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PREPARATION OF POLYCARBONATE MICROFILTERS BY NUCLEAR
TRACKING-CHEMICAL ETCHING TECHNIQUE

Miss Pornphan Makphon

A Thesis Submitted in Partial Fulfillment of the Requirements

for the Degree of Master of Science in Petrochemistry and Polymer Science

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Thesis Title   PREPARATION OF POLYCARBONATE MICROFILTERS BY NUCLEAR TRACKING-CHEMICAL ETCHING TECHNIQUE

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การทดลองนี้ ได้เตรียมแผ่นกรองจากพอลิคาร์บอเนตด้วยเทคนิคการเจาะรูด้วยอนุภาคนิวเคลียร์ที่เกิดจากปฏิกิริยาฟิซชันของยูเรเนียม-235 และการกัดกร่อนเพื่อขยายขนาดรูด้วยสารเคมีเป็นผลสำเร็จ แผ่นฟิล์มพอลิคาร์บอเนตที่ใช้ในการทดลองนี้มีความหนาโดยเฉลี่ยประมาณ 13 ไมโครเมตร การศึกษาถึงผลกระทบของปัจจัยต่างๆ ที่มีต่อความหนาแน่นของรู ขนาดของรู และลักษณะรูปร่างของรู ได้เน้นที่ผลของระยะเวลาที่แผ่นฟิล์ม (ซึ่งถูกประกบอยู่กับสารประกอบของยูเรเนียม-235) ที่ถูกอาบด้วยนิวตรอน ผลของชนิด ความเข้มข้น และสภาวะของสารเคมีที่ใช้ในการกัดกร่อนเพื่อขยายขนาดของรู

งานวิจัยนี้พบว่าแผ่นกรองที่เตรียมได้มีระยะเส้นผ่าศูนย์กลางของรูอยู่ในช่วงระหว่าง 0.486 ถึง 9.514 ไมโครเมตร โดยมีค่าความหนาแน่นของรูอยู่ในช่วงประมาณ 50,000 ถึง 155,000 รูต่อตารางเซนติเมตร แต่อย่างไรก็ตามพบว่า ความหนาของแผ่นกรองลดลงเมื่อใช้เวลากัดกร่อนเพิ่มขึ้น ในการศึกษานี้จะมุ่งเน้นไปที่ผลของสภาวะในการกัดกร่อนด้วยสารเคมีต่อสมบัติเชิงกลของแผ่นกรอง พบว่าแรงยืดดึงที่จุดขาด (tensile strength) แรงยืดดึงที่ทำให้ไม่สามารถคืนรูปได้ (yield strength) และความต้านทานการเสียรูปของชิ้นงาน (modulus) มีค่าลดลง ในทางตรงกันข้ามอัตราเปลี่ยนแปลงการยืดดึง (% elongation) มีค่าเพิ่มขึ้น ผลการวิเคราะห์ลักษณะโครงสร้างทางเคมีของแผ่นกรองด้วยฟูเรียร์ทรานสฟอร์มอินฟาเรดสเปกโทรสโคปี(FTIR) พบว่าเปลี่ยนแปลงไปเมื่อเจาะรูด้วยอนุภาคนิวเคลียร์ และกัดกร่อนเพื่อขยายขนาดรูด้วยสารเคมี ผลการศึกษาการผ่านได้ของน้ำบ่งชี้ว่า ปริมาณน้ำผ่านได้มากขึ้นเมื่อขนาดของรูเพิ่มขึ้น

สาขาวิชาปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์   ลายมือชื่อนิสิต..........................

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#:4572405023 MAJOR PETROCHEMISTRY AND POLYMERSCIENCE

KEY WORD: NUCLEAR TRACKING-CHEMICALETCHING/ FISSION FRAGMENT

PORNPHAN MAKPHON : PREPARATION OF POLYCARBONATE MICROFILTERS BY NUCLEAR TRACKING-CHEMICALETCHING TECHNIQUE. THESIS ADVISOR : ASSOC. PROF. SUPAWAN TANTAYANON, Ph.D., THESIS COADVISOR: ASST. PROF. PITT SUPAPHOL, Ph.D. [135] pp. ISBN: 974-17-4177-4

Track-etched PC microfilters have been successfully prepared by first irradiating 13-micron polycarbonate films with fission fragments from uranium (U-235) in a nuclear reactor and subsequently subjecting the treated films through an etching procedure in order to enlarge the radiation-induced damaged tracks to produce a porous structure in the finished films. Varying the irradiation time in nuclear reactor and the average pore diameter by varying the etching conditions can readily control the porosity of the filters.

In this work, the as-prepared microfilters had the average pore diameter ranging from ca. 0.486 to 9.514 micrometers and the pore density ranging from 50,000 to 155,000 pores per square centimeter. However, the thickness of microfilter was found decreasing with increasing pore diameter.   This study focuses on the effects of etching conditions on the mechanical integrity of the as-prepared filters. It was found that the tensile strength, yield strength and modulus decreased with porosity but % elongation was increased. Fourier transformed infrared spectroscopy (FTIR) results suggested some modifications to the chemical structure after fission fragment tracking and chemical etching. The water permeability seems increasing with increasing pore diameter of track-etched PC microfilters.

Field of study Petrochemistry and Polymer Science   Student's signature……………………..………

Academic year 2003   Advisor's signature……………………..……

                                                         Co- advisor's signature…………………………

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LIST OF ABBREVIATIONS

NaOH	Sodium hydroxide
OM	Optical microscope
SEM	Scanning electron microscope
FTIR	Fourier transform infrared
°C	Degree Celsius
U-235	Uranium-235
U-238	Uranium-238
MeV	Million electron volts
V_G	General surfaces etch rate
V_T	Track etch rate
PC	Polycarbonate
PET	Polyethylene terephthalate
PP	Polypropylene
PVDF	Polyvinylidene fluoride
PI	Polyimides
CR-39	Polyallyl diglycol carbonate
St	Styrene
MAA	Methacrylic acid
VP	N-vinyl pyrrolidone
2M5VP	2-methyl 5-vinyl pyridine

CHAPTER I

INTRODUCTION

In the past several years, various new microporous membranes and filters have been developed for use in various fields, viz. health, medicine, air pollution, beverage industries, development of microtubules, materials science characterization, etc. These filters are generally made from polymeric materials. Price and Walker [1] first discovered a technique leading to the development of track-etched microfilters. They found that latent tracks forming on a material caused by ionization as charged particles are traveling through could be revealed by chemically etching to form cylindrical pores. Since the early 1970s, track microfilters were available on the market. Since then, much development has been done. Today, the methods for producing latent tracks in polymer films have two basic methods. The first is based on irradiation with fragments from a fission reaction of a heavy nucleus such as californium or uranium. The second is based on irradiation with ion beams from an accelerator.

In this research, fission fragments from uranium-235 were used to produce latent tracks in polycarbonate films in a nuclear reactor. The irradiation by fission fragments was chosen to produce the latent tracks because it was cheaper to do so in Thailand as we has a nuclear reactor which can be utilized immediately. Flow-through pores were generated by chemically etching with basic solutions. The resulting values of average pore density and pore diameter were characterized against the preparation conditions.

Objectives

1. To prepare polycarbonate microfilters by nuclear tracking-chemical etching technique.

2. To characterize the as-prepared polycarbonate microfilters for their effective average pore diameter, pore density, water permeability, and mechanical properties.

Scope of the investigation

In the preparation of polycarbonate (PC) microfilters by nuclear tracking-chemical etching (NTCE) method, PC films were bombarded with fission fragments from nuclear reactions of uranium-235 with thermal neutrons, generated by a nuclear reactor. The exposure time was varied. Latent tracks formed by the bombardment were enlarged to form flow-through channels or holes across the film thickness by chemically etching in sodium hydroxide (NaOH) solutions. In order to observe the effects of etching conditions on the average pore size, pore density, water permeability, and mechanical properties of the as-prepared PC microfilters, concentration of NaOH, etching time, and etching temperature were varied. The pore geometry, average pore size, and pore density were characterized by optical microscopy (OM) and scanning electron microscopy (SEM) techniques. Fourier transformed infrared spectroscopy (FT-IR) technique was used to observe any change in chemical functional groups, which might occur during nuclear tracking or chemical

etching step. The as-prepared PC microfilters were also tested for water permeability and their mechanical integrity was also tested.

CHAPTER II

THEORETICAL AND LITERATURE REVIEWS

2.1 Technique for preparation of microfilter

Four common techniques for preparing porous polymeric microfilters are 1) expanded film [1], 2) template leaching [2], 3) phase inversion [3-4], and 4) irradiation [5-9]. Most of these techniques have been developed based on the nature of types of polymers used as a raw material. In addition, each technique results in microfilters of diverse characters (i.e. average pore size, uniformity of the pore size, porosity, mechanical properties, and even selectivity towards permeate species).

Expanded film technique [1] is used for preparing expanded film microfilters, which are made from crystalline polymers by an orientation, and stretching process. In the first step of the process, a highly oriented film is produced by extruding the polymers at a temperature close to their melting points, followed with a very rapid drawdown. After cooling, the film is stretched the second time, up to 300%, at right angles to the original orientation of the polymer crystallites. The second elongation deforms the structure of the crystallites to produce slit-like voids of 20 to 250 nm in width between adjacent crystallites. Expanded film membranes had relatively poor tear strength along the original orientational direction.

Template leaching [2] offers an alternative way for preparing microfilters from incompatible blends of a polymer and a leachable component. First, an incompatible blend of a polymer and a leachable component is casted into film. After the film is formed, the leachable component is removed by a suitable solvent (for that particular component) and a porous film then results. The leachable component could be a soluble, low molecular weight solid or liquid, or even a polymeric material such as polyvinyl alcohol or polyethyleneglycol. The same general method is used to prepare microporous glass. In this case, a two-component glass melt is formed into sheets or small tubes, after which one of the components is leached out by extraction with an alkaline solution. This technique is suitable for preparing microfilters from polymers, which do not dissolve in common organic solvent.

Phase inversion [3-4], also known as solution precipitation or polymer precipitation, is the most important asymmetric microfilters preparation method. In this process, a clear polymer solution is precipitated into two phases: a solid polymer-rich phase that forms the matrix of the membrane, and a liquid polymer-poor phase that forms the microfilter pore. The microfilters prepared by this technique are usually used in ultra filtration and reverse osmosis. This technique is unsuitable for preparing porous polymeric microfilters from polyolefin.

Lastly, nuclear tracking-chemical etching (NTCE) technique [5-9] is use for preparing track-etched microfilters. The NTCE technique comprises two steps. First, a polymer film is irradiated with fission fragments produced by a nuclear reaction between uranium-235 and thermal neutrons in a nuclear reactor. The high-energy

fragments induce polymer chains in the vicinity of their passage to break, leaving behind sensitized or damaged tracks or latent tracks. The latent tracks are then enlarged in an etching solution, which merely dissolves away polymer molecules along the latent tracks, which causes the flow-through channels or pores to form across the thickness of the film. Both the exposure time in the nuclear reactor and the etching time in the etching solution of varying conditions affect the pore density and average pore size.

In the present study, NTCE technique is use to made track-etched microfilters from polycarbonate (PC). This is because, on one hand, the availability of a nuclear reactor at the Office of Atomic Energy for Peace (OAEP) and because of the flexibility of the technique in controlling the average pore size and pore density.

2.2   Nuclear fission [10-12]

2.2.1   Uranium

Uranium is a heavy metal, which can be used as an abundant source of concentrated energy. It is gray, hard, and very dense (18.7 g/cm^3) metallic element. It melts at 1,133 °C. It reacts with air and water to produce a brown, dense (11 g/cm^3) oxide compound, UO_2, which melts at 2,800 °C. The chemistry of uranium is extremely complex, and its compounds may have several degrees of oxidation. In addition to being radioactive, they are toxic. Uranium metal takes on different crystal structures at different temperatures. It is orthorhombic at room temperature but resorts

to a triclinic structure at 660 °C, resulting in a marked difference in the lattice volume. This transformation precludes its use at temperatures higher than 600 °C.

Uranium in nature is a mixture of three isotopes: uranium-238 (99.276% and decaying half-life of 4.5 billion years), uranium-235 (0.718% and decaying half-life of 713 million years), and uranium-234 (0.0056% and decaying half-life of 248,000 years). All three of them are radioactive because they emit alpha particles, which are in fact helium nuclei, but they are only slightly radioactive because of their exceedingly long half-life. Uranium undergoes spontaneous fission reactions; however, its spontaneous fission half-life is so long as to be barely measurable, viz. 5 million billion years for uranium-238 and 30 million billion years for uranium-235.

Uranium-238 is the father of a various other radioactive elements. Its descendants include uranium-234 and lead-210. Other members are radium with a half-life of 1,600 years, radon with a half-life of 3.8 days, and several others. Another family, this time descending from uranium-235, has a stable isotope of lead as a final descendant.

There are two steps for extraction of uranium. First, the ore is rough-crushed, sorted according to radioactivity, fine-crushed, and processed by acid or carbonate leaching as indicated by the nature of the ore. Leaching dissolves the uranium, which is then recovered from the solution either by organic solvents or ion-exchange resins. Second, the uranium is then re-extracted and precipitated by chemicals in the form of ammonium diuranate, sodium diuranate, or magnesium diuranate. The resulting

concentrate is bright yellow, hence the term yellowcake, which contains 75% of uranium. This yellowcake can react with thermal neutrons in a nuclear reactor to produce fission fragments.

2.2.2 Fission fragments

Fission fragment is a fragment of an atomic nucleus coming from a fission reaction. Fission fragments are therefore widely used in the calibration and testing of detectors intended for general applications to heavy ion measurements. All heavy nuclei are, in principle, unstable against spontaneous fission reactions into two lighter fragments; however, the process is inhibited by the larger potential barrier energy that must be overcome in the distortion of the nucleus from its original near-spherical shape. Spontaneous fission is therefore, not a significant process except for some transuranic isotopes. Each fission gives two fission fragments, which by conservation of momentum, are emitted in opposite directions. Because the normal physical form for a spontaneous fission source is a thin deposit on a flat backing, only one fragment per fission can escape from the surface, while the other fragment is lost by absorption within the backing. The fission fragments are medium-weight positive ions. The fission is predominately asymmetrical so that the fragments are clustered into a light group and a heavy group. The fragments appear initially as positive ions for which the net charge approaches the atomic number of the fragment, but as they are slowed down by interacting with matter through which they pass, additional electrons are picked up, reducing their effective charge. The energy shared by the two fragments is

about 200 MeV. The distribution of the energy is also asymmetrical with the light fragment receiving the greater fraction.

2.2.3   Nuclear fission reaction

A fission reaction may take place in any of the heavy nuclei after their capturing of a neutron. However, low-energy (slow or thermal) neutrons are able to cause fission only in those isotopes of uranium and plutonium whose nuclei contain odd numbers of neutrons (e.g. uranium-233, uranium-235, and plutonium-239). Thermal fission may also occur in some other transuranic elements whose nuclei contain odd numbers of neutrons. For nuclei containing an even number of neutrons, fission can only occur if the incident neutrons have energy above about one million electron volts (MeV).

The probability for a fission or any another neutron-induced reaction to occur is described by the cross-section for that reaction. The cross-section may be imagined as an area surrounding the target nucleus and within which the incoming neutron must pass if the reaction is to take place. The fission and other cross-sections increase greatly as the neutron velocity reduces. Hence, in nuclei with an odd-number of neutrons, such as uranium-235, the fission cross-section becomes very large at thermal energies.

The mechanism for a nuclear fission reaction can now be explained, using uranium-235 as the model system. When a thermal neutron is captured within the

nucleus of uranium-235, the total energy is distributed amongst the 236 nucleons (protons and neutrons) being present in the compound nucleus. This nucleus is relatively unstable and is likely to break into two fragments of around half the mass of the original nucleus. These fragments are nuclei found around the middle of the Periodic Table and the probabilistic nature of the break-up leads to several hundred possible combinations. Creation of the fission fragments is followed almost instantaneously by emission of a number of neutrons (typically 2 or 3 with the average number being 2.5), which enable chain reactions to be sustained (see Figure 2.1).

Figure 2.1   The process of nuclear fission.

The number of neutrons and the specific fission products from many fission events are governed by statistical probability, in that the precise break up of a single nucleus cannot be predicted. However, conservation laws require that the total number of nucleons and the total energy to be conserved. The fission reaction in

uranium-235 produces fission products such as Ba, Kr, Cs, I and Xe with atomic

masses distributed around 95 and 135 (see Figure 2.2).
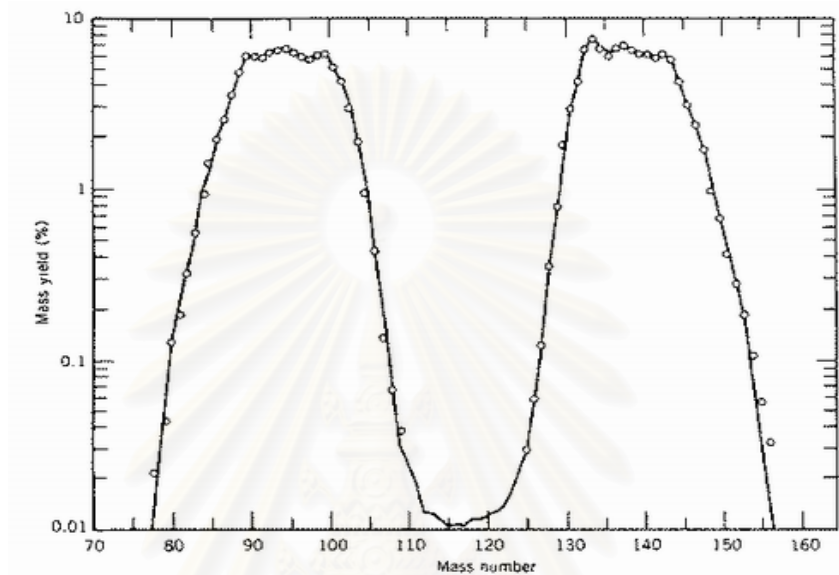
Figure 2.2   Atomic mass distribution of fission products.

Typical reaction products are, for examples,

$$\text{URANIUM-235} + n \rightarrow \text{Ba-144} + \text{Kr-90} + 2n + \text{energy}$$

$$\text{URANIUM-235} + n \rightarrow \text{Ba-141} + \text{Kr-92} + 3n + 170 \text{ MeV}$$

$$\text{URANIUM-235} + n \rightarrow \text{Zr-94} + \text{La-139} + 3n + 197 \text{ MeV}$$
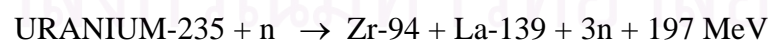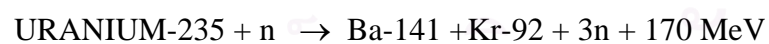
In such equations, the number of nucleons is conserved, e.g. 235 + 1 = 141 +

92 + 3, but a small loss in atomic mass may be equivalent to the energy released. Both

barium and krypton isotopes subsequently decay and form more stable isotopes of

neodymium and yttrium, by emitting several electrons from the nucleus in the

process. It is beta decays, with some associated gamma ray, which make the fission products highly radioactive. This radioactivity decreases with time. The total energy released in fission varies with a precise break up, but averaging about 200 MeV or 3.2 x 10^{-11} juole per uranium-235 fission. Fission products from uranium-235 have different atomic masses so it has different energies. The momentum law suggests that when atomic mass increases, the energy of the fission products will decrease. The energy of fission products is averaging about 64.1 and 93.1 MeV (see Figure 2.3).
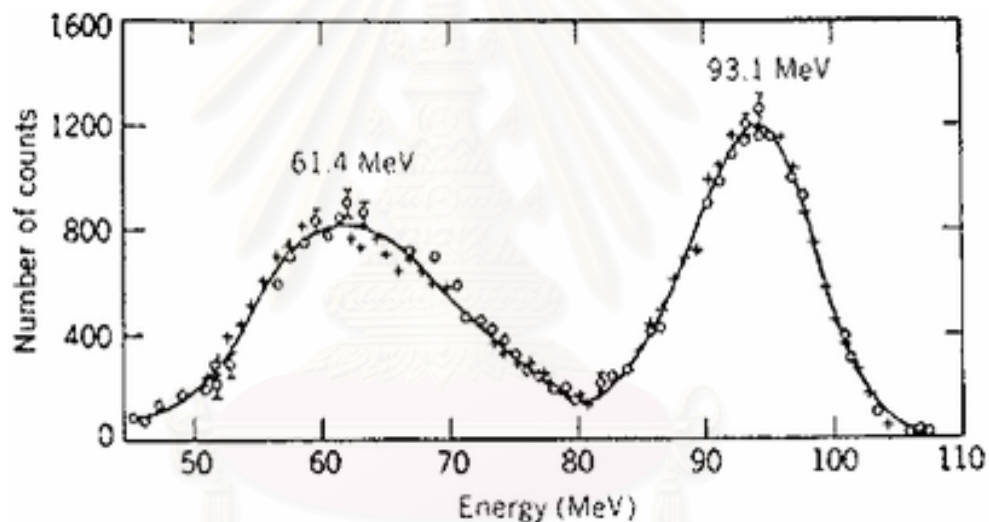
Figure 2.3   Energy of fission fragment of uranium-235.

2.3   Nuclear tracking-chemical etching (NTCE) method [13-15]

Use of NTCE method for producing microfilters was proposed almost immediately after the discovery of the nuclear tracking phenomenon in thin sheets of

materials by Fleischer et al., 1964. The technique has been adopted by the industry soon after the discovery. Since the early 1970s, track-etched polycarbonate (PC) microfilters have been available on the market. Basic information on the technique and the targeted applications for these microfilters are presented in the following paragraphs.

2.3.1   Nuclear tracking

The most plausible theory for latent track formation in polymers is based on a radiochemical damage mechanism. Etchable tracks are formed by radiolytic scission of long polymer chains into shorter fragments and production of reactive, low molecular weight radiolytic products which are more easily to dissolve by an etchant solution than are the surrounding, undamaged bulk molecules (see Figure 2.4).
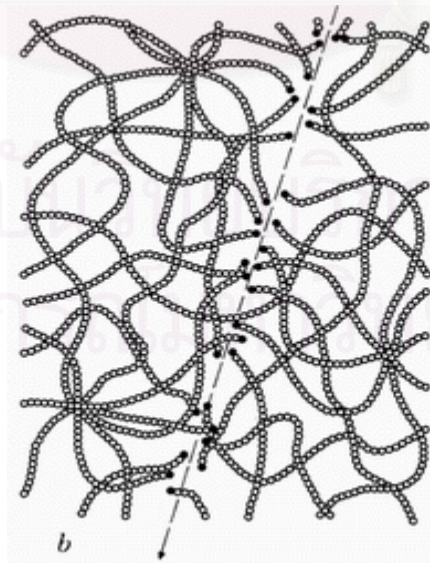
Figure 2.4   The latent track formation phenomenon in polymers.

There are two basic methods for producing latent tracks in polymer films, which can later transformed into porous polymeric microfilters. The first method is based on irradiation of polymer films with fragments from a fission reaction of heavy nuclei such as californium or uranium [16]. For examples, exposing a uranium target to a neutron flux from a nuclear reactor initiates the fission reactions of uranium-235. The fission fragments coming from a thin layer target have an almost isotropic angle distribution leading to an array of latent tracks in the films.
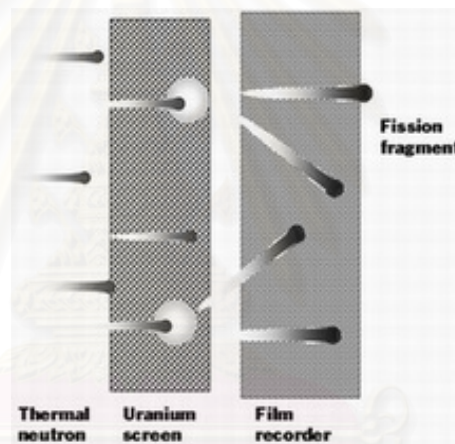
Figure 2.5   The latent track formation by fission fragments from uranium-235.

The advantages of the fission fragment tracking are, for examples,

(i)   good stability of a particle flux in time

(ii)   a non-parallel particle flux (enables the production of microfilters with

a high porosity and low percent of overlapping pore channels)

(iii)   relatively low cost

The limitations of the method are, for examples,

(i)   contamination of the tracked foil with radioactive products ("cooling" of the irradiated material is needed)

(ii)   a limited range of the fission fragments = limited thickness of the microfilter

(iii)   limited possibilities for creating various angle distributions of pore channels

(iv)   fragments of different masses and energies produce tracks with different etching properties

The second method is based on the use of ion beams from an accelerator [17-20]. Modern accelerators provide beams of high intensity heavy nucleus ions. The energies of accelerated ions are a few MeV per nucleon. The beams can be pulsed or continuous. To irradiate large areas, a scanning beam is normally used.

