

Polycarbonate Microfilters by Nuclear Tracking and Chemical Etching (Track-Etching) Technique: Preparation and Characterization

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Received 1 October 2004; accepted 17 April 2005

DOI 10.1002/app.22558

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Track-etched polycarbonate (PC) microfilters were successfully prepared by tracking with fission fragments in the Thai Research Reactor-1/Modification-1 nuclear reactor and chemical etching with sodium hydroxide aqueous solutions. The porosity of the as-prepared microfilters can be controlled by varying the exposure time in the nuclear reactor and the average pore diameter by varying the etching conditions. In the present work, the average pore diameter of the as-prepared microfilters ranged from ~ 2.0 to $9.5 \mu\text{m}$ (workable values) and the highest pore density achieved was $\sim 150,000$ pores cm^{-2} . It was found that chem-

ical etching also caused a reduction in the film thickness. Fourier-transform infrared spectroscopy results suggested some modifications in the chemical structure of the PC molecules after both nuclear tracking and chemical etching steps. Lastly, water permeability of the as-prepared, track-etched PC microfilters was found to increase with increasing average pore diameter. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 982–990, 2006

Key words: track-etched microfilters; porous media; polycarbonate; water permeability; mechanical integrity

INTRODUCTION

Porous polymeric membranes have received much attention in areas such as filtration, reverse osmosis, gas separation, pervaporation, controlled release, etc. In addition to all of these applications, they can be used as supports for the preparation of porous ceramic membranes or as templates for the preparation of metal nanotubes. Four common techniques for the preparation of porous polymeric membranes are (1) phase inversion,^{1–6} (2) extrusion-stretching,^{7–12} (3) template-leaching,^{13–18} and (4) track-etching^{19–24} techniques. These techniques have been developed based on the properties of the raw polymeric materials used and on the targeted applications of the resulting membranes. In addition, each technique arrives at membranes of different characteristics, e.g., average pore diameter and its uniformity, pore shape, pore density, mechanical integrity, etc.

Phase inversion,^{1–6} also known as phase separation or solution precipitation technique, is the most impor-

tant technique from which almost half of all microporous membranes are developed. In this technique, a clear polymer solution is precipitated into two phases: a solid polymer-rich phase that forms the membrane matrix and a liquid polymer-poor phase that forms the membrane pores upon evaporation. The membranes prepared by this technique are used mainly in ultrafiltration and reverse osmosis applications. This technique is, however, unsuitable for preparing porous polymeric membranes from polyolefins.

Extrusion-stretching technique,^{7–12} mainly used to prepare porous polymeric membranes from either filled or unfilled semicrystalline polymers, comprises two consecutive steps. In the first, an oriented film is produced by melt-extrusion process with a rapid haul-off speed. After solidifying, the film is stretched in either parallel or perpendicular direction to the original orientation of the polymer crystallites. For unfilled systems, the second stretching deforms the crystalline structure of the film and produces slit-like pores with diameters ranging from ~ 20 to 50 nm .^{7,9,10} For filled systems, the second stretching results in partial removal of the solid fillers, yielding porous structure.^{8,11,12} Traditionally, porous membranes prepared by this technique have relatively poor tear strength

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along the orientation direction and are not widely used as microfiltration membranes. Instead, they have been used as inert separating porous barriers in batteries and some medical devices.

Template-leaching^{13–18} is the technique suitable for preparing porous membranes from polymers, which do not dissolve in common organic solvents. In this technique, a homogeneous film is prepared from a mixture of membrane matrix material and a leachable component. The leachable component can be a soluble low molecular weight substance or even a macromolecular such as poly(vinyl alcohol) or poly(ethylene glycol). Recently, we utilized tapioca starch as the leachable component to prepare porous low-density polyethylene membranes.¹⁸ After the film has been prepared, the leachable component is removed by a suitable chemical treatment. At right conditions, porous structure is formed as a result.

Track-etching or pore-piercing technique^{19–24} is composed of two main steps. The first step involves irradiation of a polymer film with high-energy particles generated by a nuclear reactor or other types of radiation sources. The high-energy particles can be α -particles,^{19,20} accelerated heavy ions,^{21,22} and fission fragments from a reaction between uranium-235 and thermal neutrons.^{23,24} When high-energy particles pass through the film, polymer chains are broken, leaving sensitized or damaged tracks. In the second step, the film is submerged in an etching solution, which etches the polymer film along the sensitized tracks to form flow-through channels or pores. The number and the diameter of pores can be controlled by adjusting the exposure time in the first step and the concentration of the etching solution as well as the etching time in the second step.

In the present contribution, porous polycarbonate (PC) membranes were prepared by first bombarding melt-extruded PC films with fission fragments from a reaction of uranium-235 with thermal neutrons in the beam tunnel of the Thai Research Reactor-1/Modification-1 (TRR-1/M-1) (Office of Atomic for Peace, Thailand) and later by chemically etching the as-tracked films in sodium hydroxide (NaOH) aqueous solutions. The main objective was to promote utilization of the local nuclear reactor for manufacturing low-cost track-etched membranes to be used locally within academic and governmental institutions.

