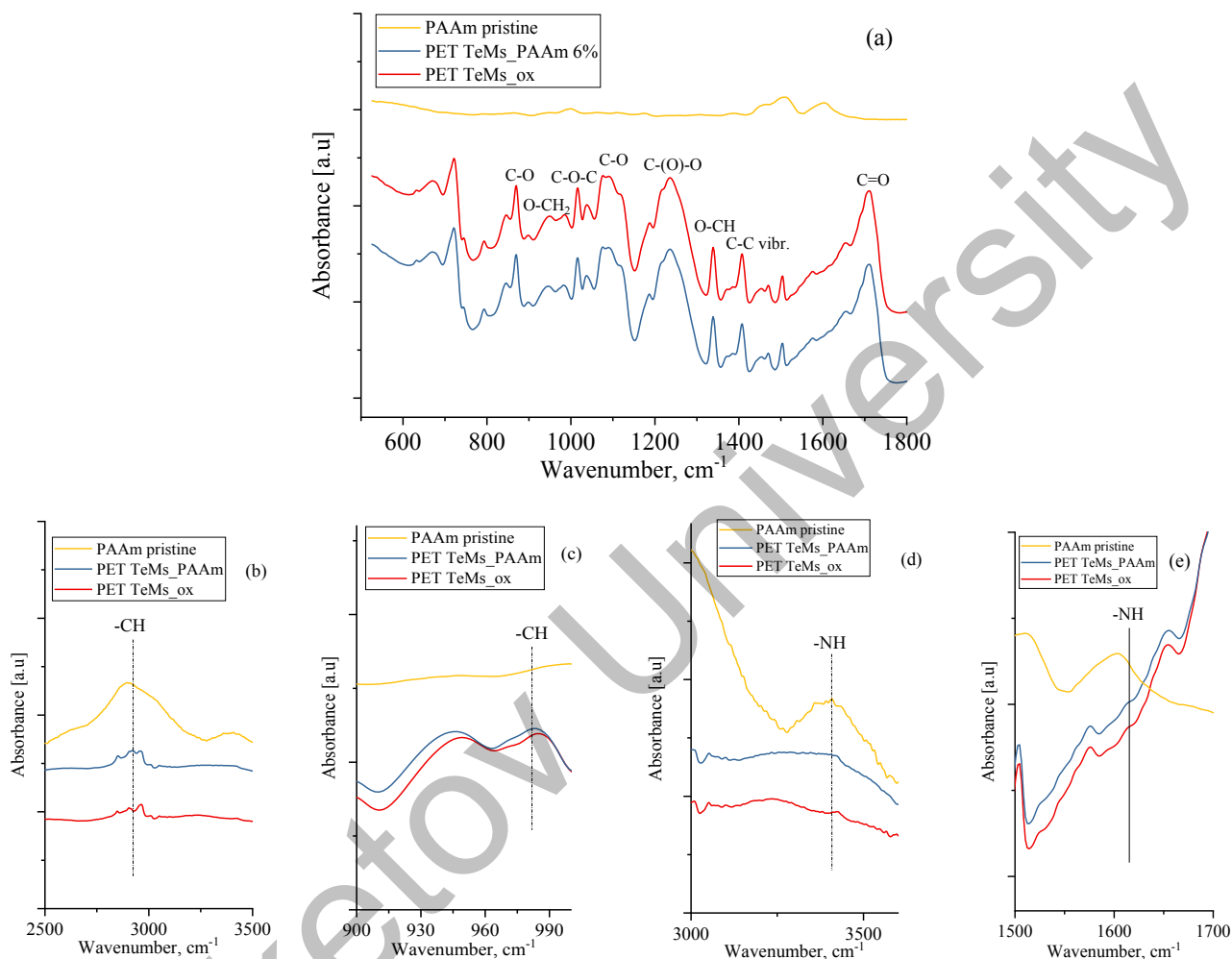
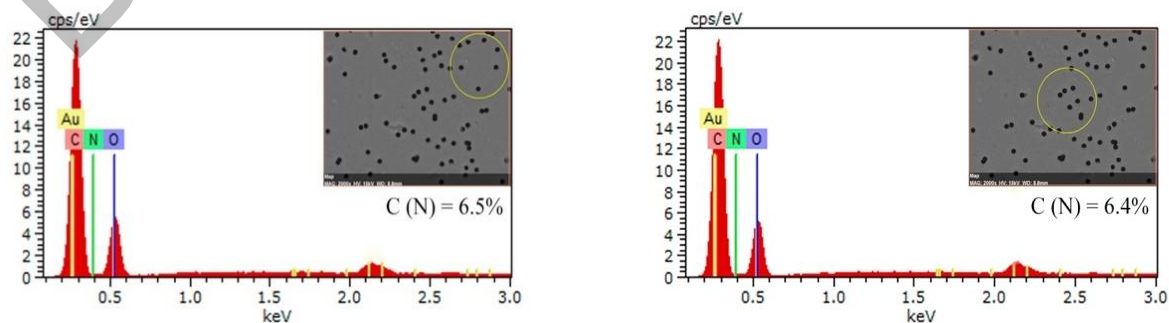


Figure 2. FTIR spectra of oxidized and modified PET TeMs-PAAm in the ranges of: $550\text{--}1800\text{ cm}^{-1}$ (a), $2500\text{--}3500\text{ cm}^{-1}$ (b), $900\text{--}1000\text{ cm}^{-1}$ (c), $3000\text{--}3550\text{ cm}^{-1}$ (d), $1500\text{--}1700\text{ cm}^{-1}$ (e)

EDX spectra of the surface of modified PET TeMs-PAAm are shown in Figure 3.