The advantages of the accelerator tracking method are, for examples,

(i)   no radioactive contamination of the material when the ion energy is below the coulomb barrier

(ii)   identity of bombarding particles = all tracks show the same etching properties

(iii)   higher energy of particles = larger range = thicker foils can be perforated

(iv)   better conditions for producing high-density ($>10^9$ cm^{-2}) track arrays

(v)   particles heavier than fission fragments can be used (U-238, for instance)

(vi)   It is easier to control the impact angle and produce arrays of parallel tracks or create some particular angular distributions for getting rid of merging pores.

The limitations of the method are, for example,

(i)   the stability of the particle flux from an accelerator is usually lower

(ii)   a higher cost of irradiation.
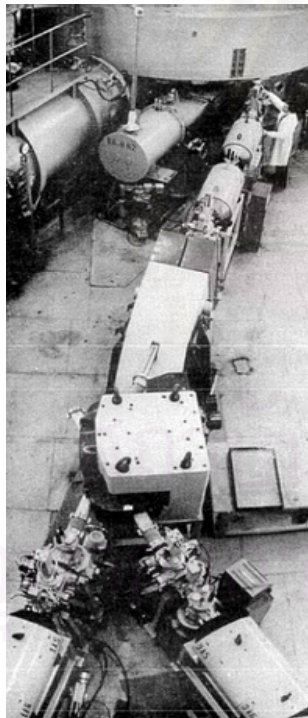
| (a) | (b) |

Figure 2.6   Irradiation source: (a) cyclotron accelerators and (b) nuclear reactor.

2.3.2. Chemical etching

Chemical etching is a process by which the latent tracks can be enlarged. During chemical etching, the damaged, short molecules along the surface of the latent tracks lead are chemically removed by an etchant solution to form larger, flow-through channel. It is the pore-size-determining and pore-shape-determining step of this technique. The simplest description of the pore geometry is based on two parameters: the general surface etching rate V_G and the track etching rate V_T (see Figure 2.7). It is applicable for larger ($\geq 1\mu m$ in diameter) pores. The conical pore shape is transformed into a cylindrical one at when $V_T \geq V_G$.
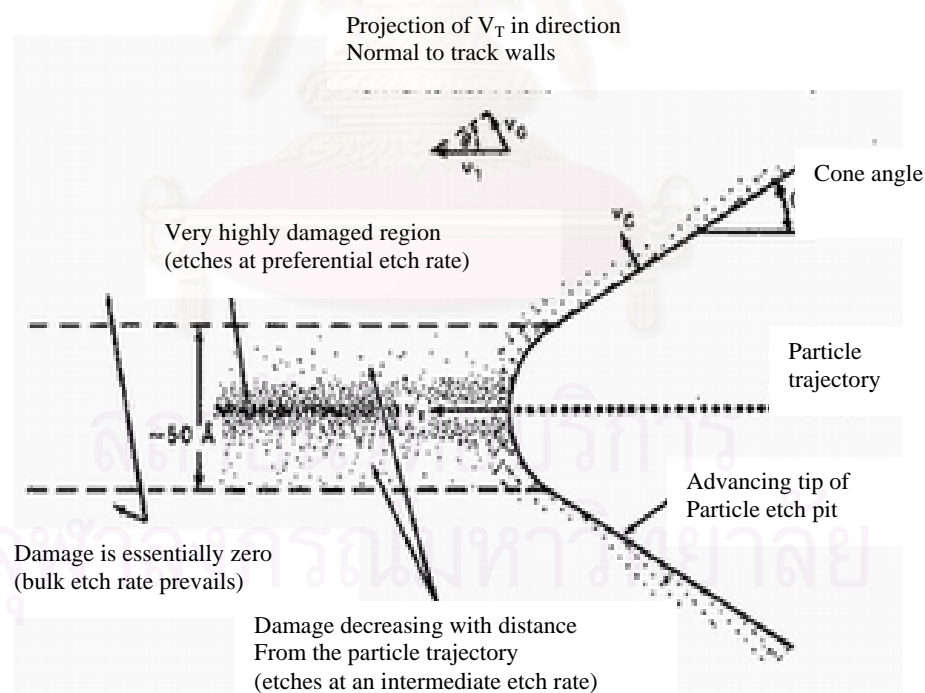
Figure 2.7   Schematic diagram of submicroscopic damage trail along particle track, and proceeding preferential etching.

The general surface etching rate depends on the type of materials used, on the etchant composition, and on the temperature. The track etching rate depends on a much greater number of factors. They can be classified into a few categories: sensitivity of the materials, irradiation conditions, post-irradiation conditions, and etching conditions.

The irradiation conditions include parameters of the bombarding particle, atmosphere (vacuum, presence of oxygen) and temperature [21-22]. In order to achieve reproducible results in subsequent NTCE process, one has to maintain the aforementioned conditions very strictly.

2.4   Properties of track-etched microfilters [23-25]

Track-etched microfilters exhibit a number of properties that give the materials a unique overall performance. Exploiting different combinations of these properties has allowed the development of applications by which track-etched microfilters offer alternatives that other microfilters cannot provide.

2.4.1   Optical transparency

Track-Etched microfilters are usually transparent when the pore size is larger than 5 µm. Microfilters with pore sizes smaller than 3 µm are opaque, although they can be made transparent through a carefully controlled manufacturing process to reduce light diffraction through the porous structure. For some applications in which

light exclusion is required, the microfilters can be dyed, and the pore angles can be controlled to ensure that no direct light can be transmitted through the channels in the microfilters.

2.4.2 Flat and smooth surface

The polymeric films used for track-etched microfilters are inherently smooth: a property that is preserved throughout the microfilter manufacturing process. This smooth, flat surface is ideally suited for microscopy applications, which require a defined distance between the objective and the target. This factor can be especially important where a defined focus must be maintained or topographical defects may interfere with quantification (e.g. epifluorescence). Thickness variations in track-etched microfilters are kept within ±1 µm and the roughness does not exceed 50 nm (measuring from peak to valley).

2.4.3 Pore size, distribution, and shape

The pore size of track-etched microfilters can vary from approximately 20 nm to 14 µm. this wide range of pore sizes allows track-etched microfilters to be used for a multitude of applications. Track-etched microfilters also have very controlled pore size distribution due to the manufacturing process, which means that pore size is usually between –20 and 0% of the stated pore size. The intralot coefficient of variance for pore size is typically 2–3%. In addition to pore size, pore density (or

porosity) can be controlled. It typically ranges from 1 x 10^5 to 6 x 10^8 pores/cm^2, and can vary within certain limits in relation to pore size.

The internal shape of the pores can also be controlled to allow the formation of a truly cylindrical pore structure. An even pore structure is essential for controlled and consistent filtration properties to the membranes—this is particularly true for nanoporous track-etched microfilters (filters with a pore size less than 100 nm).

Although most track-etched microfilters have a structure in which the pores are perpendicular to the microfilter surface, it is possible to create parallel pores with controlled angles to the surface. This is relevant when a light-impervious membrane is required, particularly in applications requiring specialized microscopy. If the pores are at right angles to the surface (otherwise known as normal to the surface), light will pass through the pores and travel through the objective, even if the microfilters is dyed. However, by having pores at an oblique angle to the microfilters surface, no direct light can pass through the microfilters, and hence no signal is seen.

2.4.4   Low reactivity

Track-etched microfilters are manufactured commercially from either polycarbonate (PC) or poly(ethylene terephthalate) (PET), both of which are unreactive to most biological materials. The very low biological activity makes these microfilters well suited for IVD applications in which very low levels of binding or interference are required. The low biological activity of these filters also allows cells

to grow on the filter surface. In addition, the physical stability of the materials in heat and under chemical attack allows them to be included in a number of different device formats and processes.

2.4.5 Thinness

Track-etched microfilters are very thin, typically less than 20 µm, which offers both advantages and disadvantages. The thin nature means that hold-up volume or depth effects is practically non-existent, especially compared with cellulose or glass fiber materials that can show considerable sample retention. However, the very thin nature of the materials can cause handling and processing problems, which may ultimately limit utility in large-scale applications.

2.4.6 Low liquid diffusion rate

Due to the low porosity of the membrane, the liquid flow rate through the filter is very low for small-pore-size materials. Controlling the average pore size and pore density can control this flow rate.

2.5 Some particular polymers for the track-etched microfilter production

2.5.1 Poly(ethylene terephthalate) (PET)

The track-etching process in PET has been reported [26]. PET is rather stable in acids, organic solvents, biologically inert, and mechanically strong. The high etching rate ratio is achievable (when using UV sensitization) which makes it possible to produce microfilters with different pore diameters. The etching procedure is simple and fast. Alkali solutions (sometimes with additives) are used to develop tracks. The microfilters are relatively hydrophilic without any additional modification.

2.5.2 Polycarbonate (PC)

Polycarbonate is the material that has been used for making track-etched microfilters since the early seventies. The production technology is very close to that for PET. Compared with PET, the sensitivity of PC is higher, which makes it possible to produce membranes with pore diameters as small as 0.01 μm and omit the UV sensitization stage. PC microfilters differ from PET microfilters by the lower resistance to organic solvents and the lower wetting ability.

2.5.3 Polypropylene (PP)

PP was investigated as a raw material for membranes to be used for the filtration of some aggressive liquids such as strong alkali solutions or inorganic acids.

Chemical etching in strong oxidizers (e.g. chromium trioxide) should be selected in order to enlarge the latent tracks in PP. The etching procedure is considerably more complex than those for PET and PC. Samples of PP track-etched microfilters with various average pore diameters ranging between 0.1 and 3 μm have been fabricated on a laboratory scale. The microfilters are hydrophobic. A commercial production of PP microfilters has not been launched because of the limited marketability.

2.5.4 Poly(vinylidene fluoride) (PVDF)

Much effort was made to develop track-etched microfilters from fluorinated polymers. The best results were obtained with PVDF which exhibits good chemical, mechanical, and particle registration properties. Researchers are drawn to PVDF due mainly to its piezoelectricity behavior. Samples of PVDF microfilters have been produced and tested in several laboratories [27-28]. Similarly to PP, the etching procedure for PVDF with strong oxidizers creates problems. Normally it takes hours to achieve a pore size in the micrometer range. Commercial production of PVDF track-etched membranes was not set up because it cannot compete with the template-etched PVDF microfilters already available on the market. Many other fluorinated polymers have been studied and etchable tracks have been observed in some of them. However, numerous attempts to obtain porous structure in fully fluorinated polymers by NTCE method have not been successful.

2.5.5 Polyimides (PI)

PI has an excellent stability at high temperatures, excellent mechanical strength, and extraordinary radiation resistance. Films of polypyrromelithimide in various thicknesses have been available for more than 20 years. Research work on PI as a track-recording medium has been performed in several laboratories [29-30]. The possibility for track-etched microfilters from PI has been experimented. A few successful recipes for track-etching in PI have been reported. NaClO and H_2O_2 provide a rather high etching rate ratio enabling one to produce a porous membrane of relatively good quality. However, instability of the etchants in time at high temperatures is a serious problem during chemical etching.

2.5.6 Poly(allyl diglycol carbonate) (CR-39)

CR-39, a highly sensitive to ionizing particles and highly transparent material, can be used to fabricate track-etched microfilters for special purposes [31]. Some copolymers of CR-39 have been shown to exhibit even higher sensitivity than CR-39 itself [32]. The polymer is inherently brittle which may pose some problems during manufacturing.

2.6 Some prospectively uses for track-etched microfilters

2.6.1 Flow controller

The combination of pore size and pore density gives a track-etched microfilter its flow characteristics. With accurate control of these properties, a smooth surface, and good thickness control, track-etched microfilters are suitable for applications in which the flow rate of a liquid through the track-etched microfilter needs to be controlled. A filter's ability to control flow rate is particularly applicable in biosensors, where liquids must be in contact with the sensor for a certain time, as well as in flow-through assays and lateral-flow tests.

2.6.2 Cell capture

Since track-etched microfilters are very accurate in their filtration characteristics, they can be used in cell-capturing applications. These applications would allow for clearer identification of marked cells in a number of formats. The retention of cells on the membrane surface allows cells to be stained and observed in a very clear environment. Any sample debris or contaminants can be washed through the pores of the microfilters away from the viewing area. This improved resolution and accuracy could have applications in any area of clinical chemistry in which cells are currently observed. The reduction in the likelihood of a false diagnosis would also have a significant impact, especially in large-scale screening procedures.

Another application is in the pharmaceutical and food industries, where detecting pathogenic bacteria in water is a crucial issue. Track-etched microfilters can be used in those automated systems in which bacteria are filtered onto the microfilters and marked by a photo-labeling marker. The system then enumerates the number of bacteria with a measurement technique such as flow cytometry. The well-defined pore size helps to select the targeted bacteria (e.g. *escherichia coil* or *cryptosporidium*), and the cleanliness associated with filters helps to achieve a zero false-positive detection rate. New developments for this application include specially dyed and metal-coated membranes that show low reflection spectra in the green and red wavelengths.

2.6.3 Biosensors

Until mid-1990s, due to technological constraints, track-etched microfilters with pore sizes of less than 100 nm were not precise in terms of pore size or pore shape, and they often appeared as hourglass or bottle-shaped structures instead of true cylinders. However, recent developments have led to almost perfect control of the shape and manufacturing of true cylindrical-shaped pores. Enhanced techniques in the irradiation process, subsequent etching, and quality control allow the production of high-quality track-etched microfilters to be used as biosensors. These recent developments open the door to precise applications in which there is a strong desire for accurate flow control or diffusion properties.

Since these recent improvements provide accurate flow control or diffusion properties, the enhanced performance has helped in biosensor applications in which the microfilter acts as a barrier to biological molecules and controls their flow to the sensor. Microfilters also serve as barriers to many potential contaminants, improving the assay's specificity. In those applications involving the presence of biochemical reagents to measure the reaction, the pores can be filled with the desired materials (e.g. antigens or enzymes). The complete biosensor can therefore be dried onto the microfilter.

2.6.4   Filtration application

Filtration is the process of removing physically suspended matters from a given volume of liquid or gas by forcing the material through a porous barrier or filter. This facilitates the extraction and analysis of the material separated from the liquid or gas. The filtration efficiency is defined as the ability of the media to distinguish between particles of different specific sizes. Track-etched microfilters have advantages over the other conventional microfilters due to their well-defined pore size, which makes it possible to remove all particles bigger than its pore size. The removal efficiency depends upon the average pore size and pore size distribution of the microfilter and also on the diameter of the pollutant.

2.6.5 Development of metal and metal-semiconductor

Fabrication of microstructures is being considered a potential technology not only for use in micromechanics and microelectronics but also in studies pertaining to behavior of materials at micro and nano levels. Microstructures comprising microdimensional devices, fibrils, wires, cones, tubules and whiskers have invited attention for uses in multidisciplinary areas.

2.6.6 Particle immunofiltration assay

The health test blood typing card by Akers Laboratories Inc. (Thorofare, NJ) uses the precise pore size distribution in track-etched microfilters to perform analyses of blood types in whole blood samples (see Figure 2.8). A bead with a diameter smaller than the pores of microfilters is mixed with the patient sample. If no antibodies are present, the bead passes through the pore, and can be seen in a capture zone. However, if the sample has antibodies against the antigen, the antibodies attach to the particle surface, a matrix of particles is formed, and no migration is seen. The success of the assay depends on the pore size distribution in the track-etched microfilter, where any significant variation would result in either all particles or no particles passing through the microfilters.
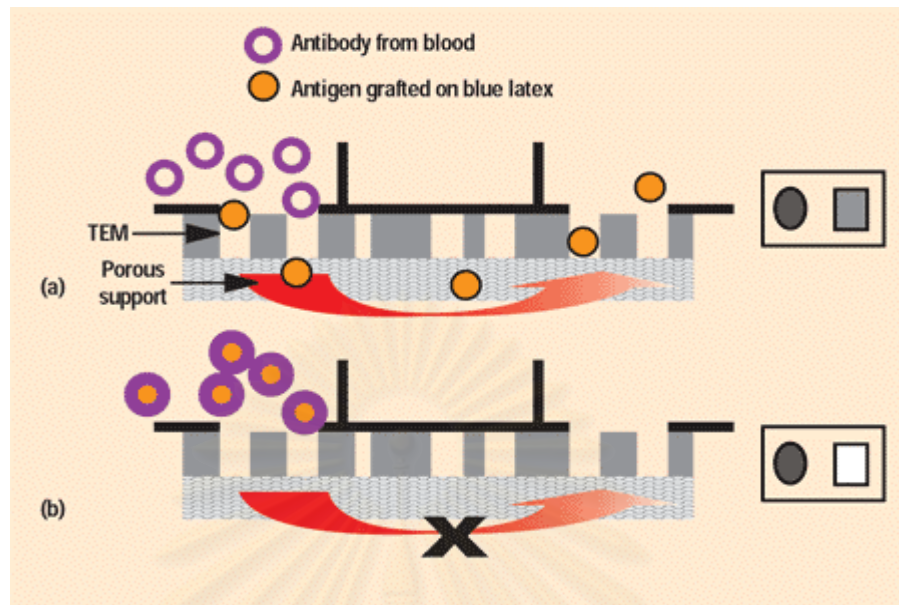
Figure 2.8 The Health Test blood typing card by Akers Laboratories Inc. (Thorofare, NJ) is a particle immunofiltration assay that employs track-etched membranes. The test is negative when the complex antigen/dyed bead can pass through the pore and migrate from one well to the other (a). The test is positive when the size of the agglutinate antibody with complex antigen/dyed latex beads does not allow the transfer from one well to the other because of the pore size of the track-etched membrane (b).

2.7 Modification methods

Modification methods that change the surface and bulk properties of track-etched microfilters or impart new functions to them are, for examples,

    (i)   sorption of some small or large molecules on the surface;

    (ii)   surface treatment with plasma;

(iii)   immobilization of functional substances on the surface by covalent binding using chemical reactions;

(iv)   graft polymerization of various monomers.

Hydrophilization of track-etched PET and PC microfilters by adsorption of poly(vinyl pyrrolidone) (PVP) is a widely used procedure in the industry. Similarly, covering the surface with silicone oil or paraffin can make the microfilters hydrophobic. Plasma treatment improves wettability of many polymers due to the formation of new polar groups on the surface. Stability of the modified surface significantly depends on the condition of the plasma treatment [33]. Electrochemical properties of track-etched pores can be modified by covalent binding of charged groups or by adsorption of ionic polyelectrolytes [34]. The immobilization of amino acids to track-etched PET microfilters based on the reactions of the end carboxyl and hydroxyl groups was reported [35]. However, the surface density of the immobilized species, in this manner, is rather low.

The radiation-induced graft polymerization onto track-etched microfilters is an alternative process, which has been studied in more detail [36-37]. Styrene (St), methacrylic acid (MAA), N-vinyl pyrrolidone (VP), 2-methyl 5-vinyl pyridine (2M5VP), N-isopropyl acrylamide (NIPAAM), and some other monomers have been grafted onto PET microfilters. Grafting of St increases the chemical resistance and makes the microfilter hydrophobic. MAA and VP were grafted onto microfilters to increase wettability, which is especially important, when aqueous solutions are filtered through small-pore membranes.

2.8   Literature reviews

Komaki and Tsujimura [38] studied the growth of fine holes in poly(ethylene naphthalate) (PEN) film which were irradiated by fission fragments from uranium-235 in nuclear reactor. Subsequently, they used 3 to 5N NaOH solutions at 45 to 65 °C to etch the irradiated films. They measured the hole diameters and hole densities of the as-prepared filters by a gas flow apparatus. They concluded from their results that the effective hole diameters were about 100 to 1000 Å and the hole densities were approximately 10^8 hole/cm^2.

Four years later, Komaki [39] studied the growth of fine holes after chemical etching of irradiated poly(vinylidene fluoride) (PVF) films. He investigated the effects of etching conditions on the growth of the holes in the irradiated films. He found that potassium hydroxide solutions were more effective in etching the films than were sodium hydroxide solutions. However, at high concentrations, the depositions of crystallites were observed and the etching rate decreased with increasing concentration.

Guillot and Rondelez [40] investigated characteristics of pores obtained by chemical etching of track-etched PC microfilters. The latent tracks were formed by irradiating 60 μm-thick PC films with high-energy krypton ions (500 MeV, Kr^{25+}) from an accelerator. 2N NaOH was used as the etchant. From conductivity studies, the ion tracks were present in three different domains: a highly damaged core of radius 50 Å with 10^4 Å/min etching rate, an intermediate zone of radius 500 Å with 0.9 Å/min

etching rate, and an outer region with 0.47 Å/min etching rate equal to that of the undamaged material.

Cui et al. [41] studied the production of nuclear track-etched microfilters and their applications. They produced 15 μm polycarbonate and 10 μm polyester track-etched microfilters by bombarding with fission fragments from uranium-235 and then etching the tracked films by 6N NaOH solution. They used SEM to investigate the pore geometry present in the obtained microfilters. They found that most of the pores were cylindrical with the pore sizes ranging from 0.2 to 12 μm and the pore densities ranging from 10^5 to 10^8 pore/cm^2.

Samoilova and Apel [42] found that solutions of barium hydroxide were effective in etching nuclear tracked PET films. In their study, they used Xe ions generated by a cyclotron to bombard PET films. They also compared the etching efficiency of barium hydroxide solutions with that of sodium hydroxide and sodium carbonate solutions. They found that barium hydroxide solutions provided much higher surface etching rate due possibly to changes in the boundary layer structure at the polymer surface caused by the divalent positive ions.

Wanichapichart et al. [43] prepared track-etched PC microfilters by bombarding 6 μm-thick PC films with alpha particles generated by a nuclear reactor. They varied bombardment and etching periods to produce a variety of microfilters and tested the obtained microfilters for their water permeability. They found those bombardment PC films with flux of neutrons of 6.5×10^8 neutron. $m^{-2}.s^{-1}$ and etching

the tracked films in 6 N NaOH at 85 °C for 5 minutes produced microfilters with a hydraulic permeability for water of 158×10^{-10} $m^3.N^{-1}.s^{-1}$, which is comparable to that reported for a commercial Millipore microfilters.

Sun et al. [44] studies swift heavy ion-induced amorphisation and chemical modification in polycarbonate. They used 15.14 MeV/amu Xe^{136} and 11.4 MeV/amu U-238 in the power range of 7.6 to 17.1 keV/nm and the fluence range of 9×10^9 to 1×10^{12} ions/cm^2. They investigated chemical degradation of the function groups of PC by FTIR spectroscopy. FTIR results revealed that PC suffers serious degradation after irradiation. Morover, they found that some aromatic ether and methyl group was damaged and produced alkyne groups after irradiation.

Ione et al. [45] designed a new experimental apparatus for porous track-etched microfilter production. They installed this apparatus near the core of the IEA-R1 nuclear reactor at IPEN-Sao Paulo. An uranium sample was deposited on a rod located at the centre of an evacuated aluminium chamber for producing fission fragments. They reported that, with this apparatus, large sheets (ca. 1000 cm^2) of track-etched microfilters having high level of pore uniformity can be prepared and the reactor time could be as short as few minutes. The average pore size was found to be around 15 nm and the pore density was found to be 8.0×10^8 pore/cm^2 .

CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1 Polycarbonate (PC) films

Polycarbonate (PC) films were melt-extruded using a chill roll line (Labtech Co., Ltd, Thailand). The average thickness of the cast film is 13 μm.

3.1.2 Fission plate

Fission plates used to generate fission fragments when being bombarded by thermal neutrons were made from yellow cake or ammonium diurinate (($NH_4)_2U_2O_7$). Cellulose acetate was used as a backing material for ammonium diurinate. The dimension of each fission plate was 2 by 6 cm.

3.2   Chemicals

Other chemicals used in this study are listed in Table 3.1.

Table 3.1   Chemicals

Materials	Grade	Supplier
Sodium hydroxide	Analytical grade	Fluka
Ethanol	Analytical grade	Fluka
Acetone	Analytical grade	Fluka

3.3 Instruments

The instruments used in this study are listed in table 3.2.