EXPERIMENTAL

Materials

Polycarbonate (PC) resin used was Makrolon 2805 general purpose grade from Bayer Polymers (Thailand). The melt flow rate of this resin is 10 g/10 min. After drying in an oven at 80°C for 5 h, PC pellets were fed into a LabTech Engineering LCR-300 chill-roll cast-

film extruder. The die temperature was set at 300°C. The as-extruded PC films were $\sim 15 \pm 2 \mu\text{m}$ in thickness. Uranium screens, used to generate fission fragments during the tracking step, were prepared by applying ammonium diuranate $[(\text{NH}_4)_2\text{U}_2\text{O}_7]$ paste onto $2 \times 6 \text{ cm}^2$ cellulose acetate backing sheets. Etching solutions were aqueous solutions of sodium hydroxide (NaOH) [analytical grade, Sigma-Aldrich, St. Louis, MO], which were prepared in various concentrations ranging from 4 to 8N. Ethanol [analytical grade, Sigma-Aldrich] was used to terminate the etching reaction of the as-tracked films.

Tracking and etching procedure

Tracking of the as-extruded PC films was carried out by bombarding a PC specimen, consisting of a small PC film of $2.5 \times 8 \text{ cm}^2$ in its dimension laying side by side with an as-prepared uranium screen in a neutron-transparent cassette, in the beam tunnel of the TRR-1/M-1. The effect of the tracking distance on track density and track flux of the as-tracked PC films was investigated by placing the as-prepared PC specimens at 70, 80, 90, or 100 cm from the reactor core for a fixed exposure time of 60 min. The neutron flux corresponding to each tracking distance was measured to be 1.09×10^6 , 1.05×10^6 , 9.16×10^5 , and 8.24×10^5 neutrons $\text{s}^{-1} \text{ cm}^{-2}$ (these values were an average over the exposure time of 60 min). The effect of the exposure time was investigated at a fixed tracking distance of 70 cm for 60, 120, 180, or 240 min. It should be noted that the tracking characteristics of the as-tracked PC films were observed after etching in 6N NaOH solution at 70°C for 60 min.

Chemical etching is a process to enlarge the damaged tracks formed during the tracking step to form flow-through pores. In this step, the effects of the concentration of the etchant, the etching temperature, and the etching time on the quality of the as-etched microfilters (by considering average number of pores per unit area and average pore diameter of the as-etched microfilters obtained) were investigated. On the effect of etchant concentration, the initial concentration of NaOH aqueous solutions was 2, 4, 6, or 8N. On the effect of etching temperature, the temperature of the etchant was 50, 60, 70, or 80°C. Lastly, on the effect of etching time, the submersion time during etching was 30, 45, 60, 75, 105, 120, 135, or 150 min. In these experiments, the tracking distance and the tracking exposure time were fixed at 70 cm and 180 min, respectively.

Characterization

The average number of pores per unit area (i.e., average pore density) and the average pore diameter were examined by an Olympus BH-2 optical microscope,

TABLE I
Neutron Flux, Track Density, and Track Flux as a Function of Tracking Distance from the Reactor Core in the Beam Tunnel of the TRR-1/M-1 Reactor^a

Tracking distance (cm from the reactor core)	Neutron flux (neutron s ⁻¹ cm ⁻²)	Track density (track cm ⁻²)	Track flux (track s ⁻¹ cm ⁻²)
70	1.09 × 10 ⁶	4.96 ± 0.49 × 10 ⁴	14
80	1.05 × 10 ⁶	4.23 ± 0.43 × 10 ⁴	12
90	9.16 × 10 ⁵	3.40 ± 0.09 × 10 ⁴	9
100	8.24 × 10 ⁵	2.99 ± 0.10 × 10 ⁴	8

^a The exposure time was fixed at 60 min.

using the eye piece lens of 10× and the objective lens of either 40× or 100×. A JSM-5800 LV scanning electron microscope (SEM) was used to determine the geometry of pores of the as-prepared microfilters. Prior to SEM examination, each specimen was coated with a thin layer of gold to enhance the conductivity of the surface. A Mitutoyo digital micrometer was used to measure the thickness of neat PC films and as-etched PC microfilters, to observe whether the etchant used had an adverse effect on the thickness of the resulting PC microfilters. A Nicolet impact 4.1 Fourier-transform infrared spectroscope (FTIR) was used to verify whether there was any chemical change to the PC films after the nuclear tracking and the chemical etching steps, respectively. The scan range was between 400 and 4000 cm⁻¹. Water permeability of the as-prepared PC microfilters was measured under a constant pressure of ~1170 N m⁻² at room temperature on a circular area of 0.025 m². Lastly, the as-prepared PC microfilters were tested for their mechanical integrity. Tensile strength, yield strength, Young's modulus, and percentage of elongation at yield were measured on a Lloyd LR10K universal testing machine, following ASTM D882 standard test method. A load cell of 100N, a crosshead speed of 50 mm min⁻¹, and a gauge length of 250 mm were used.

RESULTS AND DISCUSSION

Formation of latent tracks

Formation of latent tracks in PC films was a direct result of the bombardment of fission fragments originated from a nuclear reaction between uranium-235 and thermal neutrons in the TRR-1/M-1 reactor. The mechanism for a nuclear reaction of uranium-235 and thermal neutrons is a common knowledge. When a thermal neutron is captured within the nucleus of uranium-235, the total energy is distributed among the 236 nucleons (i.e., protons and neutrons) being present in the compound nucleus. This instantaneous nucleus is unstable and is likely to break off into two fragments of approximately equal masses. Fission products of the fission reaction between uranium-235 and a thermal neutron are, for examples, barium, krypton,

zirconium, and lanthanum. These high-energy fission fragments can pass through a PC film, which causes some PC molecules to break, leaving sensitized, damaged tracks along their path.