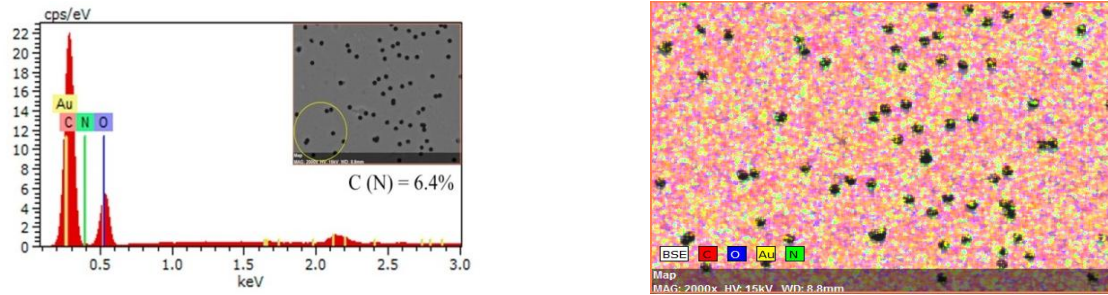


Figure 3. EDX spectra of modified PET TeMs-PAAm (6 %)

Figure 3 shows, that there is a uniform coverage of nitrogen (from PAAm) on the membrane surface. The presence of carbon and oxygen on the surface refers to PET TeMs. It should be noted that the gold content is present due to vacuum deposition prior to SEM analysis. Elemental analysis showed that the average content of nitrogen was ~6.4 %.

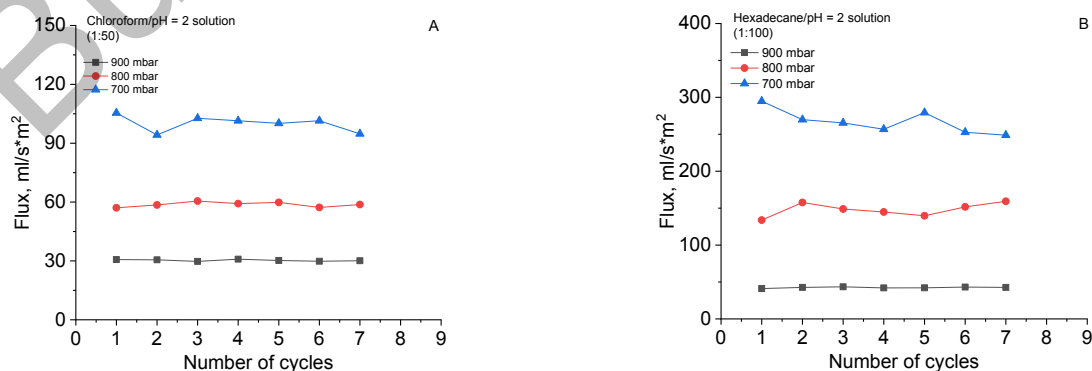
Table shows that the effective pore diameter decreases slightly after modification. This is probably due to the formation of a polyallylamine layer on the surface and inner pore walls of PET TeMs.

Table

Characteristics of the pristine (oxidized) and modified PET TeMs-PAAm

Sample	CA (pH=2), °	CA (hexadecane), °	Effective pore diameter, nm
PET TeMs ox.	44°±4	-	5061±19
PET TeMs-PAAm (6 %)	68°±4	13°±5	4862±16
PET TeMs-PAAm (6 %) (after soaking at pH = 2)	59°±11	29°±5	4810±9
PET TeMs-PAAm (6 %) (after soaking at pH = 9)	72°±5	20°±5	4806±15

The results of performance of modified PET TeMs-PAAm using chloroform-water (pH=2) solution (1:50), hexadecane-water (pH=2; 1:100) emulsions are shown in Figure 4. Since the hexadecane/water (pH=2) emulsion is more viscous, their ratio was greater than when using a mixture of chloroform/water (pH=2; 1:100 to 1:50). This resulted in greater performance of the hexadecane/water (pH=2) emulsion compared to chloroform/water (pH=2) at different pressures. At a pressure of 700 mbar, the average performance of chloroform/water (pH=2) was 100 ml/s·cm², and the mixture of hexadecane/water (pH=2) was 267 ml/s·cm². The performance was studied over 10 cycles, the surface stability of the modified layer of the membrane was observed. The degree of separation in almost all cases was more than 98 % and only slight decrease in flux was detected. Moreover, CA was controlled after each cycle of separation, which showed changes in CA within the standard error. Thus, a simple and effective method for the modification of PET TeMs with a pH-responsive surface has been developed, which can be successfully applied to separate oil-water emulsions.

Figure 4. The fluxes of modified PET TeMs-PAAm with pore density $1 \cdot 10^6$ for chloroform/pH=2 solution (1:50) and hexadecane/pH=2 solution (1:100) oil-water emulsions after each cycle at different pressure

Conclusions

In this study, we presented the results of modification of PET TeMs by soaking in poly(allylamine) solution. The effect of pH solutions and monomer concentrations providing the highest contact angle for hexadecane was studied. PET TeMs with pore diameters 5.1 μm ($1 \cdot 10^6$ pore density) were tested in oil-water emulsion separation by using hexadecane/water (pH=2; 1:100) and chloroform/water (pH=2; 1:50) as a model emulsions. Membranes showed that the average performance of chloroform/water (pH=2) was 100 ml/s·cm², and the mixture of hexadecane/water (pH=2) was 267 ml/s·cm².

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