Table 3.2   Instruments

Instrument	Manufacturer	Model
Thai Research Reactor–1/Modification1	GA	Triga mark3
Fourier transform infrared spectrometer	Nicolet	Impact 4.1
Tensile testing machine	Lloyd	LR10K
Digital thickness gauge	Mitutoyo	
Microscope	Olympus	BH-2
Scanning electron microscope	JEOL	JSM-6400
Digital camera	Olympus	CAMEDIA C-200Z

2.4 Flow chart of experiment

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                          ┌──────────────┐
                          │   PC film    │
                          └──────┬───────┘
        ┌────────────────────────┼────────────────────────┐
┌───────────────┐      ┌──────────────────┐      ┌──────────────────────┐
│  Am/Be source │      │ Beam tube source │      │ Thermal column source│
└───────┬───────┘      └──────────────────┘      └──────────┬───────────┘
        │                                          ┌─────────┴───────────┐
        │                                          │ Etching at 6N NaOH  │
        │                                          │  70 °C  60 min      │
        │                                          └─────────┬───────────┘
        │                                          ┌─────────┴───────────┐
        │                                          │     Microscope      │
        │                                          └─────────────────────┘
```

Vary etching temperature (50-80 ^{0}C)	Vary etching time (30-150 min)	Vary etching concentration of NaOH (2-8N)

Microscope

PC microfilters
(0.4-10 μm)

Characterisation	Mechanical properties

SEM

thickness

FTIR

Water permeability test

Tensile test
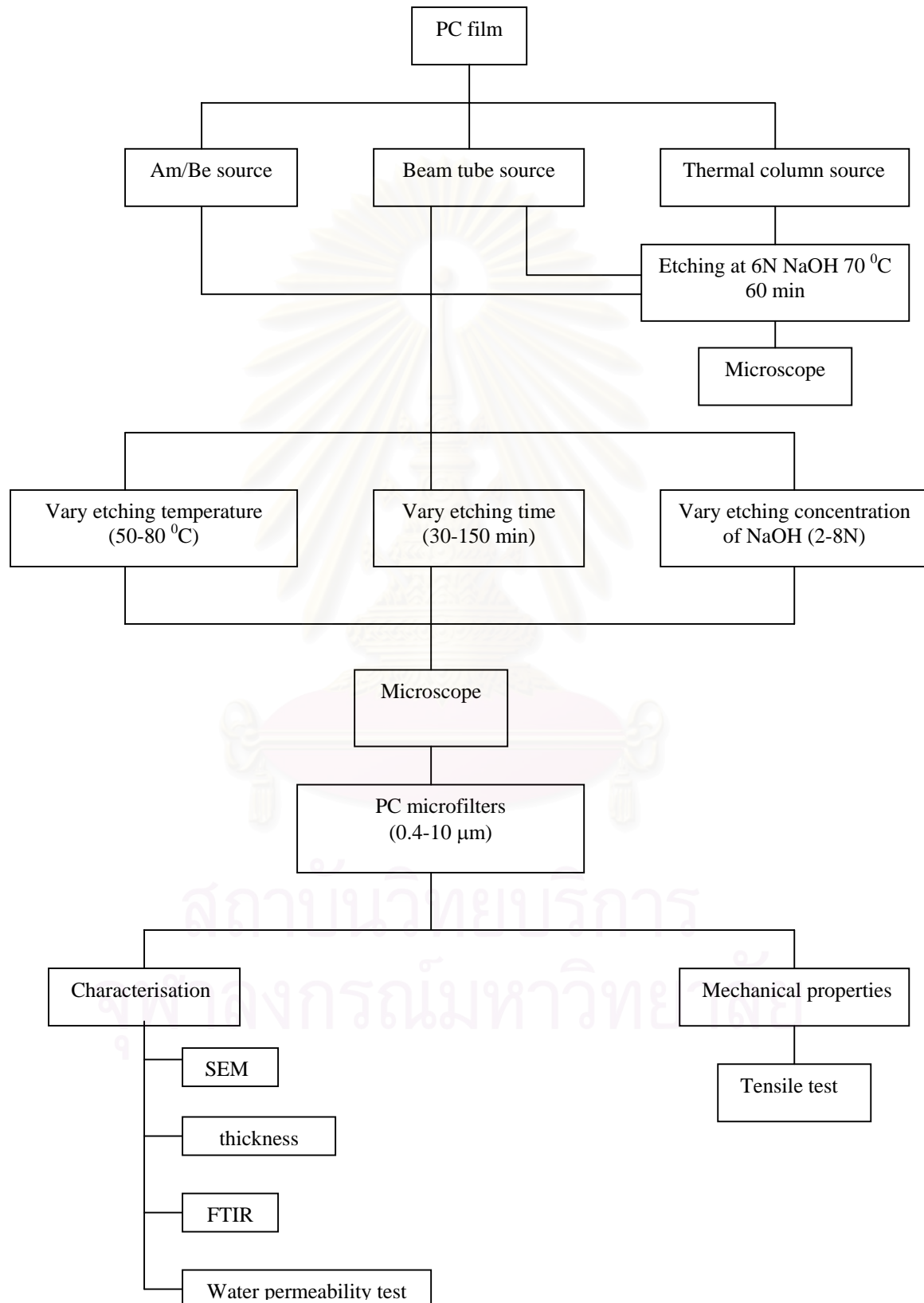
3.5   Procedures

3.5.1   Formation of latent tracks in polycarbonate (PC) films

PC microfilters were first prepared by irradiating PC films with fission fragments from nuclear reactions of uranium-235 and thermal neutrons in the only nuclear reactor of Thailand, Thai Research Reactor–1/Modification1 (TRR-1/M1), located at the Office of Atomic Energy for Peace (OAEP), Thailand. When a thermal neutron is captured in the nucleus of uranium-235, the energy is dissipated amongst the 236 nucleons, which causes the nucleus to be energetically unstable. Due to the instability of the nucleus, it is likely to break off into two fragments of equal masses. These high energy fragments can penetrate thin PC films to create an array of latent tracks in the films.

In this study, three thermal neutron sources have been tested: they are Am/Be source, beam tube, and thermal column.

3.5.1.1   Am/Be source

A 2.5×8 cm^2 PC film was laid side by side with a fission plate, composed mainly of yellow cake (i.e. a uranium rich compound). The specimen was then irradiated with thermal neutrons generated by Am/Be source, which is capable of generating the neutron flux of about 3.63×10^4 neutron/s/cm^2. In this experiment, only the exposure time was varied (i.e. 24, 48, and 72 hours, respectively). The irradiated

films were later etched in 2N sodium hydroxide (NaOH) solution, with the etching temperature being 70°C and the etching time being 60 minutes in order to enlarge the radiation-induced damaged tracks.

3.5.1.2 Beam tube

A 2.5×8 cm^2 PC film was laid side by side with a fission plate, composed mainly of yellow cake (i.e. a uranium rich compound). The specimen was then irradiated with thermal neutrons the beam tube of the TRR-1/M1 reactor. The films were first irradiated at different placement distance of 70, 80, 90, and 100 cm, which has neutron fluxes of about 1.09×10^6, 1.05×10^6, 9.16×10^5 and 8.24×10^5 neutron /s/cm^2 for 60 minutes. To investigate the effect of irradiation exposure time, the films were irradiated at the placement distance of 70 cm for 60, 120, 180, and 240 minutes. The films were later etched in 6N NaOH solution, with the etching temperature being 70°C and the etching time being 60 minutes in order to enlarge the radiation-induced damaged tracks.
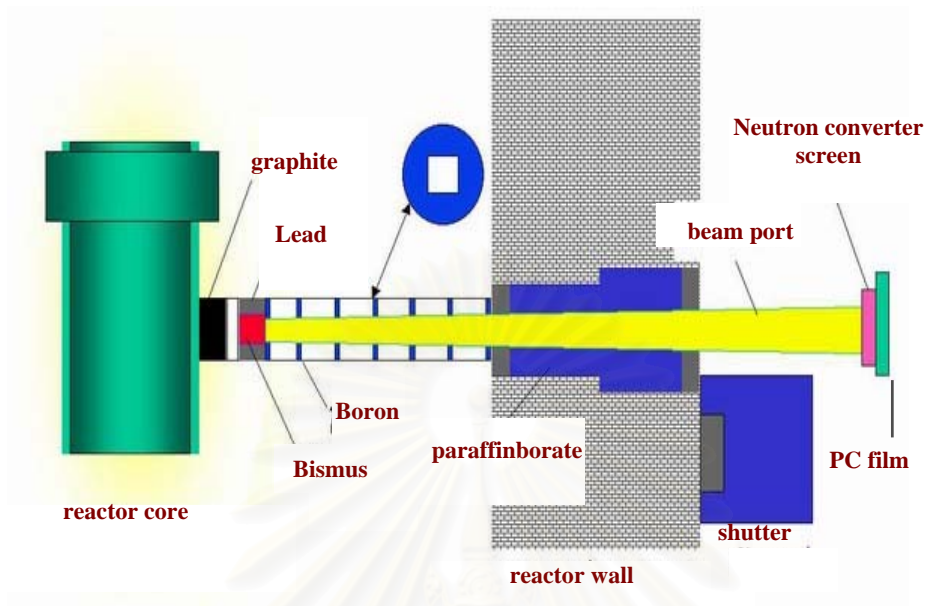
Figure 3.1 Schematic of beam tube at the nuclear reactor.

3.5.1.3 Thermal column

A 2.5×8 cm^2 PC film was laid side by side with a fission plate, composed mainly of yellow cake (i.e. a uranium rich compound). The specimen was then irradiated with thermal neutrons the thermal column of the TRR-1/M1 reactor, with the measured neutron flux being 2.51×10^{10} neutron /s/cm^2. Due to the very high neutron flux of the thermal column, the irradiation exposure time was varied for 3, 5, 7, and 9 seconds. The irradiated films were later etched in 6N NaOH solution, with the etching temperature being 70°C and the etching time being 60 minutes in order to enlarge the radiation-induced damaged tracks.
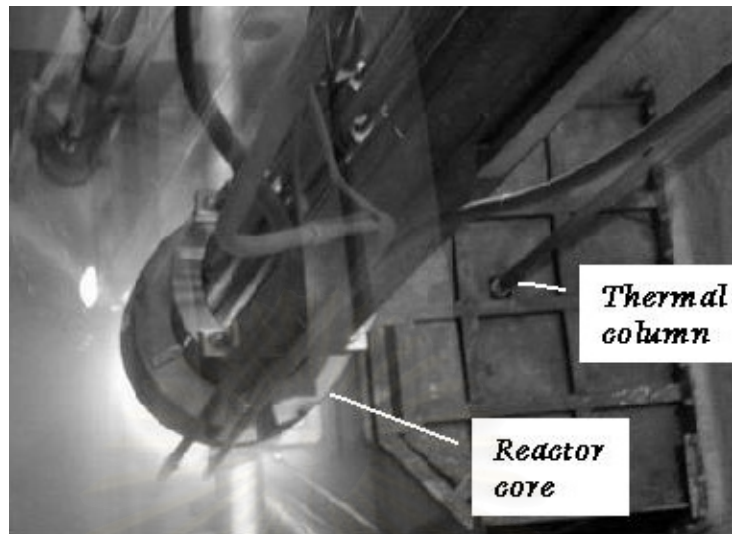
Figure 3.2   Position of thermal column at the nuclear reactor.

3.5.2   Chemical etching experiment

Chemical etching is a process to enlarge the damaged tracks formed during the tracking step into flow-through pores. The irradiated PC films were etched in NaOH solutions having various initial concentrations (i.e. 2, 4, 6, and 8 N). The etching temperature and etching time were also varied (i.e. 50, 60, 70, and 80°C for the etching temperature and 30, 45, 60, 75, 90, 105, 120, and 150 minutes for the etching time, respectively).

3.6 Characterization of track-etched PC microfilters

3.6.1 Optical microscope (OM)

The average numbers of pores per unit area and the average pore diameter were studied by an Olympus BH-2 optical microscope, using the eyes piece lens of $10\times$ and objective lens of $40\times$ or $100\times$.

3.6.2 Scanning electron microscope (SEM)

A JSM-5800 LV scanning electron microscope (SEM) was used to determine the geometry of pores in the obtained microfilters. Before examination, each specimen was coated with a thin layer of gold to enhance the conductivity of the surface.

3.6.3 Thickness measurement

A Lloyd LR10K digital thickness gauge was used to measure the thickness of both neat PC films and as-prepared PC microfilters.

2.6.4 FTIR measurement

A Nicolet impact 4.1 FTIR was used to characterize the chemical functional groups of the PC films both after irradiation and chemical etching with NaOH. The wave number in the range of 400 to 4000 cm^{-1} was analyzed.

3.6.5 Water permeability test

Water permeability of the as-prepared microfilters was measured at room temperature and at a constant pressure of 1172.43 N/m^4 on a circular area of 0.025 m^2.

3.7 Tensile property measurement

The as-prepared microfilters were tested for their mechanical integrity. Tensile strength, yield strength, Young's modulus, and the percentage of elongation at yield were measured based on ASTM D882 standard test method using a Lloyd LR10K universal testing machine. A load cell of 100 N was used with a 50 mm/min crosshead speed and 250 mm gauge length.

CHAPTER IV

RESULTS AND DISCUSSION

This chapter is organized into six sections. In the first section (i.e. 4.1), latent or damaged track formation in PC films is discussed. The second section (i.e. 4.2) presents the effect of chemical etching condition on average pore diameter and average pore density of the as-prepared PC microfilters. In the third section (i.e. 4.3), the effect of chemical etching condition on the thickness of the PC microfilters is presented. In the fourth section (i.e. 4.4), alteration in the chemical structure of PC microfilters is discussed. The fifth section (i.e. 4.5) presents the mechanical properties, while the sixth section (4.6) is focused on water permeability, of the obtained PC microfilters.

4.1 Formation of latent tracks in PC films

The formation of latent tracks on PC films were produced by irradiating the PC films with fission fragments from a nuclear reaction of uranium-235 with thermal neutrons in a nuclear reactor. Figure 4.1 shows a scanning electron micrograph of a as-tracked PC film. The latent tracks were formed by the fission fragments from the dissociation of uranium-235 once it receives a thermal neutron into its nucleus. These fission fragments are of equal mass and have high energy. High-energy fission fragments can pass through a polymer film, which causes polymer molecules to break and leave sensitized, damaged tracks along their path.
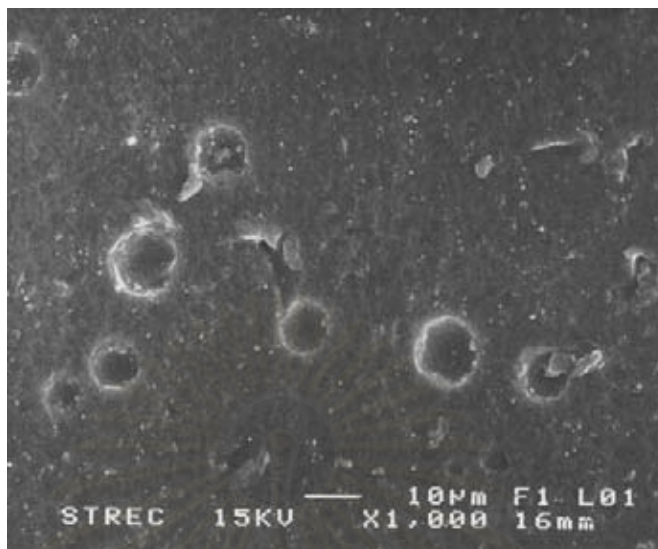
Figure 4.1   Scanning electron micrographs of polycarbonate film, which was tracked by fission fragments from a nuclear reaction of uranium-235 and thermal neutrons at beam tube at the length of 70 cm for 180 minutes.