The density of the latent tracks within PC films irradiated in the beam tunnel of the TRR-1/M-1 reactor depended on the tracking distance (or, to be exact, the neutron flux) and the exposure time. Since neutron flux was found to be a decreasing function of the tracking distance, both track density (i.e., the average number of tracks per unit area) and track flux should also be a decreasing function of the tracking distance, as evidently shown in Table I. Based on the results shown, the tracking distance of 70 cm from the reactor core was chosen to be the tracking distance because of the resulting highest track density and track flux values. Table II shows the effect of exposure time on track density and track flux of as-tracked PC films at a fixed tracking distance of 70 cm. Obviously, even though track density was found to be an increasing function of the exposure time, normalization of the observed track density values with corresponding exposure time resulted in relatively constant track flux of ~14 track s⁻¹ cm⁻². Even though the exposure time of 240 min gave the highest track density value, the exposure time of 180 was chosen, since the as-etched PC microfilters obtained after irradiating for 240 min showed a large number of overlapping pores.

Formation of flow-through pores by chemical etching

In the tracking step, it was found that both the tracking distance and the exposure time affected a great

TABLE II
Track Density and Track Flux as a Function of Exposure Time in the Beam Tunnel of the TRR-1/M-1 Reactor^a

Exposure time (min)	Track density (track cm ⁻²)	Track flux (track s ⁻¹ cm ⁻²)
60	4.96 ± 0.49 × 10 ⁴	13
120	1.02 ± 0.07 × 10 ⁵	14
180	1.54 ± 0.13 × 10 ⁵	14
240	1.95 ± 0.08 × 10 ⁵	14

^a The tracking distance was at 70 cm.

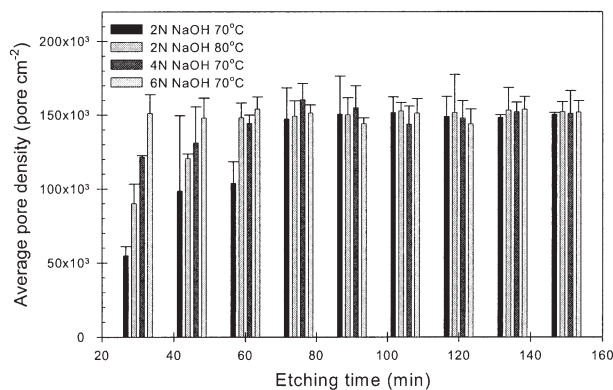


Figure 1 Average number of pores per unit area (i.e., pore density) as a function of etching time for four different etching conditions of the obtained track-etched PC microfilters.

deal the track density and the track flux of the as-tracked PC films. Among the tracking parameters investigated, the tracking distance of 70 cm and the exposure time of 180 min were chosen based simply on the track density and the geometry of the resulting pores (i.e., nonoverlapping pores). In this chemical etching step, the effects of etching conditions, e.g., concentration of the etchant, etching temperature, and etching time on average number of pores per unit area and average pore diameter were thoroughly investigated.

Figure 1 shows the effects of etching conditions on average pore density of the resulting PC microfilters. It should be noted that only some etching conditions were illustrated. The results obtained suggest that the concentration of the etching solution played a major role in widening the latent tracks to form through holes. In the mildest etching condition shown (i.e., 2N and 70°C), the average pore density was found to increase from $\sim 55,000$ pore cm^{-2} at the etching time of 30 min to $\sim 151,000$ pore cm^{-2} after being etched for 90 min and, with further increase in the etching time, the average pore density did not vary appreciably (i.e., the pore density averaging over the etching times between 90 and 150 min being $\sim 150,000$ pore cm^{-2}). In the second mildest etching condition shown (i.e., 4N and 70°C), the average pore density increased from $\sim 122,000$ pore cm^{-2} at the etching time of 30 min to $\sim 161,000$ pore cm^{-2} after being etched for 75 min and, with further increase in the etching time, the average pore density did not vary appreciably (i.e., the pore density averaging over the etching times between 75 and 150 min being $\sim 152,000$ pore cm^{-2}).

Obviously, in mild etching conditions (i.e., 2 and 4N and 70°C), the average pore density increased initially with increasing etching time and later levelled off at higher etching times. At higher concentrations of the

etchant (i.e., 6 and 8N and 70°C), the average pore density was independent of the etching time, with the average pore density for the etchant concentrations of 6 and 8N being $\sim 150,000$ and $151,000$ pore cm^{-2} (reported as an average value over the etching times between 30 and 150 min), respectively. The effect of etching temperature can also be observed in Figure 1. At 2N, an increase in the etching temperature resulted in an increase in the average pore density (especially at "low" etching times). Specifically, the average pore density increased from $\sim 90,000$ pore cm^{-2} at the etching time of 30 min to $\sim 150,000$ pore cm^{-2} after being etched for 90 min and, with further increase in the etching time, the average pore density did not vary appreciably (i.e., the pore density averaging over the etching times between 90 and 150 min being $\sim 152,000$ pore cm^{-2}).

Figure 2 illustrates the effects of etching conditions on average pore diameter of the resulting PC microfilters. According to the results obtained, the average pore diameter increased monotonically with increasing NaOH concentration, etching temperature, and etching time. Interestingly, the mildest etching condition of 2N NaOH at 50°C was not enough to enlarge the latent tracks to form through holes of large enough diameters to be visible under an optical microscope. If the etching temperature was increased to 60°C, enlarged pores were clearly visible under the optical microscope only after the as-tracked PC films were etched for at least 60 min (i.e., the average pore diameter being ~ 0.49 μm). On the contrary, the harshest etching conditions (i.e., prolonged etching at 6N NaOH at 80°C and 8N NaOH at 80°C) resulted in the disintegration of the PC films. Qualitatively, based on the results shown, the average pore diameter of the obtained PC microfilters was in the range of 0.49 ± 0.19 to 10.31 ± 0.47 μm .