The density of the latent tracks within a PC film depended strongly on the source of radiation used, or, to be exact, on the neutron flux for each of the source investigated, and on the irradiation time. Three types of radiation source, having different neutron fluxes, were investigated.

4.1.1 Am/Be source

For this source, a mixture of americium and beryllium was used to produce thermal neutrons. The neutron flux achieved was measured and reported by staff members at the Office of Atomic Energy for Peace (OAEP) to be about 3.63×10^4 neutron/s/cm^2.

Table 4.1 shows track density and track flux of PC microfilters. The results obtained suggest that the pore density of PC microfilters depended on irradiation time in the Am/Be source. When the irradiation time increased, the pore density of PC microfilters increased (see Figure 4.2). It indicates that track density of the PC microfilters was found to increase with increasing residence time in the Am/Be source due to the increased number of fission fragments to which the films were exposed. The track flux is defined as the track density of fission fragments per second. For Am/Be source, it was about 0.46 tracks/sec/cm^2, which was found to be lower than those of thermal column and beam tube (see later).

Figure 4.3 shows an optical micrograph of tracked PC films under a 400x. The irradiation time was varied between 24 and 72 hours. Obviously, due to the very low track density, the very low track flux, and the long irradiation time, Am/Be source is not suitable for preparing tracked PC films.

Table 4.1   Track density and track flux for track-etched PC microfilters, which were tracked in Am/Be source in various irradiation times.

Irradiation time at Am/Be (hr)	Track density (tracks/cm^2)	Track flux (tracks/s/cm^2)
24	$4.53 \times 10^4 \pm 3.10 \times 10^3$	0.5
48	$8.74 \times 10^4 \pm 1.51 \times 10^4$	0.5
72	$1.05 \times 10^5 \pm 1.99 \times 10^4$	0.4
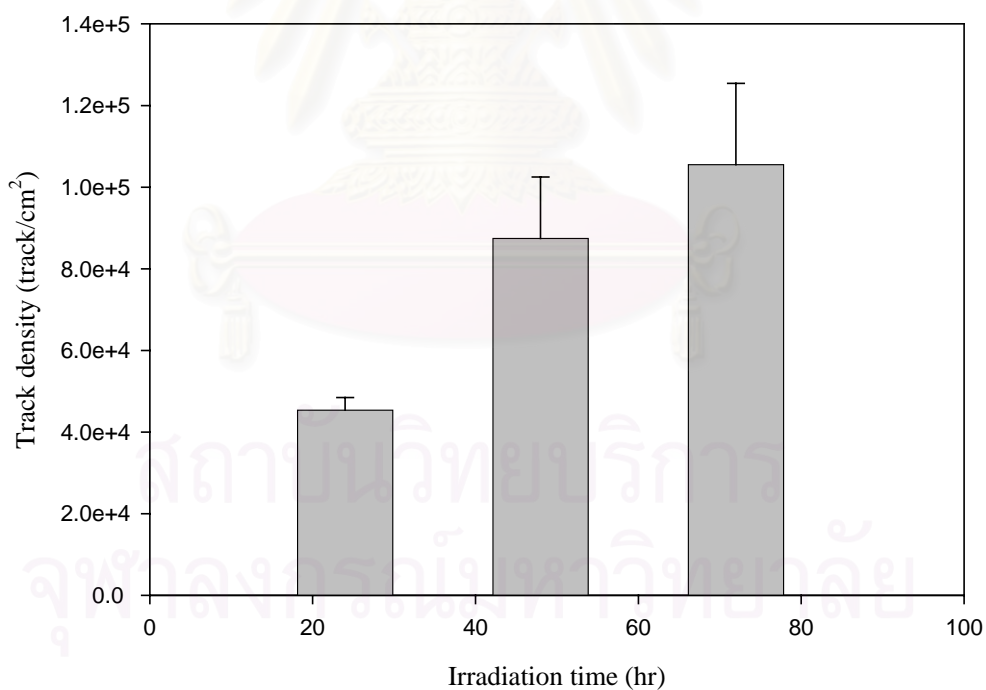
Figure 4.2   Track density of track-etched PC microfilters at Am/Be source as a function of irradiation time.

(a)

(b)

(c)
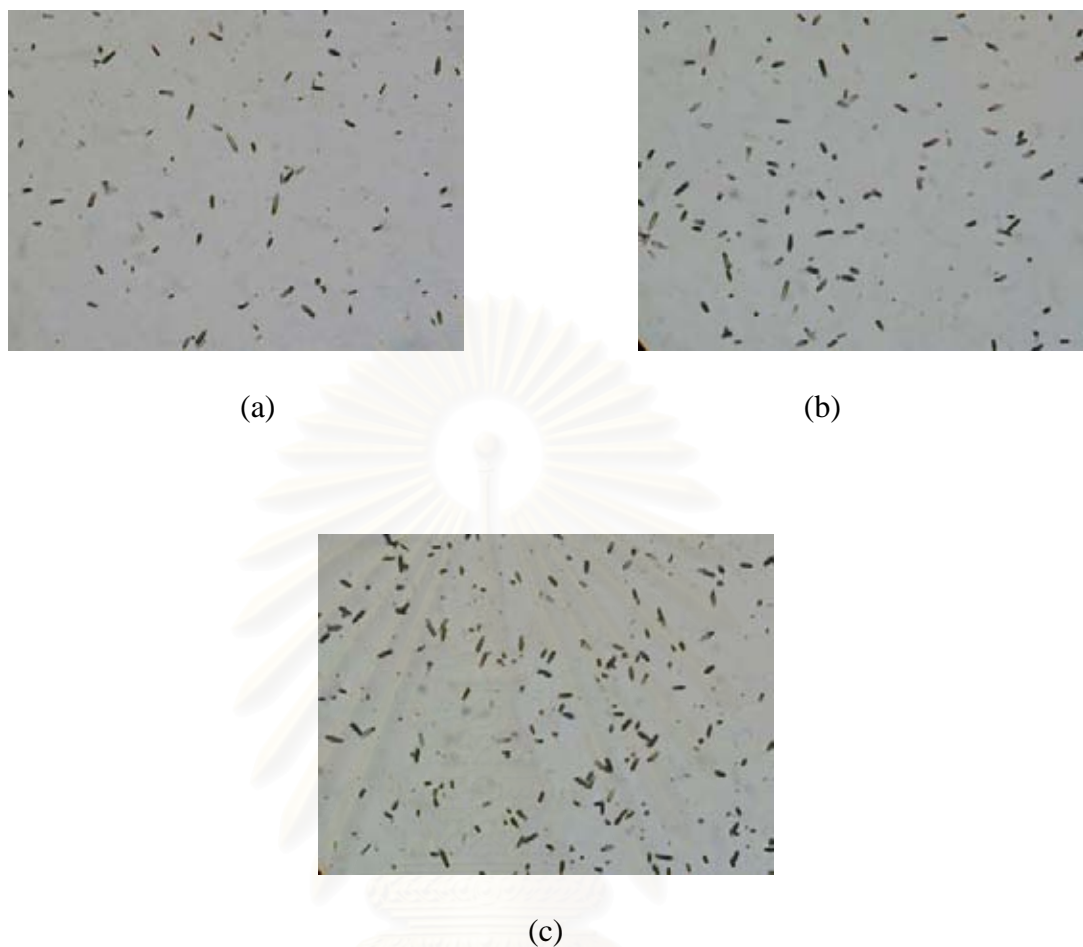
Figure 4.3   Optical micrographs of track-etched PC microfilters, which were tracked in Am/Be source at different irradiation times of (a) 24 hr, (b) 48 hr, and (c) 72 hr and later etched in 6N NaOH at 70 ^{0}C for 60 minutes.

4.1.2   Thermal column source

A thermal column is located close to the reactor core (see Figure 3.2). This source is in nuclear reactor, which can generate thermal neutron for reacting with uranium-235 for forming fission fragment. In this source, it has neutron flux about 2.51×10^{10} neutron/s/cm^2, which is much greater than that of Am/Be and beam tube

sources. Greater neutron flux translates into greater track flux. Table 4.2 shows track density and track fluence of PC microfilters irradiated in thermal column at different irradiation times. Clearly, both track density and track flux were found to increase with increasing irradiation time and the values were much greater than those reported for Am/Be source. Figure 4.5 shows an optical micrograph of tracked PC films under a 40x objective len. From the figure, it is clear that both track density and track flux values are very large, causing adjacent pores to overlap. Even though the values are large, but, the very short exposure time (i.e. within a few seconds) may pose possible problems in the actual production. Thus, the thermal column may not be an appropriate source for making tracked PC films.

Table 4.2   Track density and track flux for track-etched PC microfilters, which were tracked in thermal column in various irradiation times.

Irradiation time-TC (s)	Track density (tracks/cm^2)	Track flux (tracks/s/cm^2)
3	$3.6 \times 10^5 \pm 7.58 \times 10^4$	1.20×10^5
5	$5.75 \times 10^5 \pm 3.81 \times 10^4$	1.15×10^5
7	$7.32 \times 10^5 \pm 5.88 \times 10^4$	1.05×10^5
9	$9.07 \times 10^5 \pm 1.14 \times 10^4$	1.01×10^5
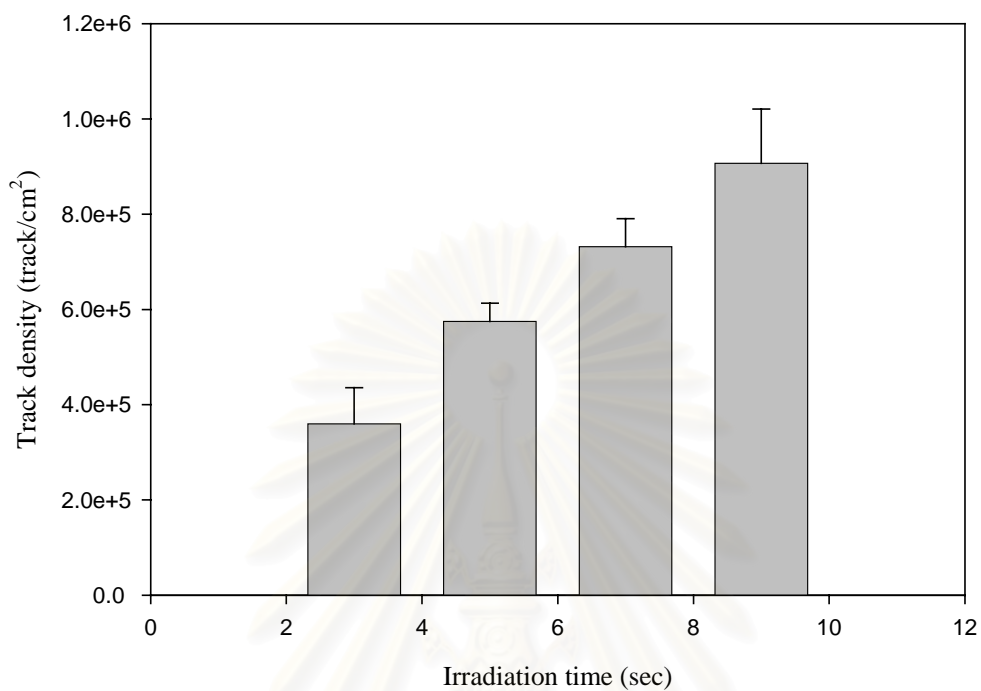
Figure 4.4   Track density of track-etched PC microfilters in thermal column source as a function of irradiation time.

(a)

(b)

(c)

(d)
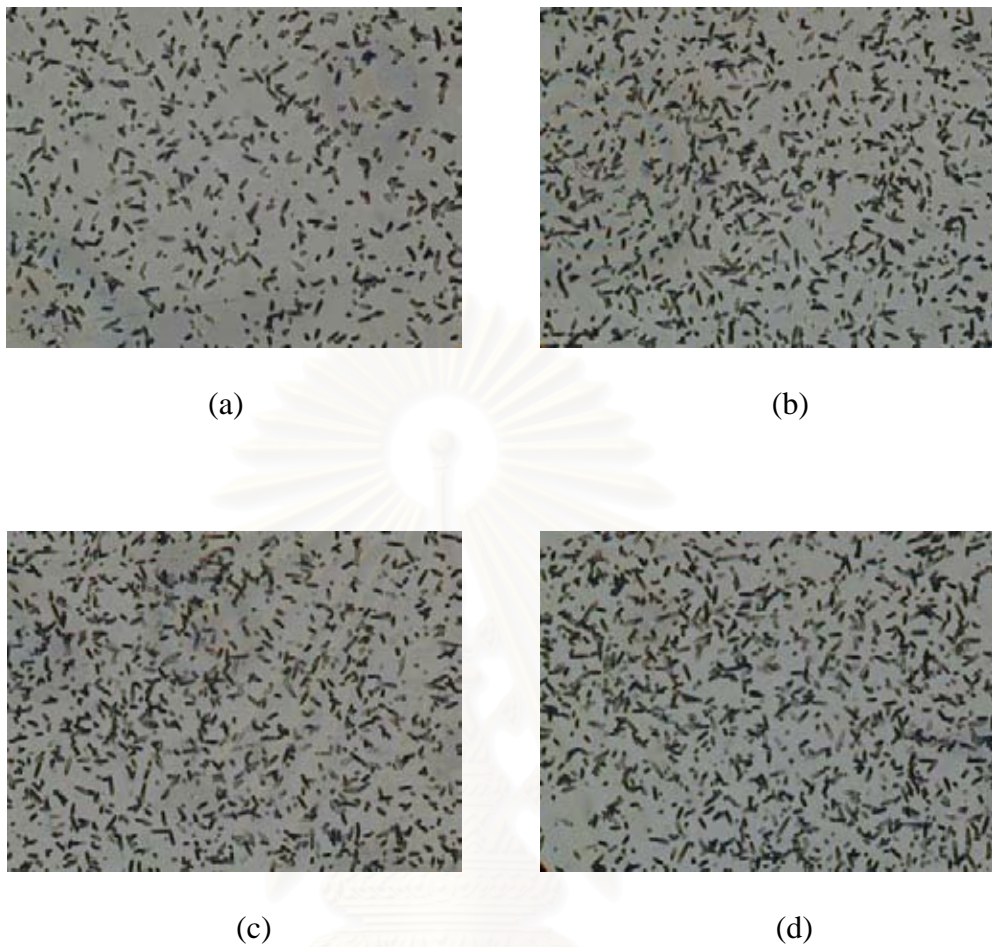
Figure 4.5 Optical micrographs of track-etched PC microfilters, which were tracked in thermal column at different irradiation times of (a) 3 sec, (b) 5 sec, (c) 7 sec, and (d) 9 sec and etched in 6N NaOH at 70 ^{0}C for 60 minutes.

4.1.3 Beam tube source

The last neutron source for preparing PC microfilters is the beam tube source. In the experimental setup, four positions for placing a sample were considered. Each position would have a different neutron flux, since it was placed at a distance away from the reactor core. The neutron flux was found to be a decreasing function of the

placement distance (see Table 4.3). Since neutron flux was found to be a decreasing function of the placement distance, both track density and track fluence on the PC films also exhibited a similar trend, as evident from the data shown in Figure 4.3. Base of the result shown here, the placement distance of 70 cm from the reactor core was found to be the best position in the preparation of the tracked PC films.

Table 4.3   Track density and track flux for track-etched PC microfilters, which were tracked in beam tube in various placement distance.

Placement distance at BT(cm)	Neutron flux (neutron/s/cm^2)	Track density* (track/cm^2)	Track flux (track/s/cm^2)
70	1.09×10^6	$4.96 \times 10^4 \pm 4.85 \times 10^3$	14
80	1.05×10^6	$4.23 \times 10^4 \pm 4.30 \times 10^3$	12
90	9.16×10^5	$3.40 \times 10^4 \pm 8.72 \times 10^2$	9
100	8.24×10^5	$2.99 \times 10^4 \pm 1.01 \times 10^3$	8

* Irradiation time at 1 hour.
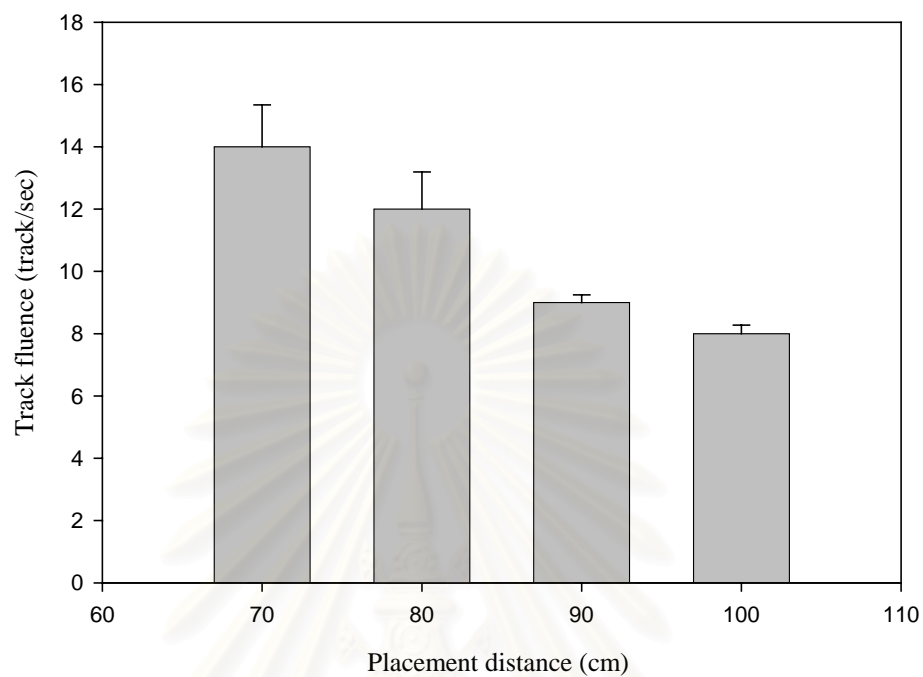
Figure 4.6   Track flux of track-etched PC microfilters at BT source as a function of placement distance.

(a)

(b)

(c)

(d)
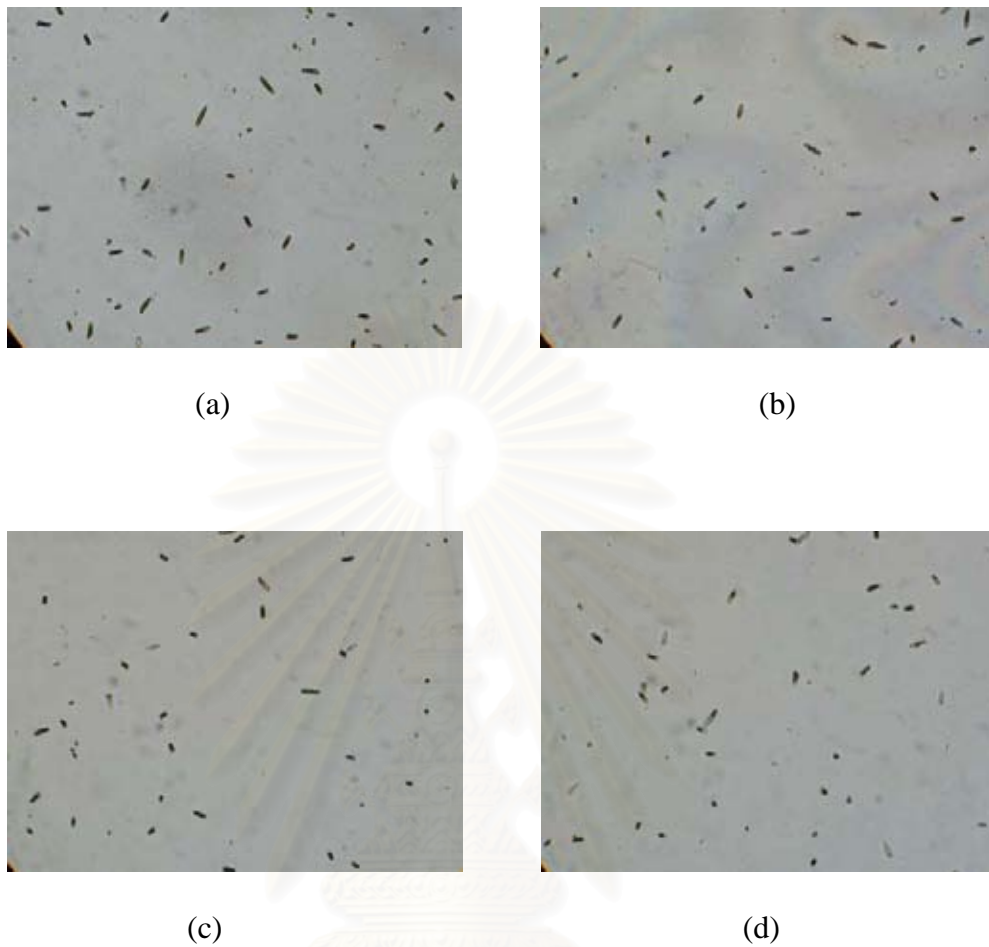
Figure 4.7 Optical micrographs of track-etched PC microfilters irradiated with fission fragment at difference placement distance [i.e. (a) 70 cm, (b) 80 cm, (c) 90 cm, (d) 100 cm] in beam tube source for 60 minute. The etching condition was 6N NaOH at 70 ^{0}C for 60 minutes.

Table 4.4 shows track density and track flux of PC microfilters with different irradiation times in beam tube source. It can be seen that the pore density of the obtained microfilters increased with increasing irradiation time.

Figure 4.9 shows an optical micrograph of tracked PC films under a 400x. From the figure, it is clear that both track density and track flux values were found to increase with increasing irradiation time. At the longest irradiation time investigated (i.e. at 240 minutes), some adjacent pores were overlapped to one another.

According to the results obtained, irradiation at 70 cm for 180 minutes in beam tube source may be the most suitable setup for the preparation of nuclear track-etched polycarbonate microfilters. This setup was chosen based on the facts that, at this condition, there was no pore overlapping and that the values of track density and track flux were reasonable to produce PC microfilters.

Table 4.4   Track density and track flux for track-etched PC microfilters, which were tracked in beam tube in various irradiation times.

Irradiation time-BT* (min)	Track density (track/cm^2)	Track flux (track/s/cm^2)
60	$4.86 \times 10^4 \pm 3.06 \times 10^3$	13
120	$1.02 \times 10^5 \pm 7.14 \times 10^3$	14
180	$1.54 \times 10^5 \pm 1.29 \times 10^4$	14
240	$1.95 \times 10^5 \pm 8.32 \times 10^3$	14 $\pm$

* Irradiation length at 70 cm.
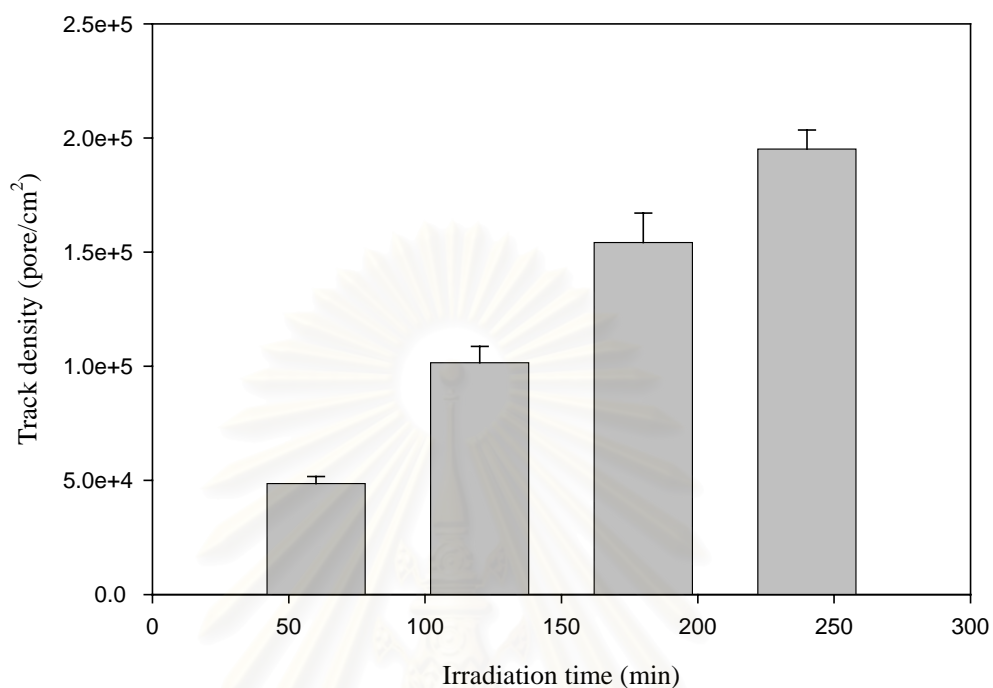
Figure 4.8   Track density of track-etched PC microfilters at beam tube source as a function of irradiation time.

(a)

(b)

(c)

(d)
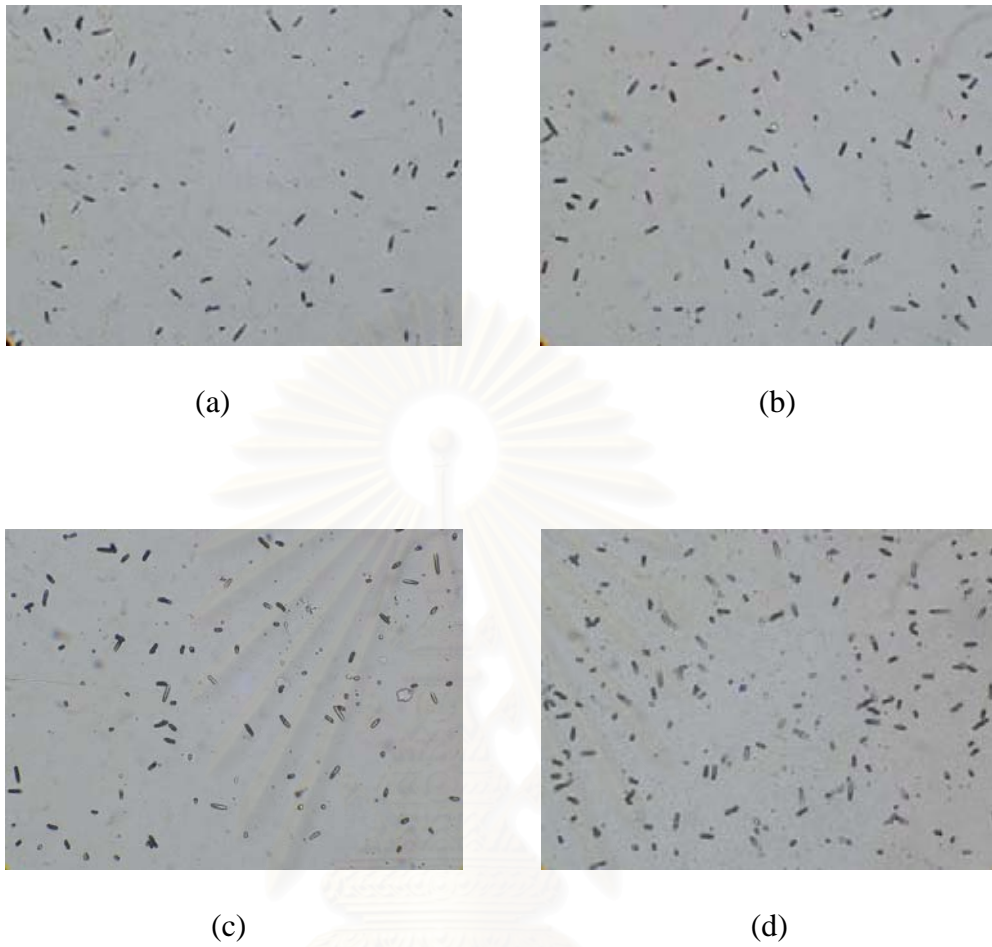
Figure 4.9 Optical micrographs of track-etched PC microfilters irradiated with fission fragment at difference irradiation time in BT source at 70 cm, (a) 60 minute, (b) 120 minute, (c) 180 minute, (d) 240 minute, which etched in 6N NaOH at 70 ^{0}C for 60 minutes.

4.2 Effect of chemical etching condition on track-etched PC microfilters

4.2.1 Effect of chemical etching condition on pore density

In the previous section, it was found that irradiation time and the value of neutron flux affected a great deal to the pore density of the PC microfilters obtained. In this section, the effects of etching conditions such as concentration of the etchant, etching temperature, and etching time on the pore density will be investigated.