The chemical etching of PC films with NaOH can occur in two ways. The first is the chemical etching along the inner surface of the latent tracks formed within the PC films and the second is the chemical etching along the general surface of the PC films.²⁵ The first is responsible for the widening of the latent tracks, while the second is responsible for the thinning of the PC films in general. Figure 3 shows the thickness of the PC microfilters as a function of the average pore diameter. It should be noted that only the data obtained at some selected etching conditions were included in the plot. Apparently, the thickness of the obtained PC microfilters was found to decrease from $\sim 15 \pm 2$ μm at the average pore diameter of ~ 0.49 μm (corresponding to the etching condition of 2N at 60°C for 60 min) down to $\sim 7 \pm 2$ μm at the average pore diameter of ~ 9.51 μm (corresponding to the etching condition of 6N at 80°C for 120 min).

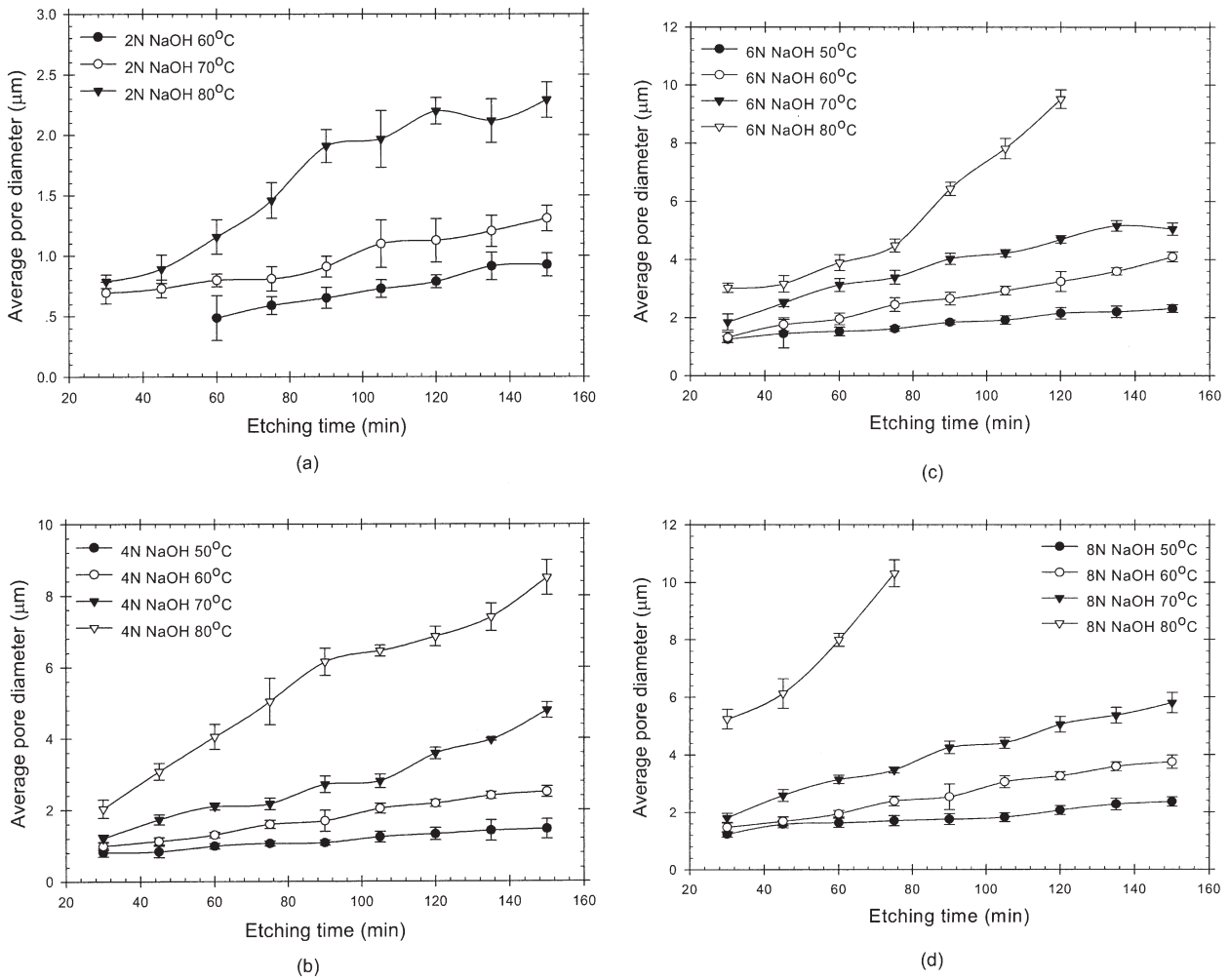


Figure 2 Average pore diameter as a function of etching time for four different concentrations of the etchant [i.e., (a) 2, (b) 4, (c) 6, and (d) 8N] and four different etching temperatures (i.e., 50, 60, 70, and 80°C) of the obtained track-etched PC microfilters.