Figures 4.10 to 4.13 show the effects of chemical etching conditions on the average pore density for films which were irradiated for 180 minutes in beam tube source at the irradiation distance of 70 cm. 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 mild etching conditions, such as 2N NaOH at 70 °C, the average pore density was found to increase with the etching time. However, the etching time was not found to affect much the average pore density of the films etched in the most concentrated solution. For examples, chemical etching of PC films irradiated for 180 minutes in 8N NaOH at 70 °C at long enough etching time resulted in films having the pore density of 1.50×10^5 pores/cm^2.

On the effect of etching temperature on the average pore density, the average pore density was found to increase with increasing temperature of the etchant. For examples, for PC films etched in 4N NaOH solution for 30 minutes, the pore density

was found to increase from 8.71×10^4 pores/cm^2 at 50 $^\circ$C to 1.42×10^5 pores/cm^2 at 80 $^\circ$C.

The results indicated that fission fragments from uranium-235 were different in their masses and energies to track PC films. Only a fraction of tracks are flow-through tracks, so when etching the irradiated films in mild etching conditions, only a small fraction of tracks, in addition to those flow-through tracks, were enlarged to form additional flow-through channels.
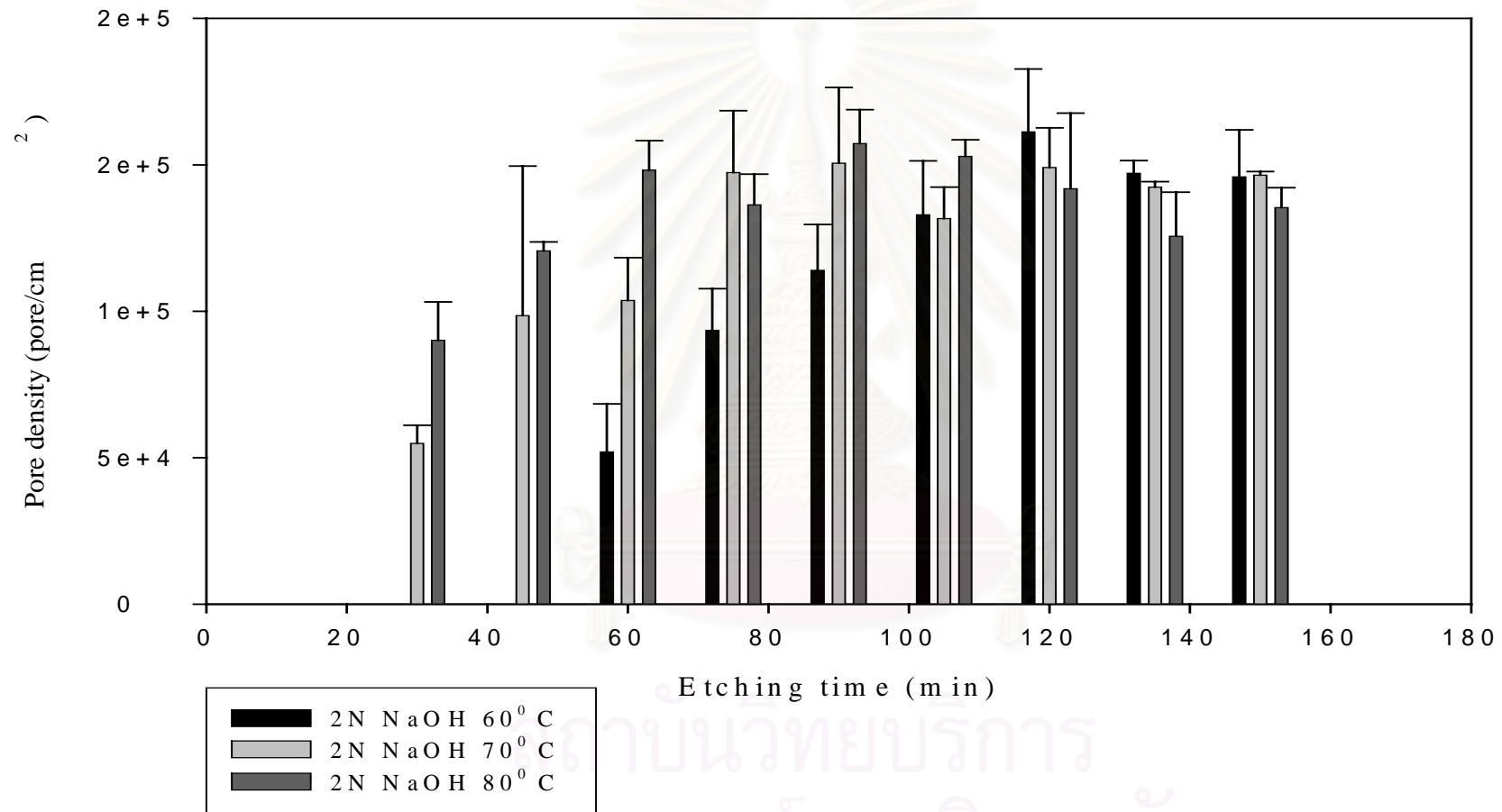
Figure 4.10   Effects of etching conditions on the average pore density of track-etched PC microfilters at 2N NaOH
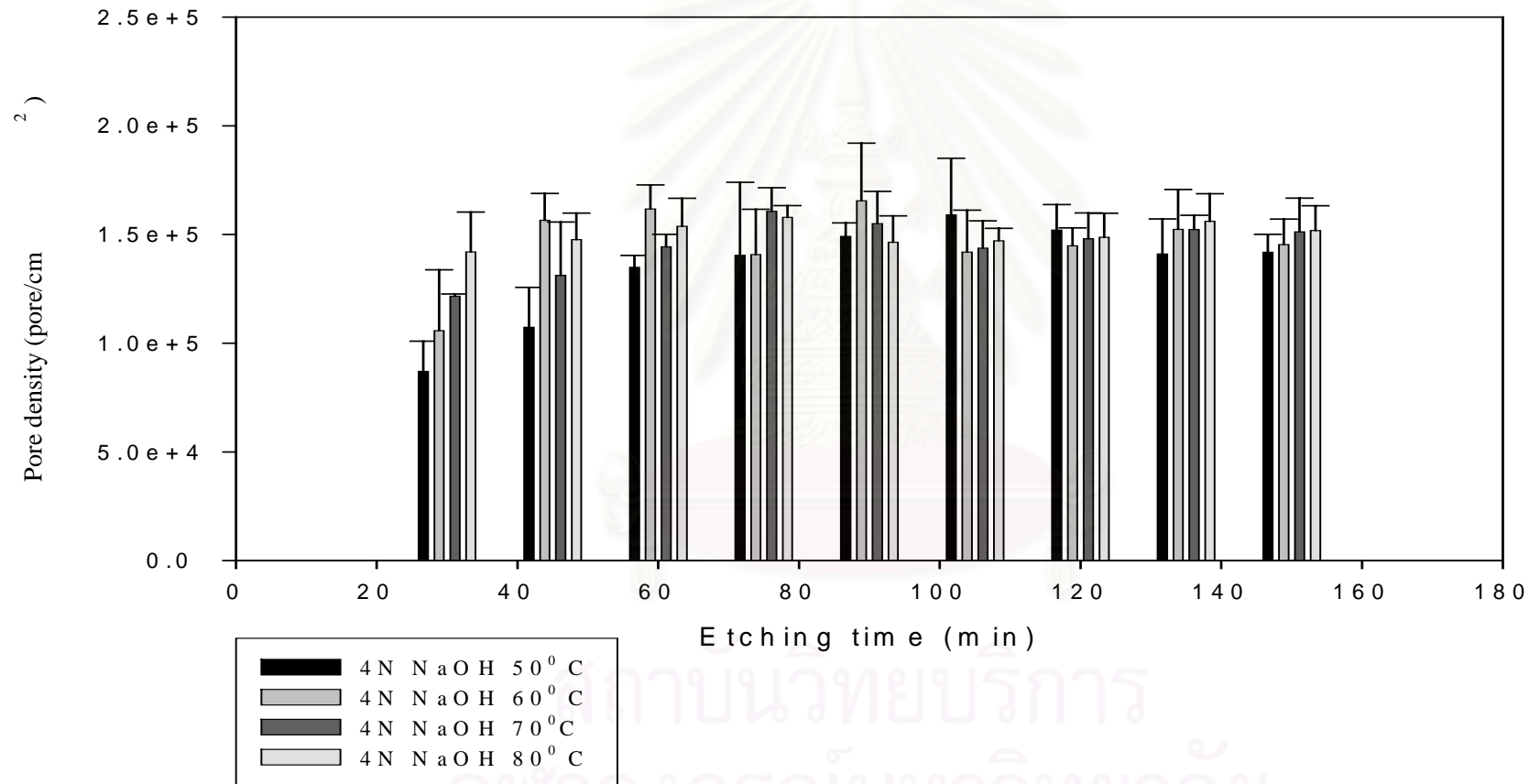
Figure 4.11 Effects of etching conditions on the average pore density of track-etched PC microfilters at 4N NaOH.
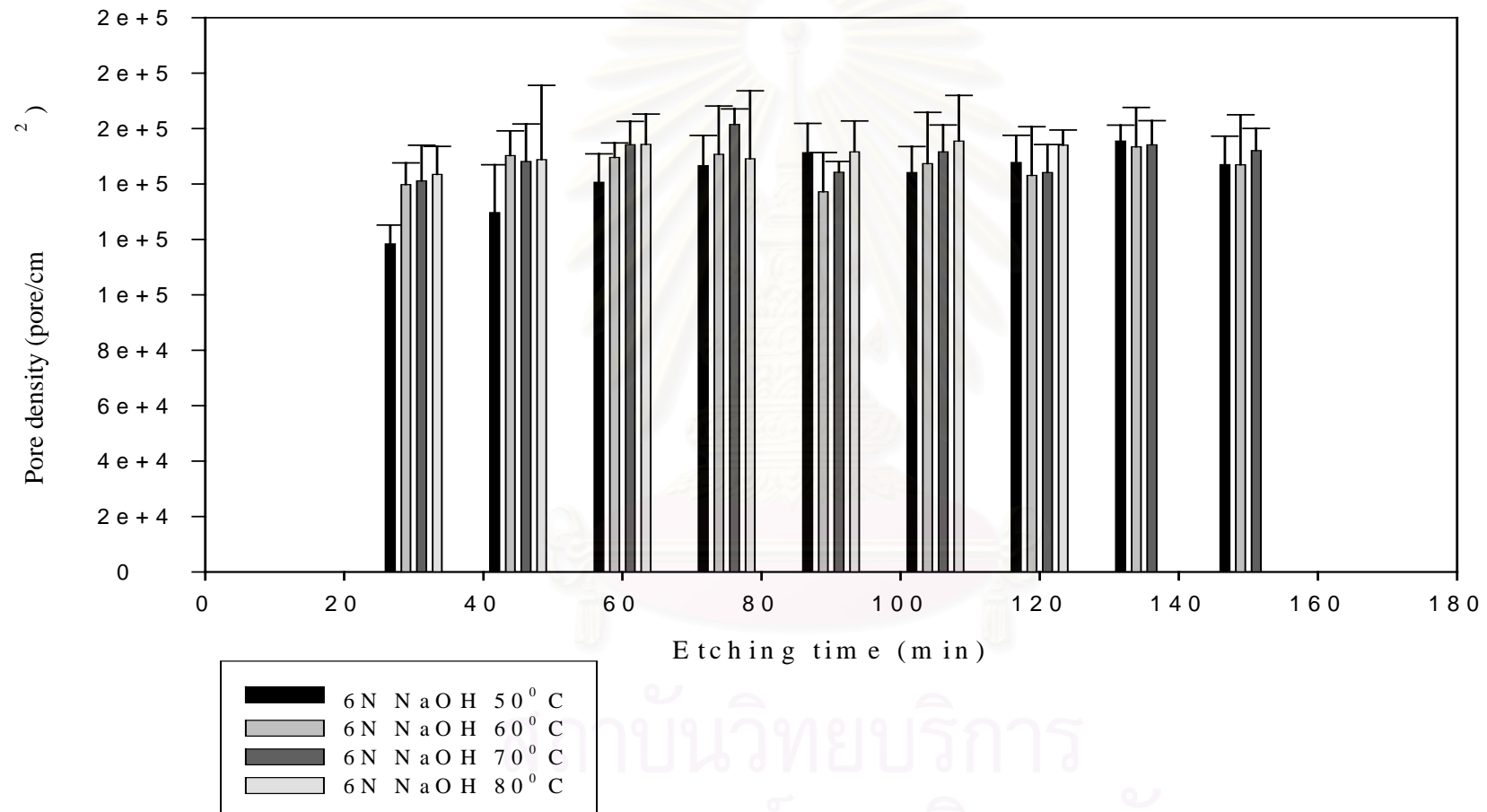
Figure 4.12   Effects of etching conditions on the average pore density of track-etched PC microfilters at 6N NaOH.
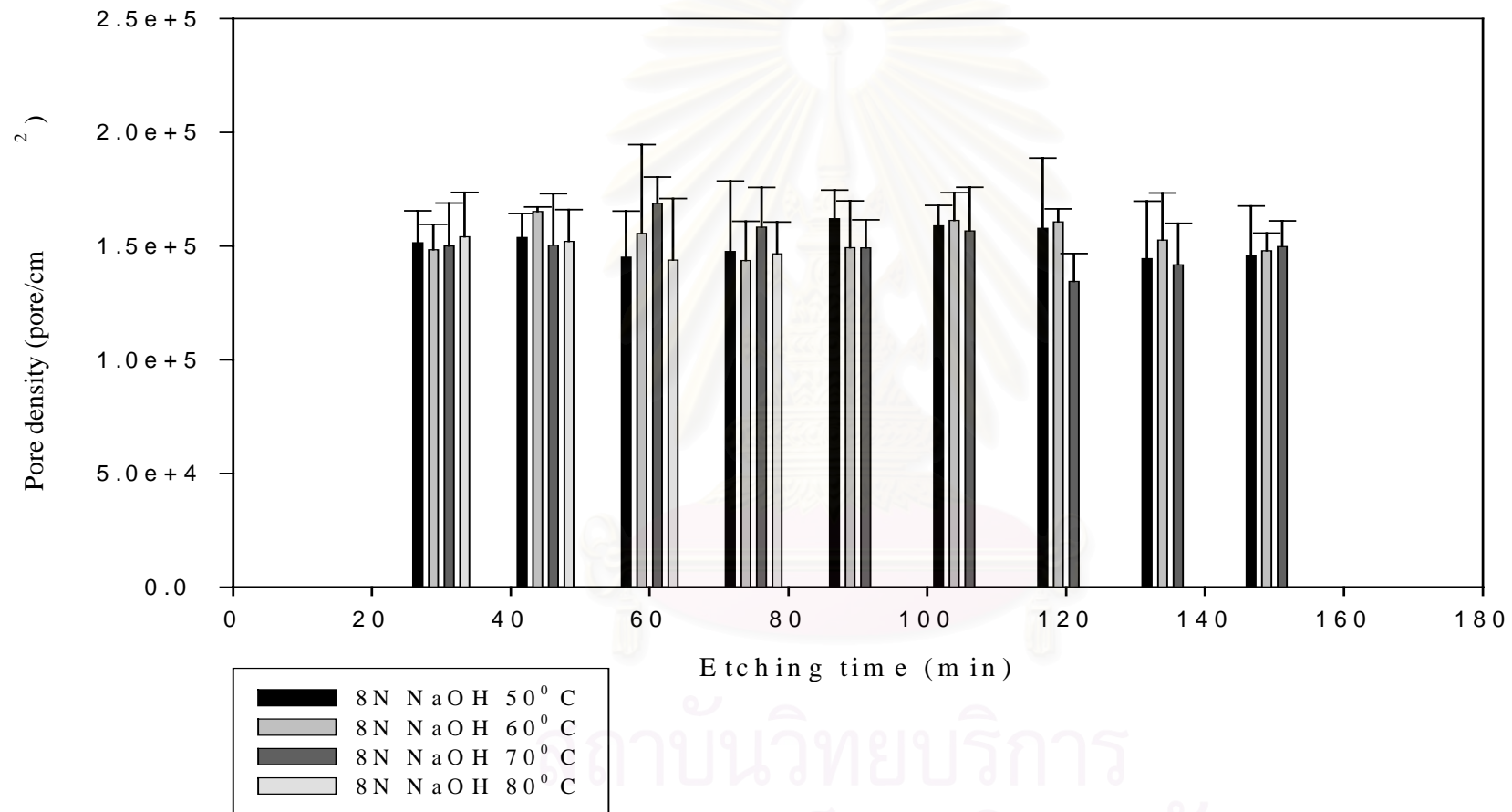
Figure 4.13 Effects of etching conditions on the average pore density of track-etched PC microfilters at 8N NaOH.

4.2.2   Effect of chemical etching condition on pore diameter

The investigation on the mechanism for pore enlargement during etching was carried out by varying the concentration of the etching or NaOH solution in the range of 2 to 8 N, the temperature of the etching solution in the range of 50 to 80 $^{\circ}$C, and etching time in the range of 30 to 150 minutes.

Figures 4.15 to 4.18 illustrate the effects of etching conditions on the average pore diameter for films, which were irradiated in the beam tube source at the placement distance of 70 cm for 180 minutes. According to the results, the pore diameter was found to increase monotonically with increasing concentration of NaOH, etching temperature, and etching time. Quantitatively, the pore diameter varied in the range of 0.486 to 9.514 μm. According to the results obtained, mild etching conditions of 2N NaOH at 50°C for the etching times of up to 150 minutes was not enough to enlarge the latent tracks to be visible under an optical microscope, however, if the etching temperature was increased to 60°C, enlarged pores were clearly visible under the optical microscope and the average diameter of these pores was 0.486 μm.

Fleischer et al. proposed a mechanism for pore formation in polymeric microfilters prepared by nuclear tracking and chemical etching technique. The first step is the latent track formation, which can be achieved by the bombardment of fission fragments from a nuclear reaction of an radioactive element in polymeric films. Later, these latent tracks were chemically etched in order to enlarge the latent

tracks and to form flow-through channels within the irradiated films. The chemical etching of PC films with NaOH can occur in two ways. The first is the chemical etching along the surface of the latent tracks formed and the second is the chemical etching along the general surface of the PC substrates. The mechanism of the chemical etching of PC films in NaOH is shown in Figure 4.14.
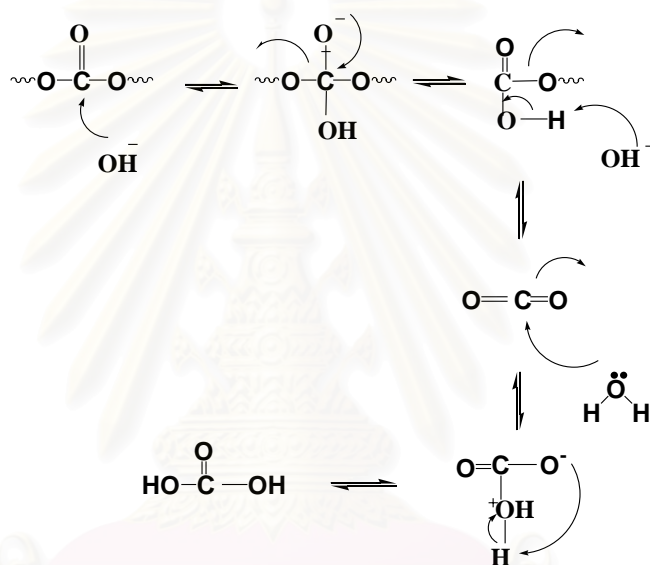
Figure 4.14   The mechanism of chemical etching of PC films in NaOH.
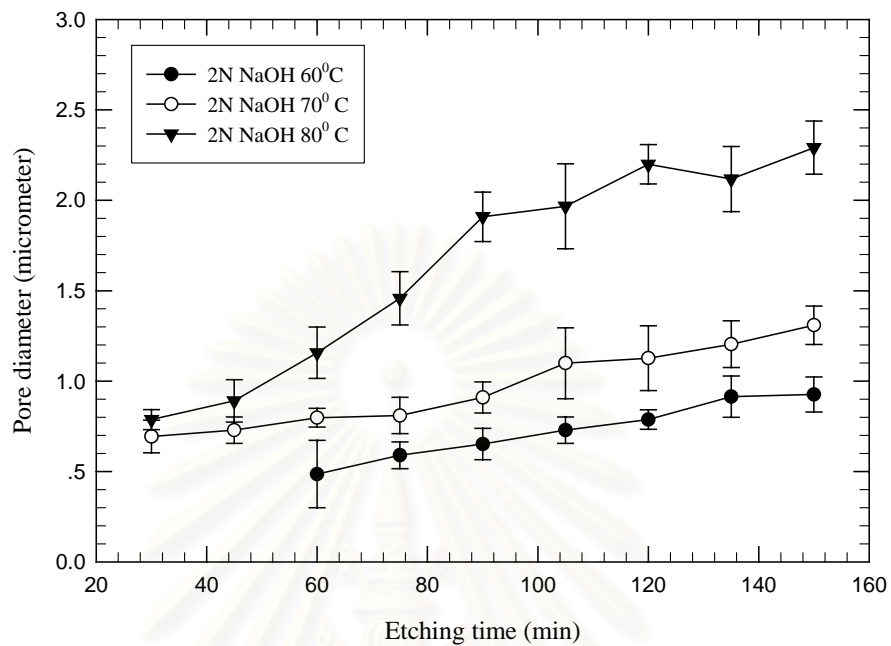
Figure 4.15 Effects of etching conditions on the average pore diameter at 2N NaOH of track-etched PC microfilters.
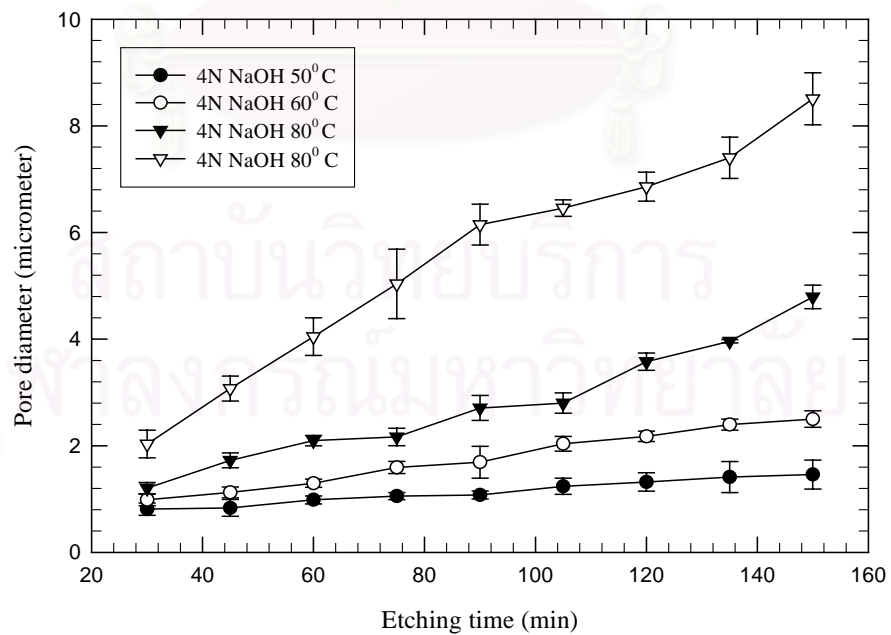
Figure 4.16 Effects of etching conditions on the average pore diameter at 4N NaOH of track-etched PC microfilters.
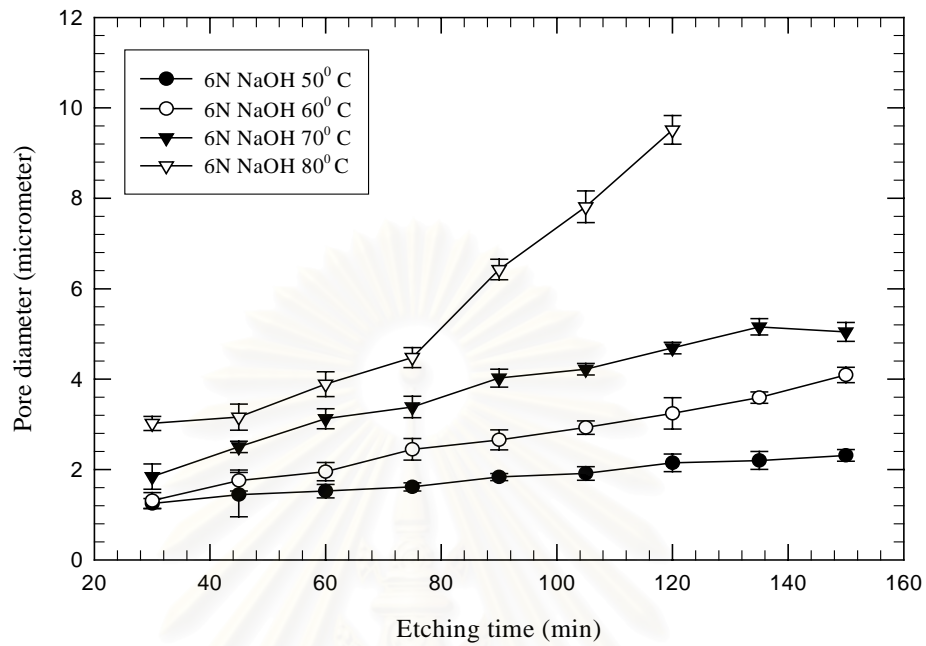
Figure 4.17   Effects of etching conditions on the average pore diameter at 6N NaOH of track-etched PC microfilters.
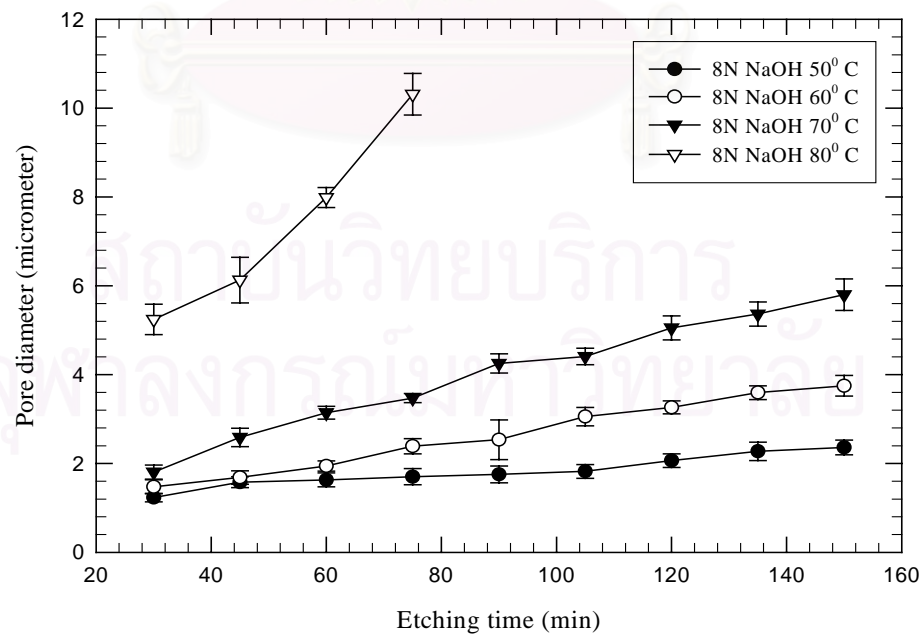
Figure 4.18   Effects of etching conditions on the average pore diameter at 8N NaOH of track-etched PC microfilters.

4.2.3   Pore geometry of track-etched PC microfilter

Figure 4.19 shows pore geometry of PC microfilters which were tracked in the beam tube source for 180 minute at the placement distance of 70 cm and etched with 5N NaOH at 70°C for 60 minutes. From this figure, several pore geometries were evident, such as circular and elliptic.

(a)   (b)
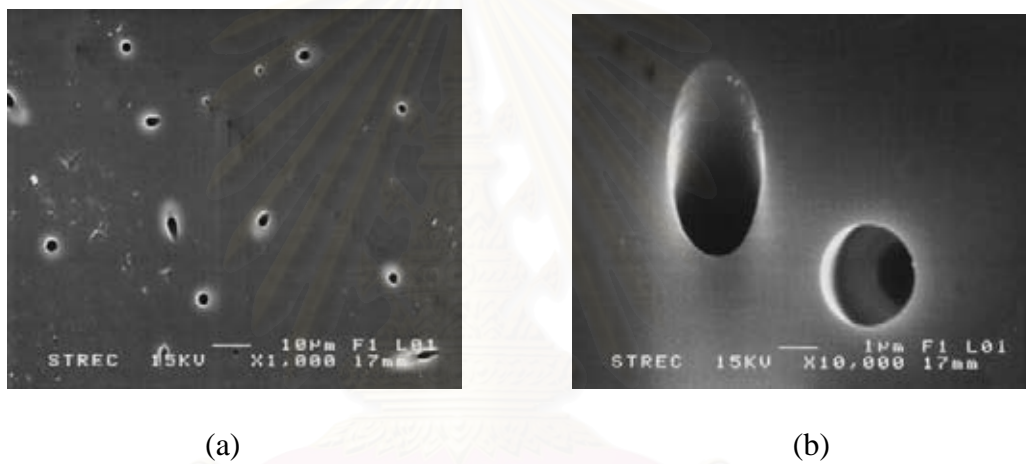
Figure 4.19   Scanning electron micrographs of track-etched PC microfilters (a) 1,000x and (b) 10,000x which was tracked by fission fragments from uranium-235 at beam tube for 180 minutes and etched with 5N NaOH at 70°C for 60 minutes.

The difference in the shape of pores can be explained very simply based on the angles at which the fission fragments came into contact with the films to form the latent tracks (see Figure 4.20). According to Figure 4.20, if the fission fragments from the yellow cake came into contact with the films at right angles, pores of circular geometry resulted. For any other angles, only elliptic pore results.
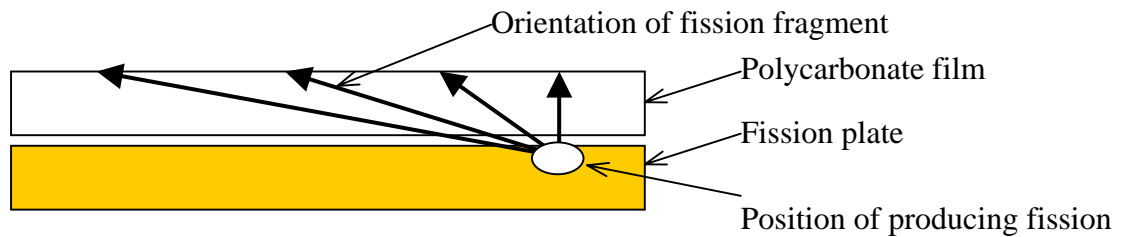
Figure 4.20   Orientation of fission fragment on PC film.

4.3   Effect of chemical etching condition on the thickness of track-etched PC microfilters

In this section, the effect of the chemical etching on the thickness of the PC microfilters is the main focus. A digital thickness gauge was used to measure the thickness of the etched PC films. Figure 4.21 shows the thickness of obtained microfilters (irradiated in the beam tube source at the placement distance of 70 cm for 180 minutes) as function of pore diameter. Clearly, the film thickness was found to decrease monotonically with increasing average pore diameter. The explanation for the obtained results is straightforward, since, during etching, the etchant did not only etch away the materials along the surface of the latent tracks, it also etched away materials along the general surface of the films. As a result, the stronger the etching conditions, the larger the average pores size and the thinner the resulting microfilter films.

Table 4.5   The thickness of track-etched PC microfilter as function of pore diameter in different etching condition.

Etching condition*	Pore diameter (μm)	Thickness (μm)
Virgin-PC	-	13
2N 60^0C 60 min	0.486	15
2N 70^0C 60 min	0.798	15
2N 80^0C 60 min	1.157	13
6N 60^0C 60 min	1.953	12
6N 70^0C 60 min	3.125	11
6N 80^0C 60 min	3.889	11
6N 80^0C 75 min	4.479	10
6N 80^0C 90 min	6.423	9
6N 80^0C 105min	7.813	8
6N 80^0C 120 min	9.514	7

* PC films were irradiated in the beam tube source for 180 minutes at the placement distance 70 cm.
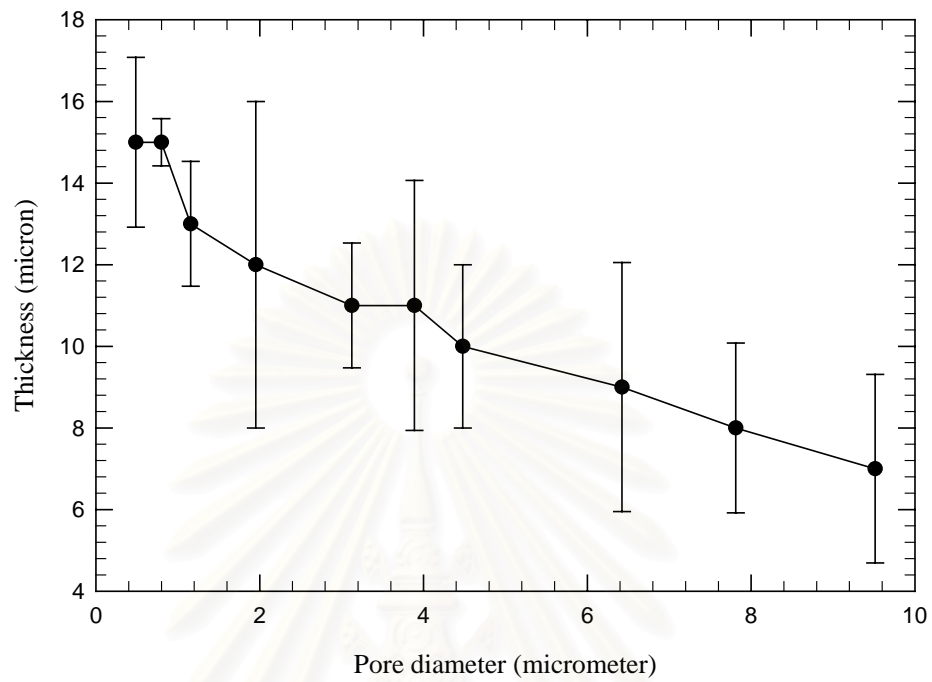
Figure 4.21 The thickness of track-etched PC microfilters as function of pore diameter.

4.4   FTIR measurement

The interaction of the heavy ions with polymers leads to bond breakage and formation of free radicals [46-48]. It has been realized that the radiation-induced effects depend not only on the properties of the targets (e.g. composition, molecular weight, etc.), but also on ion influence. In the present study, the effects of nuclear tracking and chemical etching on the chemical properties of as-prepared PC microfilters were studied using FTIR technique.

4.4.1   Tracked polycarbonate films

The material used in this study is aromatic PC. Its functional groups include methyl, phenyl ring, carbonyl, ether and hydroxyl. The infrared absorption peaks of the above mentioned groups are 2970 cm^{-1} (νCH$_3$), 1510 cm^{-1} (νC-H, aromatic), 1782 cm^{-1} (νC=O), 1012 cm^{-1} (νC-O-C) and 3500 cm^{-1} (νOH) respectively [48].
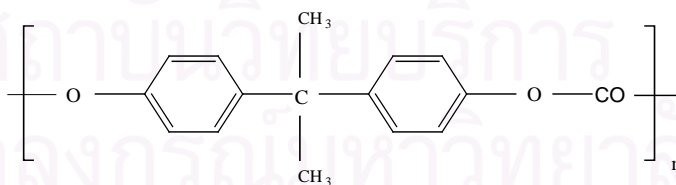
Figure 4.22   The chemical structure of polycarbonate.

The irradiation of PC films was carried out in the beam tube source at the placement distance of 70 cm. Figure 4.23 illustrates FTIR spectra of PC films irradiated with fission fragments at different irradiation times of 60, 120, 180, and 240 minutes, respectively. The corresponding track densities were 4.86×10^4, 10.15×10^4, 15.42×10^4 and 19.51×10^4 tracks/cm^2, respectively. Clearly, the absorbance of most observed peaks such as 1012, 1510 and 1782 cm^{-1} was found to decrease with increasing irradiation time. Since, as the irradiation time increased, the track density also increased, it is therefore logical to postulate that the decrease in the absorbance observed was a result of the increase in the track density. It is suggested that, during irradiation, PC molecules undergo chain scission at ether, phenyl ring, or carbonyl groups, which clearly resulted in the reduction in the number of these groups. Chain scission resulted in the reduction in the molecular weights [49], which causes an increase in the number of hydroxyl chain ends. As a result, with increasing irradiation time, the absorbance at the wave number of 3500 cm^{-1} was found to increase (see Figure 4.24).

Table 4.6   Normalized absorbance of irradiated PC films at different irradiation times in the beam tube source.

Number	Irradiation time (min)	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}
1	60	48,591	0.969	0.958	0.861	1.169
2	120	101,513	0.953	0.904	0.669	1.328
3	180	154,195	0.885	0.844	0.492	1.519
4	240	195,136	0.835	0.762	0.414	1.656
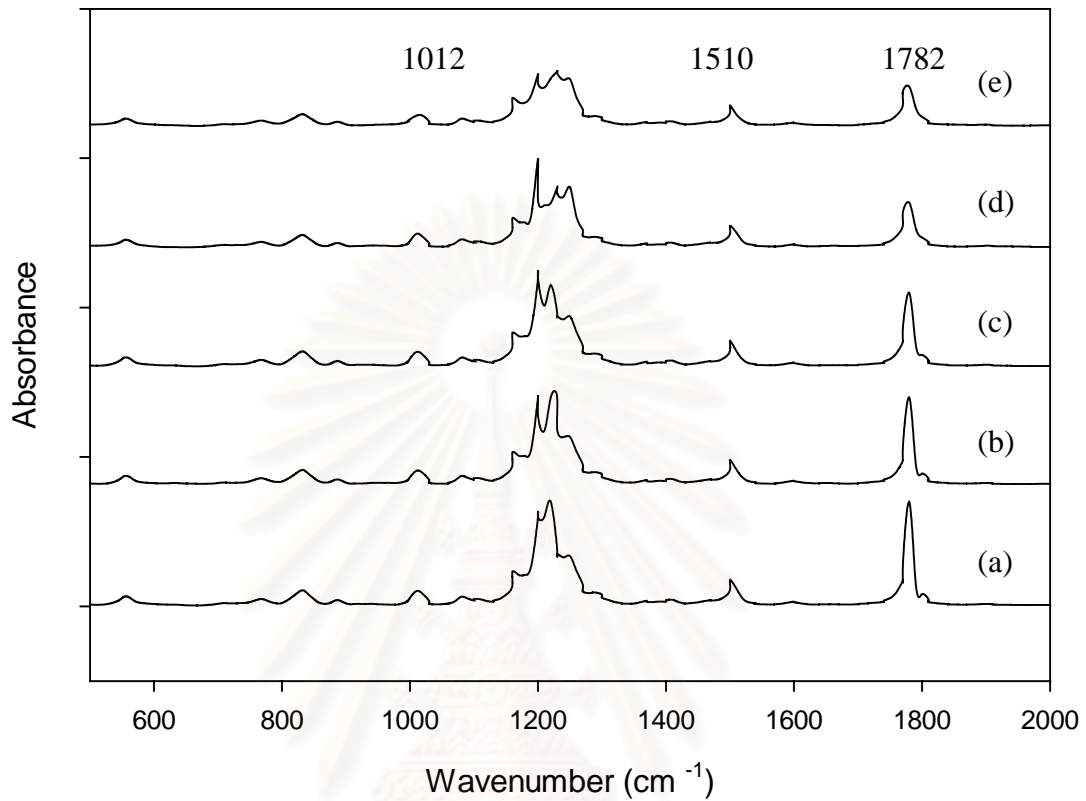
Figure 4.23 FTIR spectra of the 13 micron un-irradiated PC films (a) and irradiated PC films at different track density of 4.86x104 (b), 10.15 x10^4 (c), 15.42 x10^4 (d) and 19.51 x10^4 (e) track/cm^2 at absorbances of peaks 1012, 1510 and 1782 cm^{-1}.
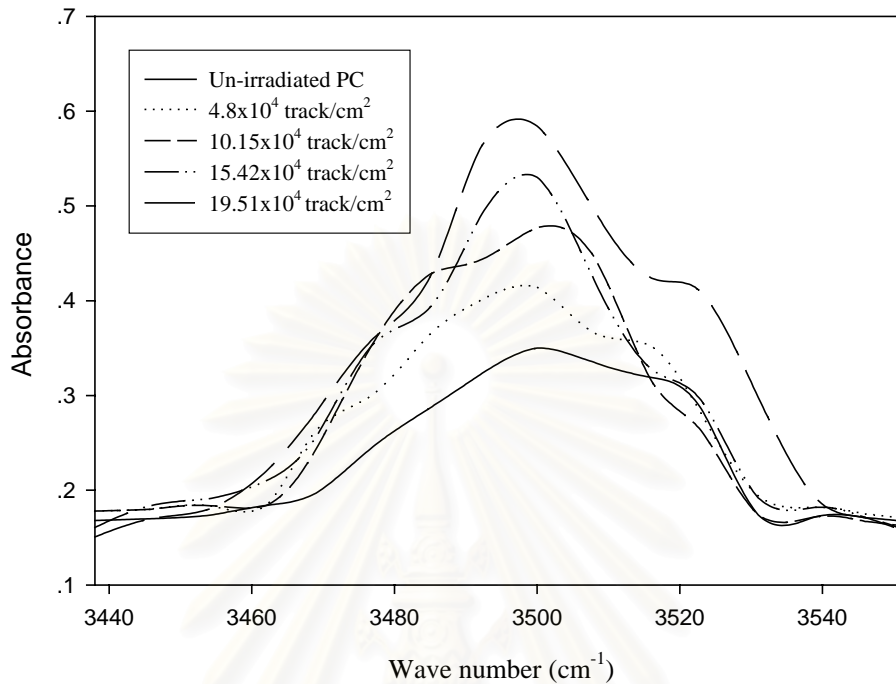
Figure 4.24 FTIR spectra of the 13-micron un-irradiated PC films (a) and irradiated PC films at different track density of 4.86×10^4 (b), 10.15×10^4 (c), 15.42×10^4 (d) and 19.51×10^4 (e) track/cm^2 at 3500 cm^{-1}.

In Figure 4.25, the normalized intensity of the absorbance bands at 1012, 1510 and 1782 cm^{-1} are plotted as a function of track density. The normalized absorbance is defined as

$$A/A_0$$

Where *A* is the absorbance of irradiated films

A_0 is the absorbance of un-irradiated PC films

It is clear that A/A_0 was found to decrease with increasing track density. On the contrary, that of the band at 3500 cm^{-1} increased with increasing track density (see Figure 4.26). An increase in the intensity of the hydroxyl band suggested an increased numbers of end groups of PC after irradiation. This is in consistent with the observation by Wang [50] that, after irradiation, the molecular weights of polymers were found to decrease, which, in turn, helped increase the etchability of the irradiated films.
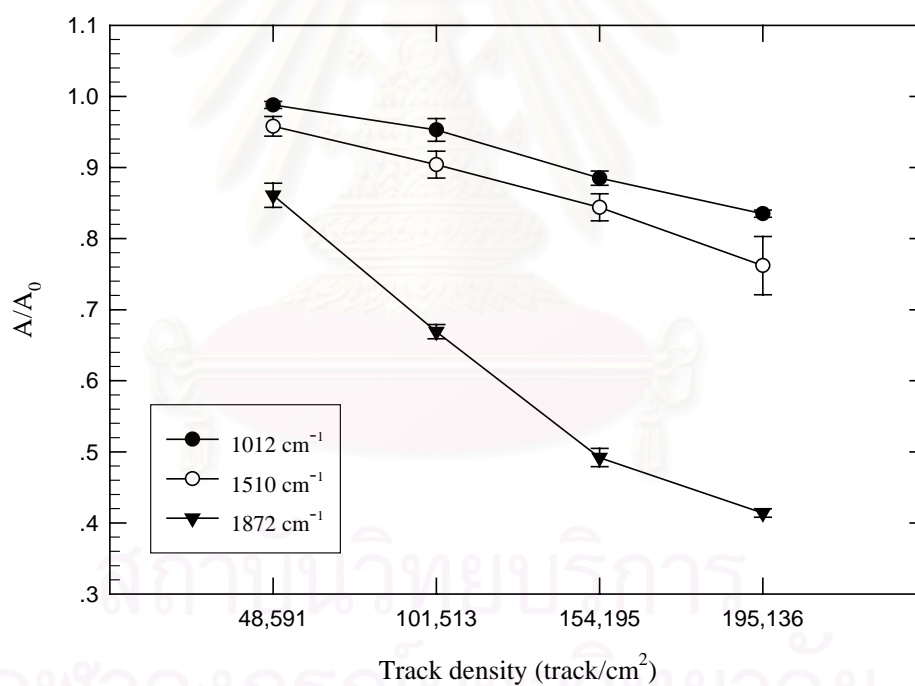
Figure 4.25   Normalized absorbances of irradiated PC films with the band at 1012, 1510 and 1782 cm^{-1} as function of the track density.
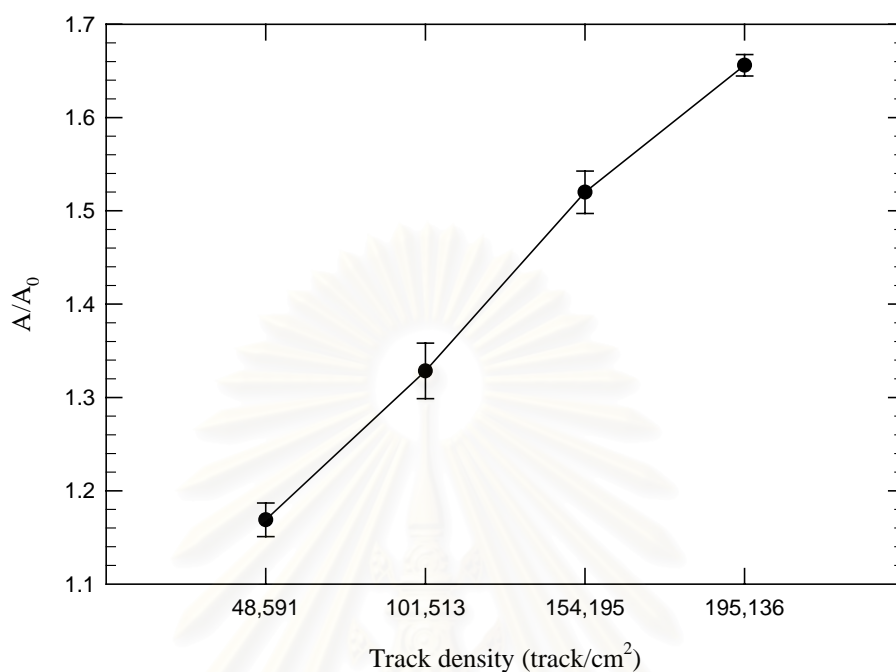
Figure 4.26 Normalized absorbance of irradiated PC films with the band at 3500 cm^{-1} as function of the track density.

4.4.2 Track-etched polycarbonate films

In the previous section, the effect of irradiation time on the chemical structure of the irradiated PC films was investigated. In this section, the effect of chemical etching on the chemical properties of etched PC films is the main focus. Specifically, the effects of etchant concentration, etching temperature, and etching time on the chemical functional groups of etched PC films were thoroughly investigated and the results are numerically summarized in Table 4.8.

Figures 4.27 and 4.28 show the absorbance bands at 1782 and 1012 cm^{-1}, respectively, of the as-etched PC films under different etching conditions. The absorbance bands of un-irradiated and irradiated PC films were also plotted for comparison. It is clear that the intensities of these bands were found to decrease from those of the un-irradiated and irradiated PC films down to that of the film etched under the mildest condition (2 N NaOH at 60^{o}C for 60 min) and were found to further decrease with increasing strength of the etching condition. On the contrary, the intensity of the absorbance band at 3500 cm^{-1} was found to increase from those of the un-irradiated and irradiated PC films up to that of the film etched under the mildest condition (2 N NaOH at 60^{o}C for 60 min) and were found to further increase with increasing strength of the etching condition. The results suggested that chemical etching causes the numbers of carbonyl and ether groups to decrease, while it causes the number of hydroxyl group to increase.

Figures 4.30 and 4.31 recapitulate the results shown in Figures 4.27 to 4.29 by plotting the normalized intensities of the absorption bands at 1782, 1012, and 3500 cm^{-1}, respectively.

Table 4.7 Normalized absorbance of track-etched PC microfilters at different etching condition.

Etching Condition*	Pore diameter (µm)	A/A_0 at 1012 cm^{-1}	A/A_0 at 1782 cm^{-1}	A/A_0 at 3500 cm^{-1}
2N 60°C 60 min	0.486	0.598	0.399	1.647
2N 70°C 60 min	0.798	0.580	0.388	1.671
2N 80°C 60 min	1.157	0.548	0.369	1.763
6N 60°C 60 min	1.953	0.500	0.358	1.791
6N 70°C 60min	3.125	0.468	0.341	1.857
6N 80°C 60min	3.889	0.450	0.325	1.949
6N 80°C 75 min	4.479	0.427	0.324	1.998
6N 80°C 90 min	6.423	0.391	0.285	2.055
6N 80°C 105 min	7.813	0.326	0.261	2.061
6N 80°C 120 min	9.514	0.273	0.156	2.101

* PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.
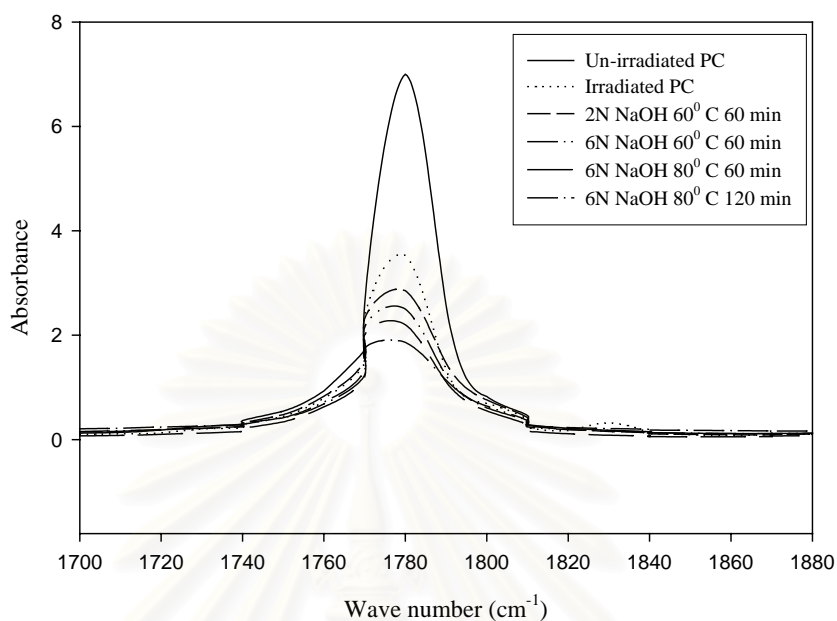
Figure 4.27   FTIR spectra of track-etched PC microfilters at 1782 cm^{-1} in different chemical etching conditions.
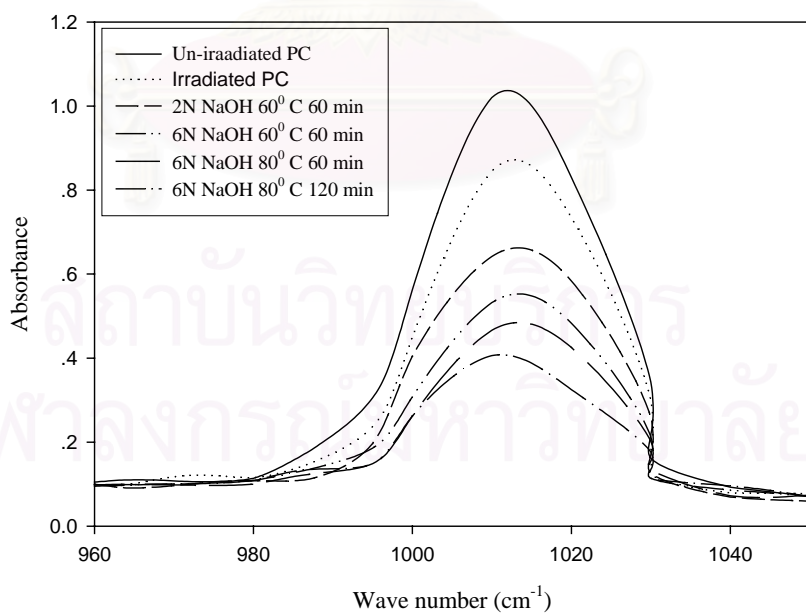
Figure 4.28   FTIR spectra of track-etched PC microfilters at 1012 cm^{-1} in different chemical etching conditions.
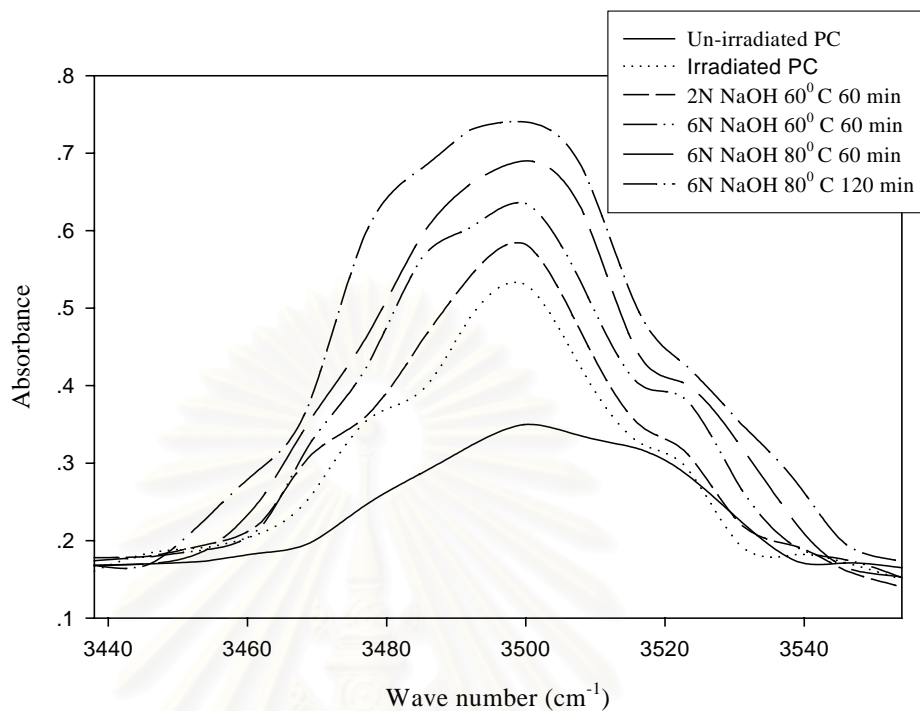
Figure 4.29 FTIR spectra of track-etched PC microfilters at 3500 cm^{-1} in different chemical etching conditions.
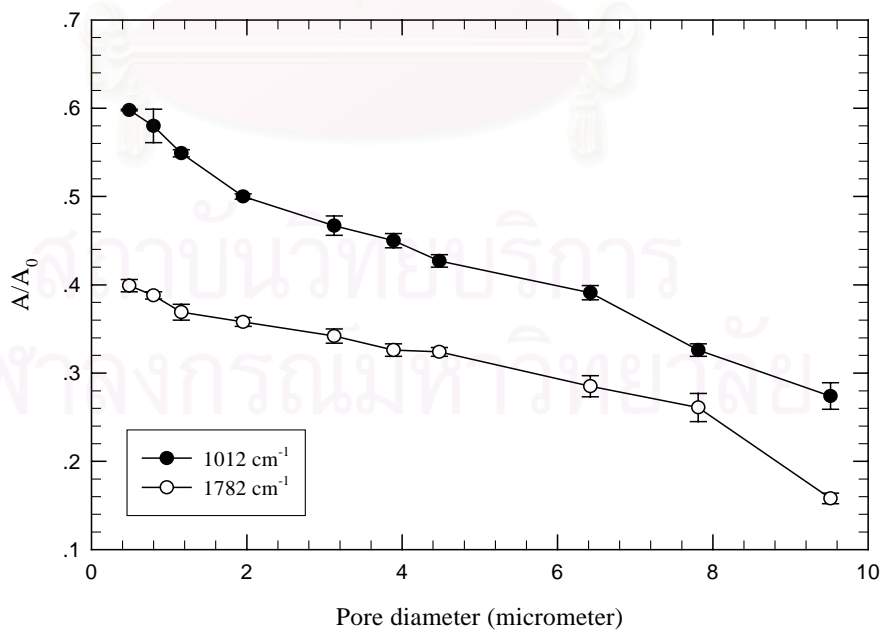
Figure 4.30 Normalized absorbances of track-etched PC microfilters with the band at 1782 and 1012 cm^{-1}.
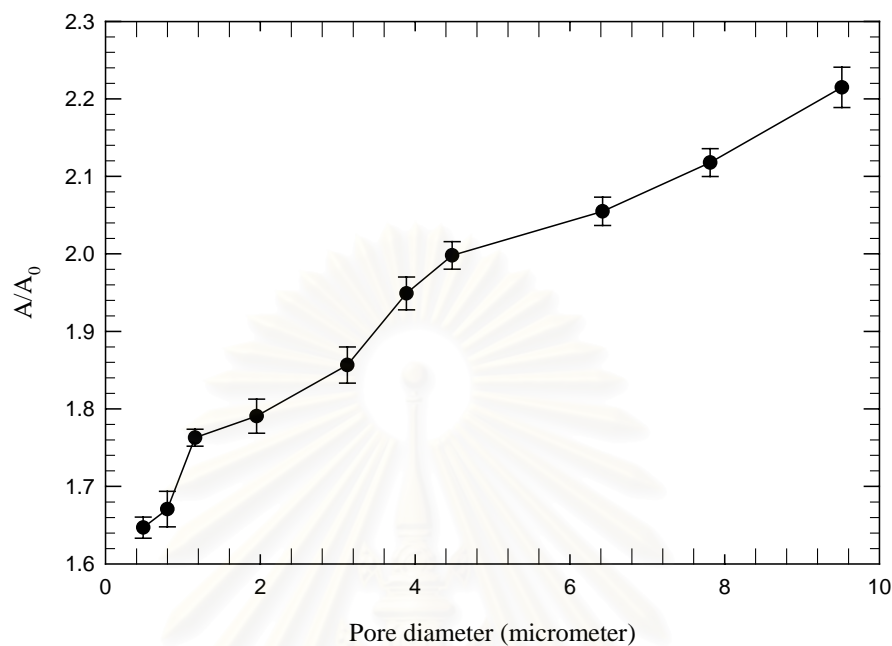
Figure 4.31   Normalized absorbances of track-etched PC microfilters with the band at 3500 cm^{-1}.

4.5   Effect of pore diameter on water flux

In this section, the water flux of the obtained track-etched PC microfilters was tested using a water flux tester and the results are summarized in Table 4.9. The effects of etching conditions were also investigated. The different etching conditions were responsible for the difference in the pore diameters observed, in which it was found the average pore diameter was an increasing function of the strength of the etching conditions, as previously noted.

Table 4.8   Water flux of track-etched PC microfilter as function of pore diameter in different etching condition.

Etching condition*	Pore diameter (μm)	Water flux (ml/min/cm^2)
2N 60^{0}C 60 min	0.486	-
2N 70^{0}C 60 min	0.798	-
2N 80^{0}C 60 min	1.157	-
6N 60^{0}C 60 min	1.953	1.769×10^{-4}
6N 70^{0}C 60 min	3.125	1.591×10^{-3}
6N 80^{0}C 60 min	3.889	4.951×10^{-3}
6N 80^{0}C 75 min	4.479	8.089×10^{-3}
6N 80^{0}C 90 min	6.423	1.305×10^{-2}
6N 80^{0}C 105min	7.813	1.967×10^{-2}
6N 80^{0}C 120 min	9.514	3.055×10^{-2}

* PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

Figure 4.32 shows a plot of water permeability of the as-prepared track-etched PC microfilters as a function of the average pore diameter. According to the data shown in Table 4.9 and Figure 4.32, it is clear that mild etching conditions were not enough to cause the latent tracks to form the flow-through channels and that the water flux was found to increase monotonically with increasing the average pore diameter.
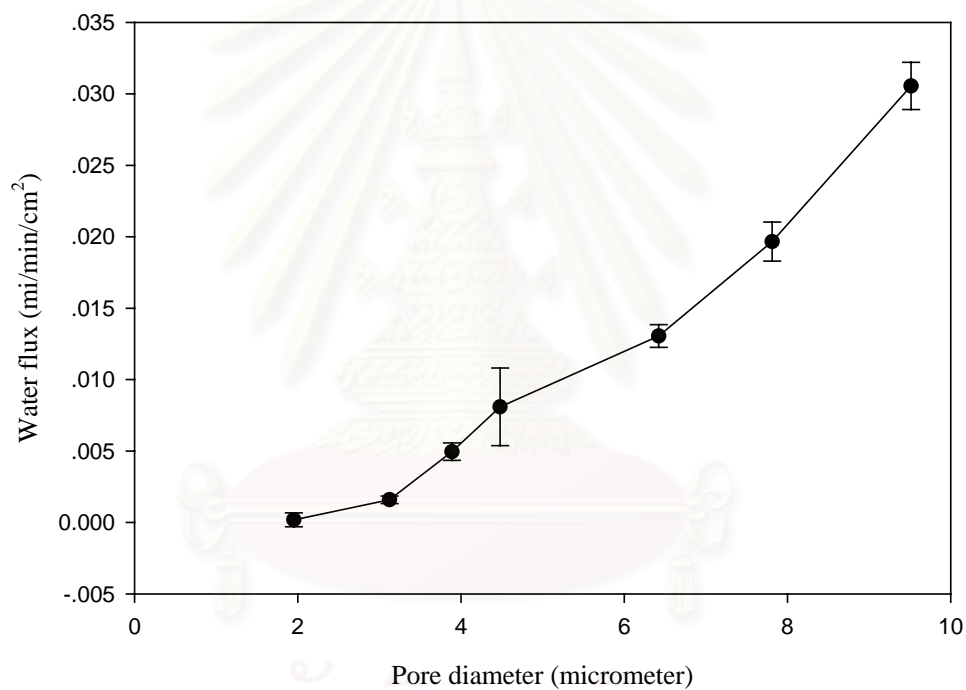
Figure 4.32 Water flux of track-etched PC microfilter as function of pore diameter.

4.6   Effect of etching condition on mechanical properties of track-etched PC microfilters