Geometry of track-etched pores

Figure 4 shows the geometry of some track-etched pores of the PC microfilters, which were tracked in the

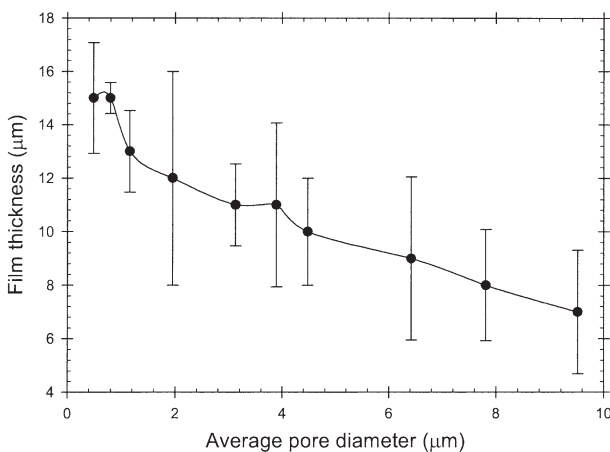
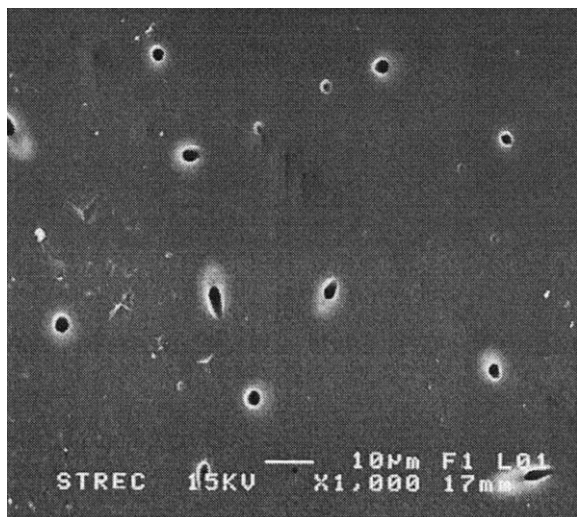


Figure 3 Thickness of track-etched PC microfilters as a function of average pore diameter.

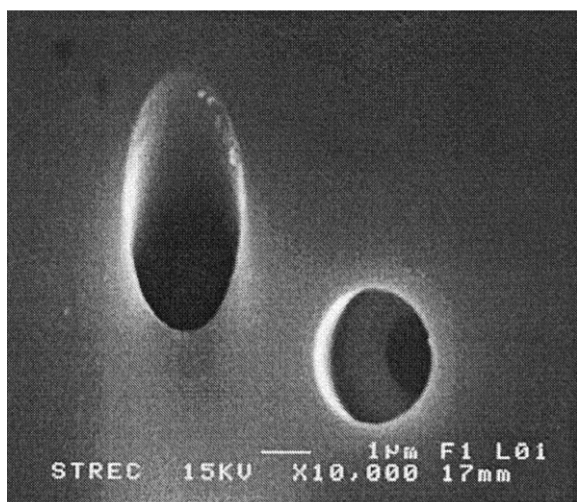
beam tunnel at 70 cm from the reactor core for 180 min and etched in 6N NaOH solution at 70°C for 60 min. Evidently, the geometry of the pores was either circular or elliptic. The formation of such appearance of the pores is a direct result of the angle at which fission fragments came into contact with PC films with respect to the surface of the films during the tracking step. Only the fission fragments penetrating the films at right angles resulted in the formation of circular pores, while those penetrating at any other angles resulted in the formation of elliptic pores.

Chemical changes after nuclear tracking and chemical etching steps

It is now a general knowledge that interaction of heavy ions with polymer molecules (e.g., in polymeric films which have been tracked by heavy ions) leads to bond breakage and formation of free radicals.²⁶⁻²⁸ In the present contribution, FTIR technique was used to investigate any change in the chemical structure of PC



(a)



(b)

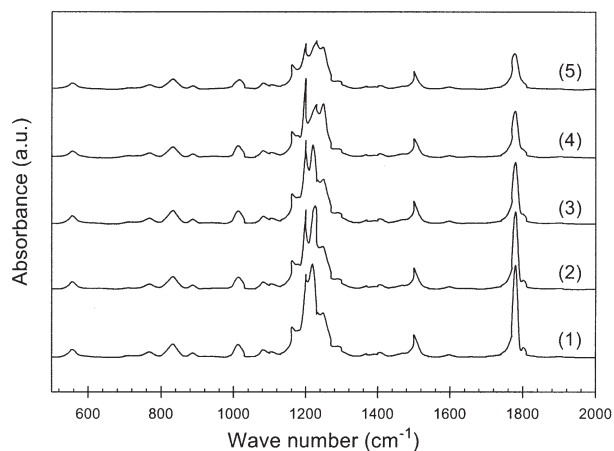
Figure 4 Scanning electron micrographs of track-etched PC microfilters at magnifications of (a) $\times 1000$ and (b) $\times 10,000$. The tracking conditions were 70 cm and 180 min and the etching conditions were 6N at 70°C for 60 min. The scale bar shown is for 1 μm .

molecules after nuclear tracking and chemical etching steps. Obviously, the chemical functional groups of a PC repeating unit include methyl, phenyl ring, carbonyl, ether, and hydroxyl. The infrared absorption peaks corresponding to such groups are 2970 cm^{-1} (νCH_3), 1510 cm^{-1} ($\nu\text{C}-\text{H}$, aromatic), 1782 cm^{-1} ($\nu\text{C}=\text{O}$), 1012 cm^{-1} ($\nu\text{C}-\text{O}-\text{C}$), and 3500 cm^{-1} ($\nu\text{O}-\text{H}$), respectively,²⁶ (compared with the FTIR spectrum of the as-extruded PC film shown in Fig. 5).