The tensile strength, yield strength, modulus and percentage of elongation of un-irradiated, irradiation, and track-etched PC films are shown in Table 4.9. The effects of etching conditions on the mechanical properties of these films were also investigated. Qualitatively, it is clear that tensile strength, yield strength, and Young's modulus were all found to decrease monotonically with increasing strength of the etching conditions or with increasing average pore diameter (see also Figures 4.33, 4.34, and 4.35) or with increasing pore density. On the contrary, the percentage of elongation was found to increase monotonically with increasing strength of the etching conditions or with increasing average pore diameter (sees also Figure 4.36).

The decrease in the strength of the track-etched PC microfilters with increasing average pore diameter and pore density is straightforward. As the number and size of the pores increase, cracks tend to propagate more easily. Simultaneously, the increase in the average pore diameter and pore density cause the track-etched PC microfilters to be more stretchable, thus the percentage of elongation was found to increase.

Table 4.9   Tensile properties of virgin, as-tracked, and as-prepared track-etched PC films.

Etching condition*	Pore diameter (μm)	Pore density (pore/cm^2)	Tensile strength (MPa)	Yield strength (MPa)	Modulus (MPa)	% Elongation
Virgin-PC	-	-	13.37	62.69	1490.29	7.13
Tracked-PC	-	-	11.86	59.00	1447.86	7.73
2N 60^{0}C 60 min	0.486	5.19x10^4	11.05	57.92	1231.49	11.23
2N 70^{0}C 60 min	0.798	1.37x10^5	10.88	54.05	1111.77	12.98
2N 80^{0}C 60 min	1.157	1.48x10^5	9.62	48.94	1062.41	15.18
6N 60^{0}C 60 min	1.953	1.49x10^5	8.96	45.93	906.19	17.47
6N 70^{0}C 60 min	3.125	1.54x10^5	7.85	43.94	809.53	22.89
6N 80^{0}C 60 min	3.889	1.54x10^5	6.24	36.82	608.5	24.15
6N 80^{0}C 75 min	4.479	1.49x10^5	5.91	30.62	569.39	26.68
6N 80^{0}C 90 min	6.423	1.52x10^5	4.90	27.94	512.58	28.83
6N 80^{0}C 105min	7.813	1.55x10^5	4.07	22.85	446.87	32.68
6N 80^{0}C 120 min	9.514	1.54x10^5	2.58	20.47	385.24	35.73

* PC films were irradiated in the beam tube source for 180 minutes at the placement distanec of 70 cm.
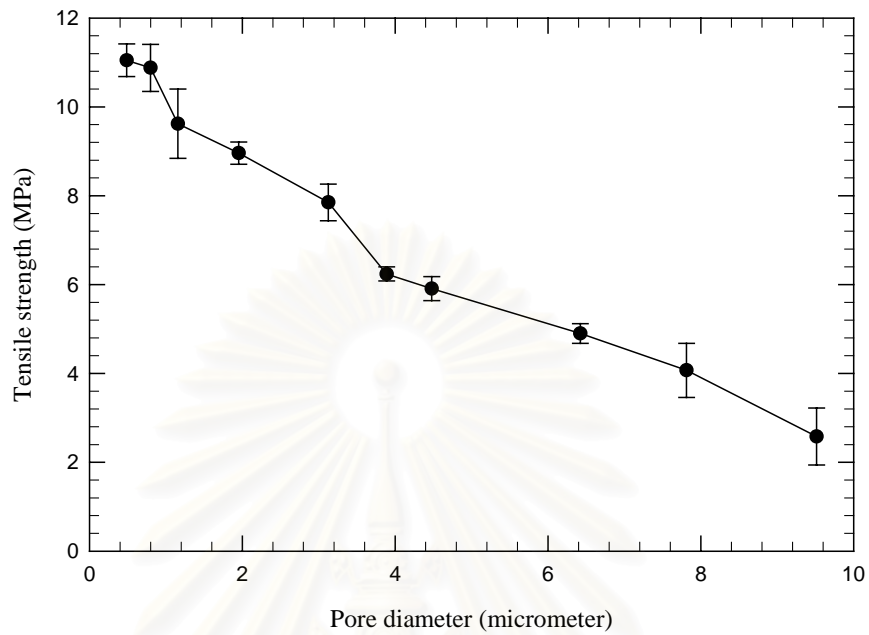
Figure 4.33   Effect of pore diameter on the tensile strength (MPa) of track-etched PC microfilters.
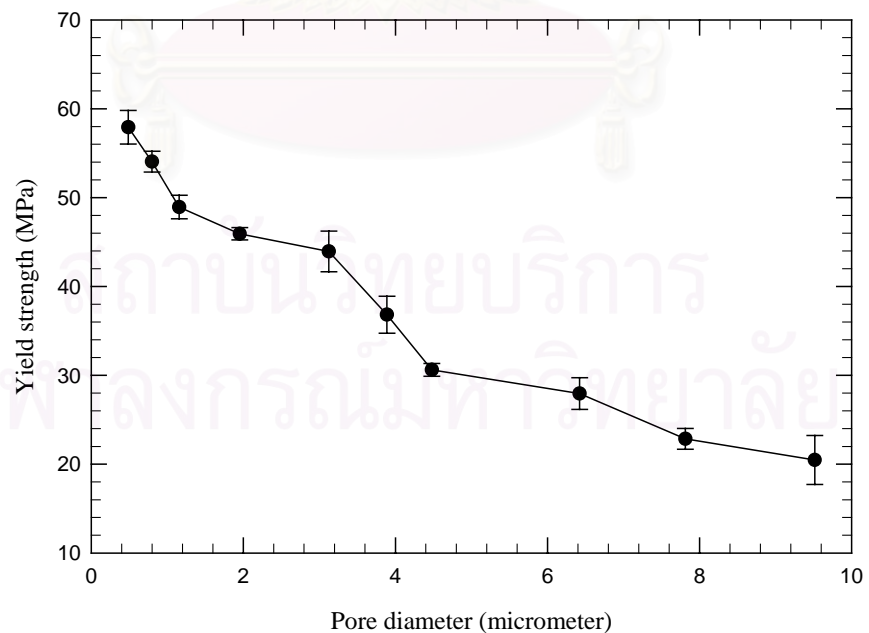
Figure 4.34   Effect of pore diameter on the yield strength (MPa) of track-etched PC microfilters.
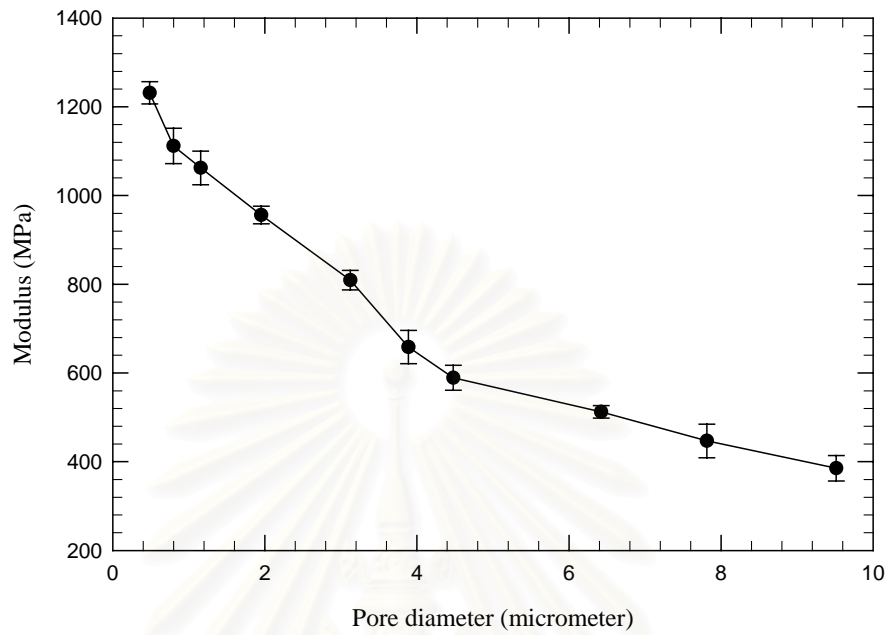
Figure 4.35   Effect of pore diameter on the modulus (MPa) of track-etched
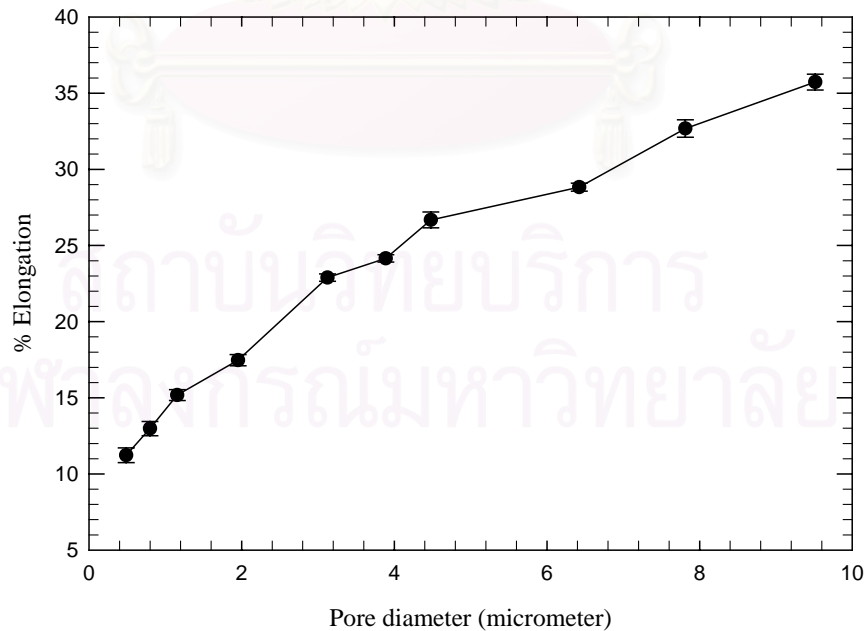
PC microfilters.

Figure 4.36   Effect of pore diameter on the %elongation of track-etched PC

microfilters.

CHAPTER V

CONCLUSION

In this work , track-etched PC microfilters were successfully prepared from irradiating by fission fragment from uranium-235 in neclear reactor and subsequently employing NaOH solution to enlarge the radiation-induced damaged tracks in order to producing the through hole. The thickness, chemical structure, water permeability and mechanical properties of as-prepared microfilters were investigated. The following conclusions could be drawn:

1. Track-etched PC microfilters have been successfully prepared by first irradiating PC films with fission fragment from ammonium diurinate in nuclear reactor and subsequently subjecting the treated films through an etching procedure. The as-prepared microfilters had the average pore diameter ranging from 0.486 to 9.514 μm and the pore density ranging from 0.50×10^4 to 1.55×10^5 pores/cm^2. This is different from the observation by Cui [41] that, using fission fragment from U-235, the average pore diameter and the pore density were found ranging from 0.2 to 12 μm and 10^5-10^8 pores/cm^2, respectively.

2. After enlarging the radiation-induced damaged tracks, The thickness of track-etched PC microfilter was discovered decreasing with increasing pore diameter.

3. Fourier transformed infrared spectroscopy (FTIR) results suggested some modifications to the chemical structure after fission fragment tracking and chemical etching.

4. Water flux of track-etched PC microfilter was seemed increasing with increasing pore diameter.

5. There is significant change of the mechanical properties after employing NaOH solution to enlarge the radiation-induced damaged tracks. For example, the tensile strength, yield strength and Modulus were found decreased with porosity but % elongation was increased.

Suggestion

1. Polycarbonate microfilters is being widely utillized in such fields as industry, medical treament and laboratory. These applications are making significant contribution toward improving the standard of living in Thai land.

2. Similar method is being applied for some particular polymers for the microfilter production such as poly(ethylene terephthalate), polypropylene, poly(vinylidene fluoride) and polyimide.

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APPENDICES

APPENDIX A

Pore Diameter and Pore Density of PC Microfilters

Table A.1 Pore diameter of PC microfilters at 2N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore diameter (micron)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	-	-	-	-	-	-	-	-	0.733	0.624	0.694	0.090	0.833	0.729	0.787	0.055
45	-	-	-	-	-	-	-	-	0.829	0.713	0.729	0.073	1.041	0.729	0.891	0.117
60	-	-	-	-	0.937	0.156	0.486	0.264	0.833	0.729	0.798	0.052	1.458	1.249	1.157	0.142
75	-	-	-	-	0.729	0.520	0.590	0.074	0.937	0.624	0.810	0.101	1.666	1.458	1.458	0.147
90	-	-	-	-	0.833	0.729	0.752	0.087	0.937	0.624	0.810	0.086	2.083	1.771	1.909	0.137
105	-	-	-	-	0.833	0.624	0.729	0.073	1.354	0.833	1.099	0.196	2.291	1.458	1.967	0.235
120	-	-	-	-	0.937	0.729	0.787	0.054	1.249	0.833	1.127	0.179	2.291	2.083	2.199	0.109
135	-	-	-	-	1.041	0.729	0.914	0.114	1.458	1.041	1.204	0.129	2.291	1.875	2.118	0.180
150	-	-	-	-	1.041	0.833	0.926	0.097	1.458	1.250	1.309	0.106	2.499	2.083	2.291	0.147

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

Table A.2 Pore diameter of PC microfilters at 4N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore diameter* (micron)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	0.937	0.208	0.810	0.114	1.146	0.833	0.984	0.107	1.354	1.041	1.204	0.106	2.187	1.562	2.031	0.259
45	1.041	0.624	0.833	0.156	1.249	1.041	1.123	0.101	1.874	1.458	1.725	0.139	3.437	2.816	3.073	0.234
60	1.041	0.833	0.984	0.076	1.458	1.249	1.296	0.076	2.292	1.979	2.095	0.097	4.375	3.437	4.045	0.353
75	1.146	0.937	1.055	0.067	1.667	1.354	1.592	0.114	2.499	1.979	2.164	0.162	5.937	4.375	5.035	0.652
90	1.250	1.046	1.076	0.074	2.083	1.249	1.689	0.298	3.124	2.499	2.708	0.233	7.187	5.937	6.146	0.383
105	1.458	1.041	1.238	0.151	2.291	1.875	2.037	0.139	3.125	2.500	2.800	0.191	6.458	5.937	6.250	0.156
120	1.667	1.042	1.319	0.173	2.292	2.083	2.175	0.097	3.750	3.432	3.576	0.164	6.875	6.250	6.858	0.271
135	1.847	1.041	1.412	0.291	2.499	2.292	2.396	0.104	4.375	3.437	3.958	0.313	7.880	6.619	7.401	0.386
150	1.874	1.041	1.458	0.272	2.708	2.291	2.499	0.156	5.00	4.375	4.79	0.221	9.063	7.813	8.507	0.488

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

**Table A.3**   Pore diameter of PC microfilters at 6N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore diameter (micron)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	1.406	1.093	1.250	0.110	1.563	1.094	1.313	0.178	2.187	1.562	1.844	0.279	3.125	2.812	3.021	0.156
45	1.562	1.250	1.445	0.149	2.031	1.562	1.757	0.234	2.656	2.343	2.500	0.127	3.750	2.812	3.159	0.289
60	1.718	1.406	1.523	0.149	2.187	1.875	1.953	0.201	3.437	2.968	3.125	0.221	4.375	3.437	3.889	0.275
75	1.718	1.562	1.615	0.090	2.656	2.187	2.448	0.238	3.593	3.125	3.385	0.238	4.687	4.062	4.479	0.221
90	1.875	1.718	1.835	0.078	2.812	2.343	2.656	0.221	4.218	3.750	4.023	0.197	6.875	6.250	6.423	0.227
105	2.031	1.718	1.914	0.149	3.125	2.813	2.929	0.149	4.375	4.062	4.219	0.127	8.437	7.500	7.813	0.349
120	2.344	1.875	2.148	0.196	3.594	2.813	3.242	0.346	4.843	4.531	4.687	0.127	10.000	9.514	9.687	0.317
135	2.344	1.875	2.201	0.197	3.750	3.437	3.593	0.127	5.312	5.00	5.156	0.180	-	-	-	-
150	2.500	2.187	2.313	0.131	4.375	3.906	4.093	0.171	5.625	5.046	5.156	0.209	-	-	-	-

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

Table A.4   Pore diameter of PC microfilters at 8N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore diameter (micron)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	1.406	1.094	1.232	0.094	1.719	1.250	1.476	0.158	2.031	1.718	1.805	0.158	5.625	5.000	5.243	0.342
45	1.875	1.406	1.579	0.122	1.875	1.563	1.684	0.152	2.812	2.187	2.586	0.208	6.250	5.937	6.128	0.512
60	1.875	1.562	1.631	0.158	2.188	1.875	1.944	0.114	3.281	2.968	3.142	0.144	8.437	7.83	7.986	0.227