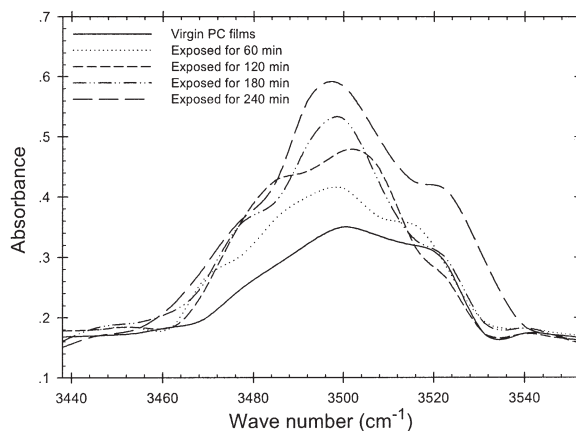
Figure 5 illustrates FTIR spectra of as-extruded PC film and PC films which were irradiated with fission fragments in the beam tunnel at 70 cm from the reactor core at the exposure times of 60, 120, 180, and 240 min (corresponding to the track densities of $\sim 4.96 \times 10^4$, 1.02×10^5 , 1.54×10^5 , and 1.95×10^5 track cm^{-2} ,

respectively). Evidently, no new peaks were observed in the FTIR spectra obtained, indicating that no new chemical functional groups were introduced during the tracking step. Careful observation, however, revealed that the absorbance at wave numbers of 1012, 1510, and 1782 cm^{-1} of the as-tracked PC films decreased, while the absorbance at 3500 cm^{-1} increased, from that of the virgin PC film and with increasing exposure time. Clearly, the normalized absorbance of the as-tracked PC films (i.e., the absorbance of the virgin PC film was used as the reference) at the wave numbers of 1012, 1510, and 1782 cm^{-1} decreased, while that at 3500 cm^{-1} increased, with increasing exposure time or with increasing average track density (see Table III).

The results suggest that, during the tracking step, bombardment of the fission fragments generated into the PC films caused some PC molecules to undergo



(a)



(b)

Figure 5 (a) FTIR spectra of (1) virgin PC film and PC films which were exposed to fission fragments in the beam tunnel at 70 cm from the reactor core for (2) 60, (3) 120, (4) 180, (5) 240 min in the wave number range of 500–2000 cm^{-1} and (b) corresponding spectra in the wave number region of 3500 cm^{-1} .

TABLE III
Normalized Absorbance at Wave Numbers of 1012, 1510, 1782, and 3500 cm^{-1} for Polycarbonate Films That Were Exposed to Fission Fragments in the Beam Tunnel at 70 cm From the Reactor Core for Different Time Intervals

Exposure time (min)	Average track density (track cm^{-2})	A/A_0 at 1012 cm^{-1}	A/A_0 at 1510 cm^{-1}	A/A_0 at 1782 cm^{-1}	A/A_0 at 3500 cm^{-1}
60	4.96×10^4	0.969	0.958	0.861	1.169
120	1.02×10^5	0.953	0.904	0.669	1.328
180	1.54×10^5	0.885	0.844	0.492	1.519
240	1.95×10^5	0.835	0.762	0.414	1.656

chain scission at ether, phenyl ring, and carbonyl groups, which apparently resulted in a decrease in the number of these groups. The longer the exposure time was, the greater the extent of the chain scission would be. Chain scission causes a reduction in the molecular weights of a polymer, which, in turn, causes the number of chain ends to increase. In the case of PC, the reduction in the molecular weights resulted in an increase in the number of hydroxyl chain ends, which is directly responsible for the observed increase in the normalized absorbance at the wave number of 3500 cm^{-1} .

The effects of etchant concentration, etching temperature, and etching time on any change in the chemical structure of PC molecules after the chemical etching step were also investigated. FTIR spectra obtained for all of the track-etched PC microfilters (i.e., the tracking conditions were fixed at 70 cm from the reactor core in the beam tunnel and 180 min exposure time) exhibited characteristic peaks similar to those shown in Figure 5 (the actual results not shown) and no new peaks were observed in the FTIR spectra obtained, indicating that no new chemical functional groups were introduced during the chemical etching step. Unlike the case of as-tracked PC films, track-etched PC microfilters only shows a reduction in the absorbance at wave numbers of 1012 and 1782 cm^{-1} and an increase in the absorbance at 3500 cm^{-1} with increasing the severity of the etching conditions (i.e., increasing etchant concentration, increasing etching temperature, and increasing etching time), while the absorbance at 1510 cm^{-1} was practically unaffected.

In stead of reporting the results obtained for every etching condition studied as a function of the individual parameter (i.e., etchant concentration, etching temperature, etching time), the normalized absorbance of some track-etched PC microfilters (i.e., the absorbance of the virgin PC film was used as the reference) at the wave numbers of 1012, 1782, and 3500 cm^{-1} was reported as a function of the average pore diameter in Figure 6. It should be noted that only the data obtained at some selected etching conditions were included in the plot. Apparently, the normalized absorbance at the wave numbers of 1012 and 1782 cm^{-1} decreased, while that at 3500 cm^{-1} increased, with increasing average pore diam-

eter. Since the average pore diameter was found to increase with increasing strength of the etching condition (i.e., increasing etchant concentration, increasing etching temperature, and increasing etching time), the results obtained imply that chemical etching caused the number of carbonyl and ether groups to decrease and the number of hydroxyl group to increase. Since the chemical etching of PC molecules with NaOH should occur via a chemical reaction between OH ions at the ester linkage, caus-

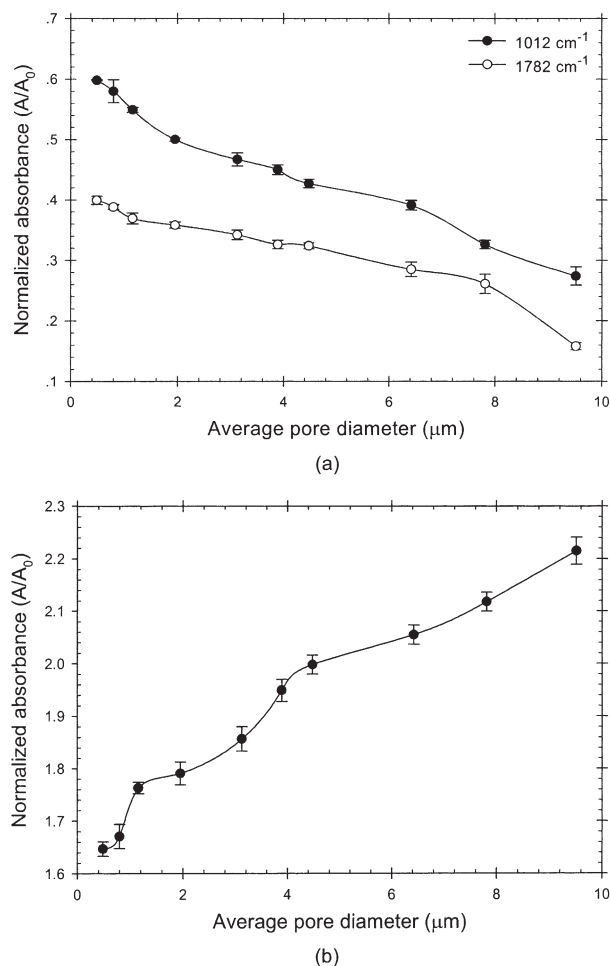


Figure 6 Normalized absorbance at wave numbers of (a) 1012, 1782 and (b) 3500 cm^{-1} for track-etched PC microfilters as a function of average pore diameter.

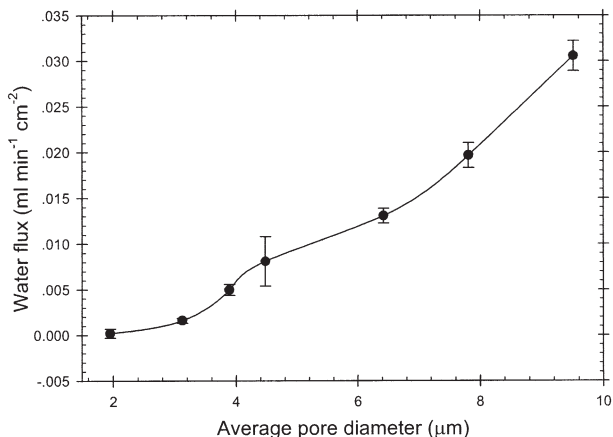
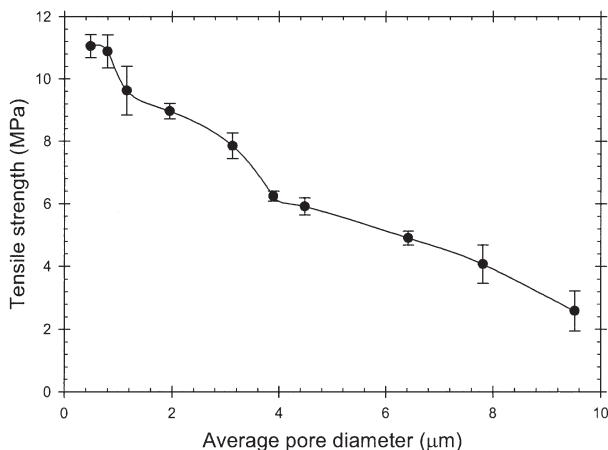


Figure 7 Water flux of track-etched PC microfilters as a function of average pore diameter.

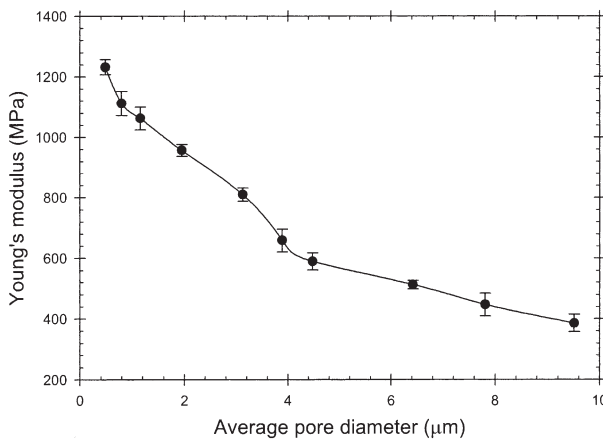
ing the number of both carbonyl and ether groups to decrease, while the number of hydroxyl group to increase, as evidently verified by the obtained results.

Water permeability

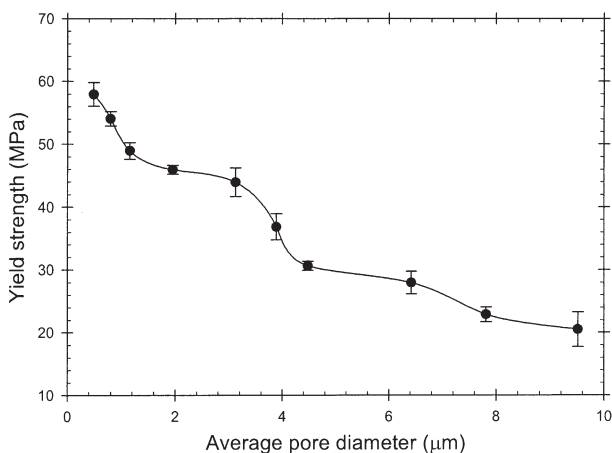
Water permeability of some of the as-prepared track-etched PC microfilters was tested and the results are plotted as a function of the average pore diameter in Figure 7. The tracking conditions of these microfilters were 70 cm from the reactor core in the beam tunnel and 180 min exposure time. Evidently, water permeability exhibited a strong correlation with the average pore diameter, in which the water flux increased monotonically from $\sim 1.80 \times 10^{-4}$ mL min⁻¹ cm⁻² when the average pore diameter was ~ 1.95 μm to $\sim 3.10 \times 10^{-4}$ mL min⁻¹ cm⁻² when the average pore diameter was 9.51 μm. The track-etched microfilters having the average pore diameter smaller than ~ 1.95 μm were also tested for their water permeability, but no water could pass through under the test condition (i.e., under the applied constant pressure of ~ 1170 N m⁻² at room temperature), which imply either that the etching conditions used to obtain the average pore diameter smaller than ~ 1.95 μm were not enough to cause the latent tracks to form flow-through pores or



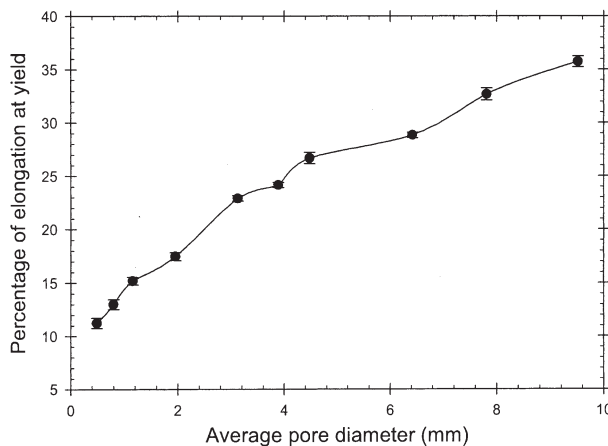
(a)



(c)



(b)



(d)

Figure 8 Mechanical properties [i.e., (a) tensile strength, (b) yield strength, (c) Young's modulus, and (d) percentage of elongation at yield] of track-etched PC microfilters as a function of average pore diameter.

that the pressure used was too low to cause water to flow through the very small track-etched pores.

Mechanical integrity

Apart from water permeability, which is one of the major criteria used to characterize track-etched microfilters, mechanical integrity of such films is also a major concern limiting the utilization in real applications. Figure 8 shows tensile strength, yield strength, Young's modulus, and percentage of elongation at yield for some of the as-prepared track-etched PC microfilters plotted as a function of the average pore diameter. Again, these microfilters were tracked at 70 cm from the reactor core in the beam tunnel for 180 min. It should be noted that mechanical integrity of the as-extruded and the as-tracked PC films was also assessed. For the as-extruded PC film, values of the tensile strength, yield strength, Young's modulus, and percentage of elongation at yield were ~ 13.4 , 62.7, 1490 MPa, and 7.1%, respectively, while for the as-tracked PC films, they were 11.7, 59.0, 1448 MPa, and 7.7%, respectively. Clearly, irradiation of the PC films in the tracking condition used deteriorated the mechanical integrity of the PC films to some extent. Greater extent in the deterioration of the mechanical integrity of the PC films arrived after chemical etching, which is reflected in the monotonic decrease in the tensile strength, yield strength, and Young's modulus with increasing average pore diameter and in the monotonic increase in the percentage of elongation at yield. The deterioration of the mechanical integrity of the obtained PC microfilters is quite straight forward, since the increase in both size and density of the track-etched pores caused cracks to propagate much easier, which, in turn, caused the microfilters to be more stretchable.

CONCLUSIONS

In the present contribution, track-etched PC microfilters were successfully prepared by irradiating as-extruded PC films by fission fragments generated by a nuclear reaction between uranium-235 and thermal neutrons in the beam tunnel of the TRR-1/M-1 nuclear reactor and enlarging the latent tracks formed by chemical etching with sodium hydroxide aqueous solutions. The average number of pores per unit area of the as-prepared microfilters can be controlled by varying the exposure time in the nuclear reactor and the average pore diameter by varying the etching conditions. The average pore diameter of the as-prepared microfilters ranged from ~ 0.5 to $10.3 \mu\text{m}$, with the workable range being between ~ 2.0 and $9.5 \mu\text{m}$. The highest number of pores per unit area which could be achieved was $\sim 150,000 \text{ pore cm}^{-2}$. It was found that chemical etching not only caused latent tracks to enlarge, but also resulted in a decrease in the film thickness. Both nuclear tracking and chemical etching were found to shorten the PC molecules. The water permeability of the as-prepared microfilters was found

to range from $\sim 1.80 \times 10^{-4}$ to $3.10 \times 10^{-4} \text{ mL min}^{-1} \text{ cm}^{-2}$. Finally, the tensile strength, yield strength, and Young's modulus of the as-prepared microfilters were found to decrease from those of the as-extruded and the as-tracked PC films, and were found to decrease with increasing strength of the etching condition or with increasing average pore diameter.

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