75	2.031	1.615	1.701	0.182	2.500	2.188	2.389	0.171	3.750	3.437	3.472	0.104	10.937	10.000	10.313	0.469
90	2.125	1.718	1.753	0.188	3.125	2.031	2.535	0.447	4.375	3.750	4.253	0.217	-	-	-	-
105	2.188	1.835	1.922	0.156	3.438	2.813	3.056	0.208	4.687	4.062	4.409	0.187	-	-	-	-
120	2.343	1.875	2.065	0.152	3.438	2.125	3.264	0.145	5.625	4.687	5.052	0.271	-	-	-	-
135	2.500	1.875	2.274	0.208	3.750	3.438	3.594	0.156	5.781	5.00	5.365	0.271	-	-	-	-
150	2.500	2.178	2.361	0.165	4.063	3.438	3.750	0.234	6.25	5.312	5.798	0.353	-	-	-	-

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

Table A.5 Pore density of PC microfilters at 2N NaOH by vary etching time and etching temperature.

| Etching time (min)	Pore density (pore/cm^2)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	-	-	-	-	-	-	-	-	6.98x10^4	3.14x10^4	5.49x10^4	6.20x10^3	9.76x10^4	7.49x10^4	9.01x10^4	1..31x10^3
45	-	-	-	-	-	-	-	-	1.50x10^5	4.84x10^4	9.85x10^4	5.11x10^3	1.24x10^5	1.20x10^5	1.20x10^5	3.06x10^3
60	-	-	-	-	6.98x10^4	3.76x10^4	5.19x10^4	1.64x10^4	1.18x10^5	8.59x10^4	1.04x10^5	1.46x10^4	1.58x10^5	1.39x10^5	1.48x10^5	1.01x10^4
75	-	-	-	-	1.07x10^5	8.06x10^4	9.35x10^4	1.42x10^4	1.59x10^5	1.23x10^5	1.47x10^5	2.11x10^4	1.47x10^5	1.26x10^5	1.36x10^5	1.05x10^4
90	-	-	-	-	1.34x10^5	101x10^5	1.14x10^5	1.57x10^4	1.70x10^5	1.21x10^5	1.51x10^5	5.86x10^4	1.70x10^5	1.47x10^5	1.57x10^5	1.16x10^4
105	-	-	-	-	1.50x10^5	107x10^5	1.33x10^5	1.84x10^4	1.44x10^5	1.24x10^5	1.32x10^5	1.07x10^4	1.59x10^5	1.47x10^5	1.53x10^5	5.67x10^3
120	-	-	-	-	1.83x10^5	140x10^5	1.61x10^5	2.15x10^4	1.62x10^5	1.35x10^5	1.49x105	1.35x10^3	1.44x10^5	1.15x10^5	1.42x10^5	2.58x10^4
135	-	-	-	-	1.72x10^5	127x10^5	1.47x10^5	1.43x10^4	1.73x10^5	1.25x10^5	1.42x10^5	1.84x10^3	1.43x10^5	1.16x10^5	1.26x10^5	1.51x104
150	-	-	-	-	1.61x10^5	145x10^5	1.46x10^5	1.60x10^4	1.71x10^5	1.33x10^5	1.46x10^5	1.23x10^3	1.43x10^5	130x10^5	135x105	6.77x10^3

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

**Table A.6**   Pore density of PC microfilters at 4N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore density (pore/cm^2)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	1.07×10^5	6.98×10^4	8.70×10^4	1.38×10^4	1.34×10^5	6.97×10^4	1.05×10^5	2.79×10^4	1.31×10^5	7.67×10^4	1.21×10^5	1.01×10^3	1.68×10^5	1.28×10^5	1.41×10^5	1.83×10^4
45	1.34×10^5	9.59×10^4	1.07×10^5	1.83×10^4	1.71×10^5	1.31×10^5	1.56×10^5	1.24×10^4	1.50×10^5	1.02×10^5	1.31×10^5	2.46×10^4	1.61×10^5	1.29×10^5	1.47×10^5	1.22×10^4
60	1.39×10^5	1.28×10^5	1.34×10^5	5.46×10^3	1.77×10^5	1.52×10^5	1.61×10^5	1.09×10^4	1.88×10^5	1.02×10^5	1.44×10^5	5.71×10^3	1.72×10^5	1.34×10^5	1.53×10^5	1.28×10^4
75	1.71×10^5	1.07×10^5	1.40×10^5	3.35×10^4	1.61×10^5	1.11×10^5	1.40×10^5	2.08×10^4	1.88×10^5	1.36×10^5	1.60×10^5	1.09×10^4	1.82×10^5	1.34×10^5	1.57×10^5	5.32×10^3
90	1.98×10^5	1.16×10^5	1.49×10^5	6.20×10^3	1.98×10^5	1.34×10^5	1.65×10^5	2.64×10^4	1.71×10^5	1.38×10^5	1.54×10^5	1.48×10^4	1.67×10^5	1.23×10^5	1.46×10^5	1.20×10^4
105	1.88×10^5	1.25×10^5	1.59×10^5	2.60×10^4	1.61×10^5	1.18×10^5	1.41×10^5	1.93×10^4	1.82×10^5	1.22×10^5	1.43×10^5	1.24×10^4	1.66×10^5	1.34×10^5	1.47×10^5	5.71×10^3
120	1.62×10^5	1.39×10^5	1.52×10^5	1.17×10^4	1.61×10^5	1.16×10^5	1.44×10^5	8.21×10^3	1.61×10^5	1.34×10^5	1.48×10^5	1.17×10^4	1.61×10^5	1.28×10^5	1.48×10^5	1.10×10^4
135	1.61×10^5	1.23×10^5	1.41×10^5	1.60×10^4	1.66×10^5	1.25×10^5	1.52×10^5	1.83×10^4	1.61×10^5	1.39×10^5	1.52×10^5	6.58×10^3	1.77×10^5	1.45×10^5	1.56×10^5	1.27×10^4
150	1.50×10^5	1.21×10^5	1.41×10^5	8.21×10^3	1.62×10^5	1.34×10^5	1.45×10^5	1.17×10^4	1.65×10^5	1.34×10^5	1.51×10^5	1.54×10^4	1.69×10^5	1.28×10^5	1.51×10^5	1.14×10^4

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

Table A.7   Pore density of PC microfilters at 6N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore density (pore/cm^2)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	1.72x10^5	8.54x10^4	1.18x10^5	6.81x10^3	1.50x10^5	1.29x10^5	1.39x10^5	7.86x10^3	1.58x10^5	1.34x10^5	1.41x10^5	1.28x10^4	1.55x10^5	1.31x10^5	1.43x10^5	1.01x10^4
45	1.50x10^5	1.18x10^5	1.29x10^5	1.72x10^4	1.61x10^5	1.28x10^5	1.50x10^5	8.90x10^3	1.61x10^5	1.29x10^5	1.48x10^5	1.35x10^4	1.82x10^5	1.12x10^5	1.48x10^5	2.67x10^4
60	1.49x10^5	1.02x10^5	1.40x10^5	1.03x10^4	1.55x10^5	1.34x10^5	1.49x10^5	5.23x10^3	1.63x10^5	1.47x10^5	1.54x10^5	8.37x10^3	1.82x10^5	1.38x10^5	1.54x10^5	1.09x10^4
75	1.58x10^5	1.34x10^5	1.46x10^5	1.09x10^4	1.88x10^5	1.23x10^5	1.50x10^5	1.74x10^4	1.82x10^5	1.50x10^5	1.61x10^5	5.60x10^3	1.68x10^5	1.22x10^5	1.49x10^5	2.45x10^4
90	1.61x10^5	1.38x10^5	1.51x10^5	1.05x10^4	1.45x10^5	1.18x10^5	1.37x10^5	1.42x10^4	1.55x10^5	1.23x10^5	1.44x10^5	3.93x10^3	1.63x10^5	1.33x10^5	1.51x10^5	1.11x10^4
105	1.98x10^5	1.22x10^5	1.44x10^5	9.32x10^3	1.60x10^5	1.34x10^5	1.47x10^5	1.84x10^4	1.82x10^5	1.34x10^5	1.51x10^5	9.72x10^3	1.93x10^5	1.29x10^5	1.55x10^5	1.64x10^4
120	1.61x10^5	1.34x10^5	1.47x10^5	9.83x10^3	1.71x10^5	1.18x10^5	1.43x10^5	1.75x10^4	1.56x10^5	1.28x10^5	1.44x10^5	1.01x10^4	1.60x10^5	1.46x10^5	1.54x10^5	5.42x10^3
135	1.73x10^5	1.47x10^5	1.55x10^5	5.72x10^3	1.98x10^5	1.39x10^5	1.53x10^5	1.42x10^4	1.68 x10^5	1.45x10^5	1.54x10^5	8.69x10^3	-	-	-	-
150	1.65x10^5	1.34x10^5	1.46x10^5	1.02x10^4	1.66x10^5	1.39x10^5	1.46x10^5	1.80x10^4	1.58x10^5	1.42x10^5	1.52x10^5	7.91x10^3	-	-	-	-

*   PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.

Table A.8 Pore density of PC microfilters at 8N NaOH by vary etching time and etching temperature.

Etching time (min)	Pore density (pore/cm^2)															
	50 ° C				60 ° C				70 ° C				80 ° C			
	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD	Max	Min	Mean	SD
30	1.66x10^5	1.37x10^5	1.51x10^5	1.40x10^4	1.60x10^5	1.34x10^5	1.48x10^5	1.12x10^4	1.77x10^5	1.28x10^5	1.50x10^5	1.89x10^4	1.82x10^5	1.28x10^5	1.54x10^5	1.95x10^4
45	1.64x10^5	1.45x10^5	1.53x10^5	1.05x10^4	1.71x10^5	1.61x10^5	1.65x10^5	2.01x10^3	1.88x10^5	1.28x10^5	1.50x10^5	2.26x10^4	1.66x10^5	1.35x10^5	1.52x10^5	1.40x10^4
60	1.77x10^5	1.28x10^5	1.45x10^5	2.02x10^4	1.93x10^5	1.02x10^5	1.55x10^5	3.90x10^4	1.82x10^5	1.61x10^5	1.68x10^5	1.15x10^4	1.61x10^5	9.36x10^2	1.43x10^5	2.70x10^4
75	1.77x10^5	9.62x10^4	1.47x10^5	3.10x10^4	1.92x10^5	1.43x10^5	1.45x10^5	1.72x10^4	1.82x10^5	1.38x10^5	1.58x10^5	1.74x10^4	1.62x10^5	1.34x10^5	1.46x10^5	1.40x10^4
90	1.78x10^5	1.45x10^5	1.62x10^5	1..25x10^4	1.67x10^5	1.18x10^5	1.49x10^5	2.06x10^4	1.59x10^5	1.28x10^5	1.49x10^5	1.23x10^4	-	-	-	-
105	1.68x10^5	1.45x10^5	1.58x10^5	9.01x10^3	1.77x10^5	1.45x10^5	1.61x10^5	1.21x10^4	1.88x10^5	1.36x10^5	1.56x10^5	1.92x10^4	-	-	-	-
120	1.93x10^5	1.12x10^5	1.57x10^5	3.07x10^4	1.73x10^5	1.39x10^5	1.60x10^5	5.71x10^3	1.52x10^5	1.18x10^5	1.34x10^5	1.23x10^4	-	-	-	-
135	1.69x10^5	1.02x10^5	1.44x10^5	2.52x10^4	1.82x10^5	1.29x10^5	1.52x10^5	2.07x10^4	1.61x10^5	1.14x10^5	1.41x10^5	1.81x10^4	-	-	-	-
150	1.64x10^5	1.11x10^5	1.45x10^5	2.18x10^4	1.55x10^5	1.39x10^5	1.47x10^5	7.84x10^3	1.66x10^5	1.36x10^5	1.49x10^5	1.13x10^4	-	-	-	-

* PC films were irradiated in the beam tube source for 180 minutes at the placement distance of 70 cm.
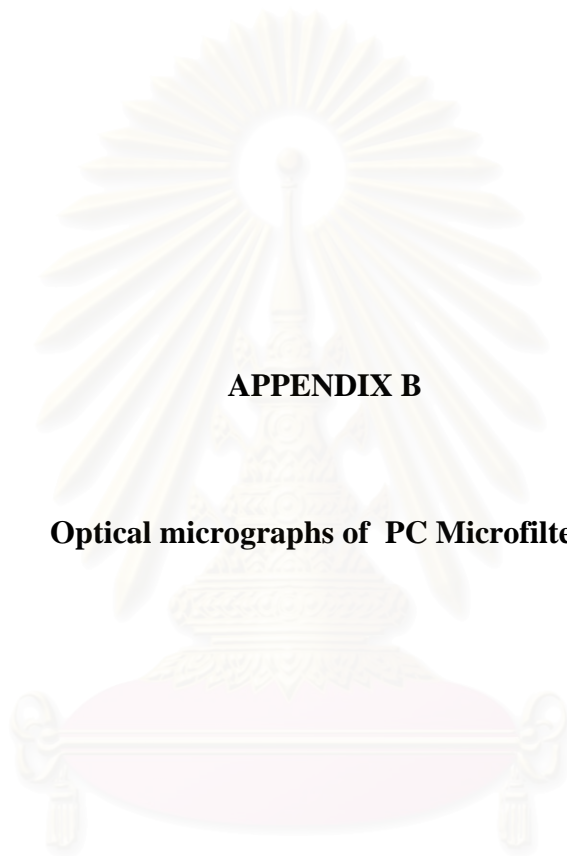
APPENDIX B

Optical micrographs of PC Microfilters
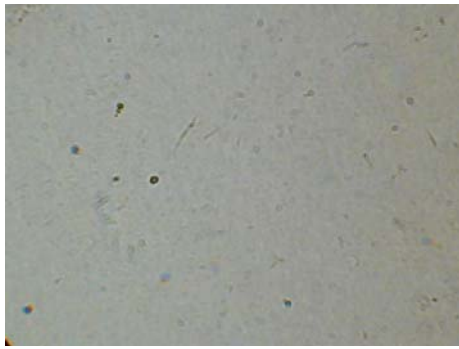
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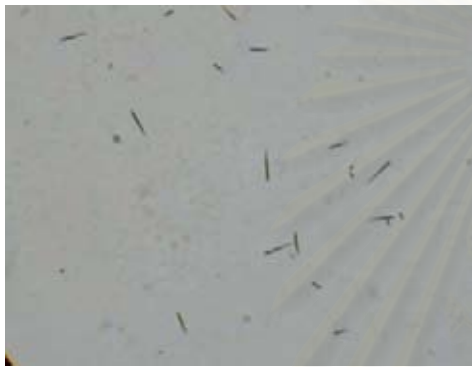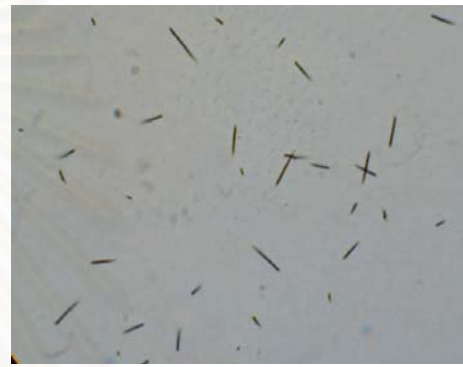
(c)　　　　　　　　　　　　(d)

(e)

Figure B.1　Optical micrographs of PC microfilters in the power of 100 times at

        (a)　2N NaOH 60 °C with 30 minutes

        (b)　2N NaOH 60 °C with 60 minutes

        (c)　2N NaOH 60 °C with 90 minutes

        (d)　2N NaOH 60 °C with 120 minutes

        (e)　2N NaOH 60 °C with 150 minutes.
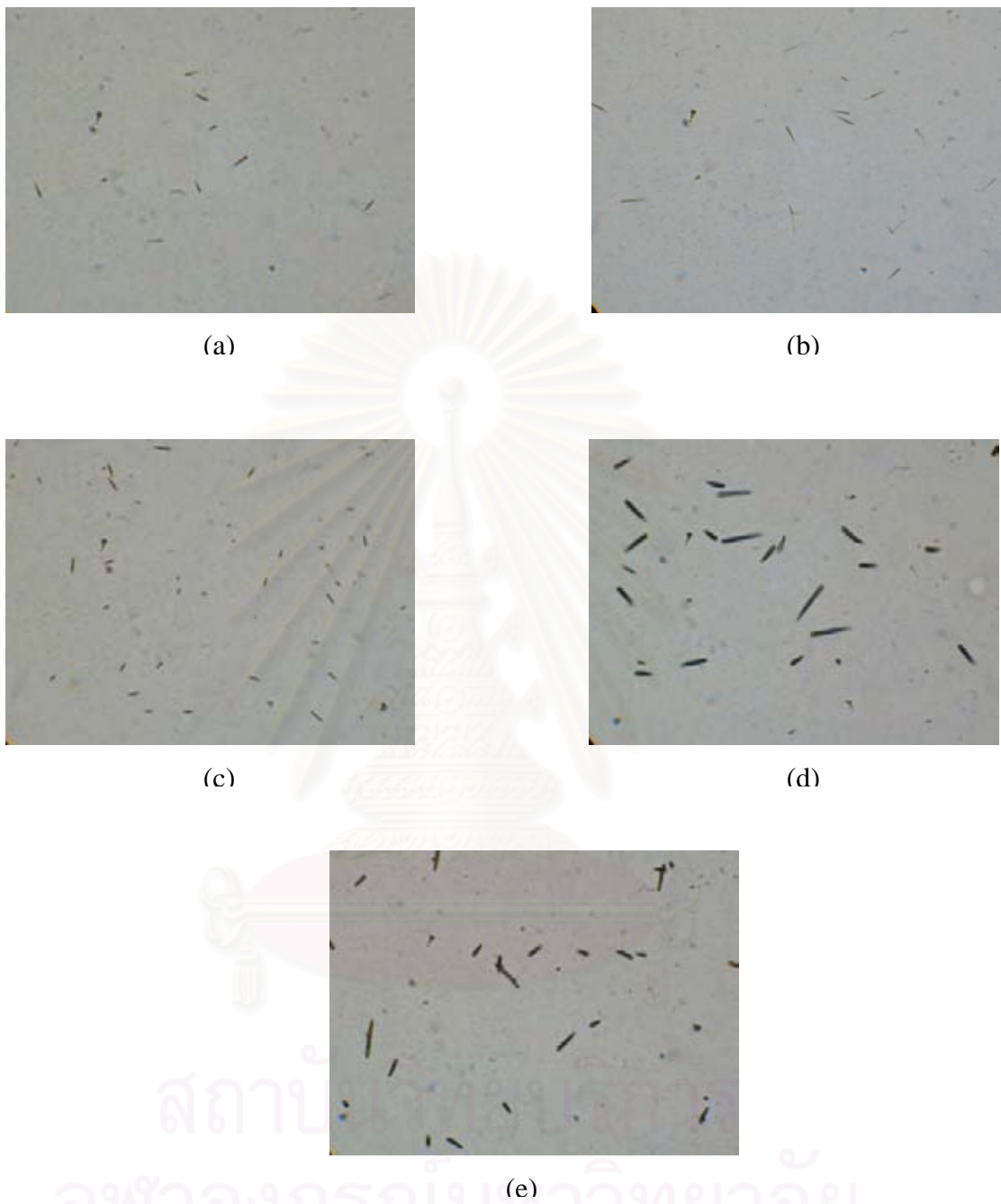
Figure B.2 Optical micrographs of PC microfilters in the power of 100 times at

(a) 2N NaOH 70 °C with 30 minutes
(b) 2N NaOH 70 °C with 60 minutes
(c) 2N NaOH 70 °C with 90 minutes
(d) 2N NaOH 70 °C with 120 minutes
(e) 2N NaOH 70 °C with 150 minutes.
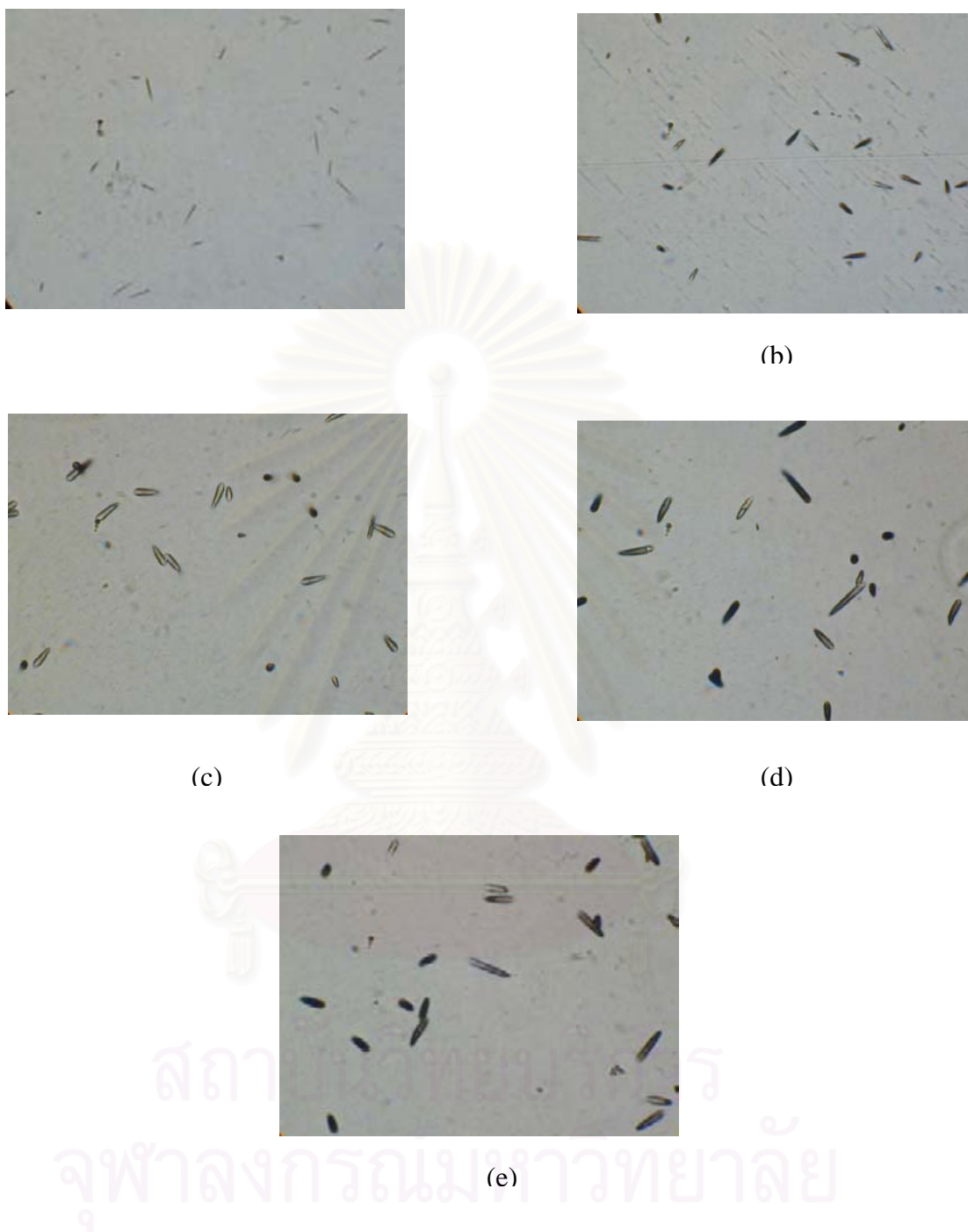
Figure B.3 Optical micrographs of PC microfilters in the power of 100 times at

(a) 2N NaOH 80 °C with 30 minutes
(b) 2N NaOH 80 °C with 60 minutes
(c) 2N NaOH 80 °C with 90 minutes
(d) 2N NaOH 80 °C with 120 minutes
(e) 2N NaOH 80 °C with 150 minutes.

(a)

(b)

(c)

(d)

(e)
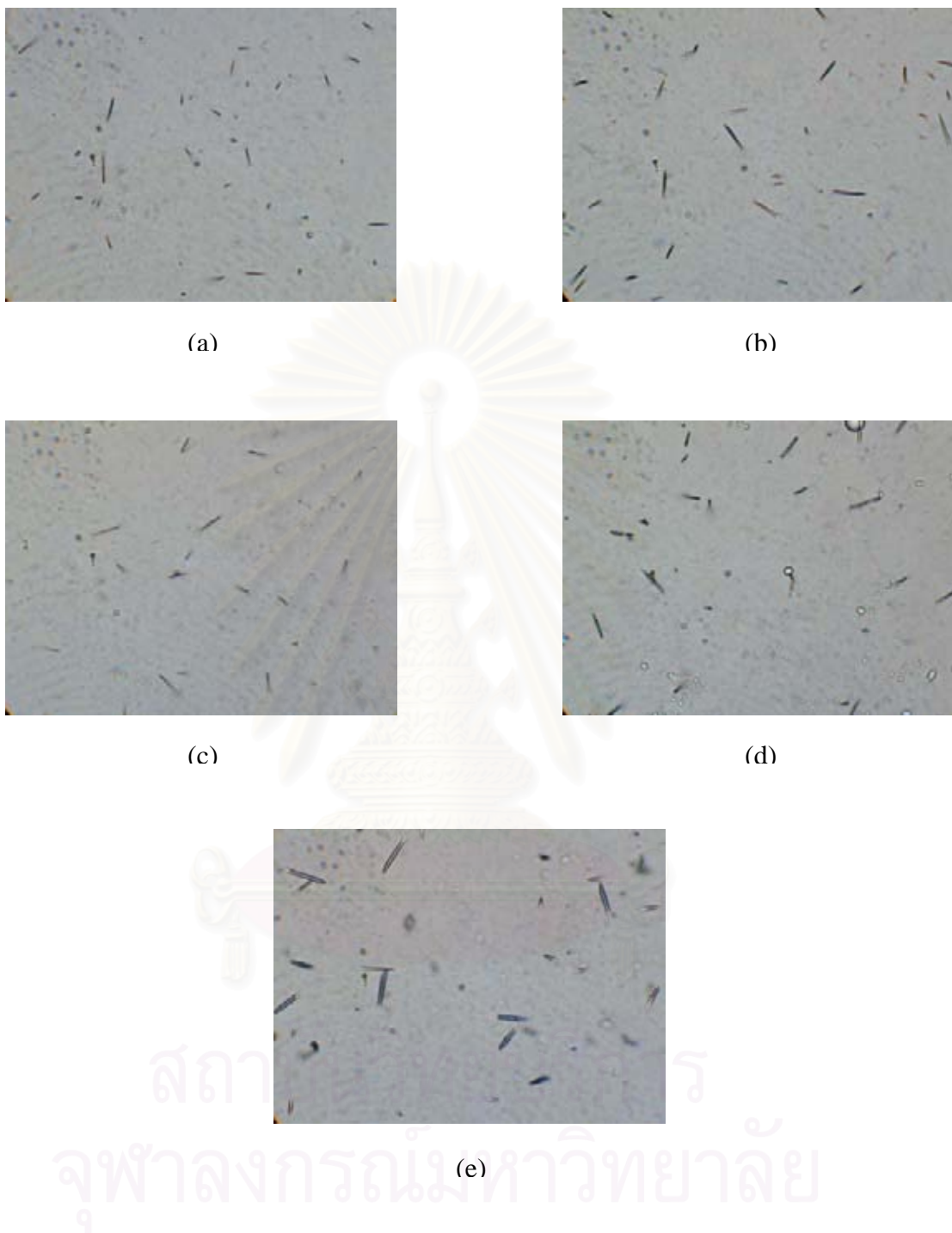
Figure B.4 Optical micrographs of PC microfilters in the power of 100 times at

(a) 4N NaOH 50 °C with 30 minutes
(b) 4N NaOH 50 °C with 60 minutes
(c) 4N NaOH 50 °C with 90 minutes
(d) 4N NaOH 50 °C with 120 minutes
(e) 4N NaOH 50 °C with 150 minutes.
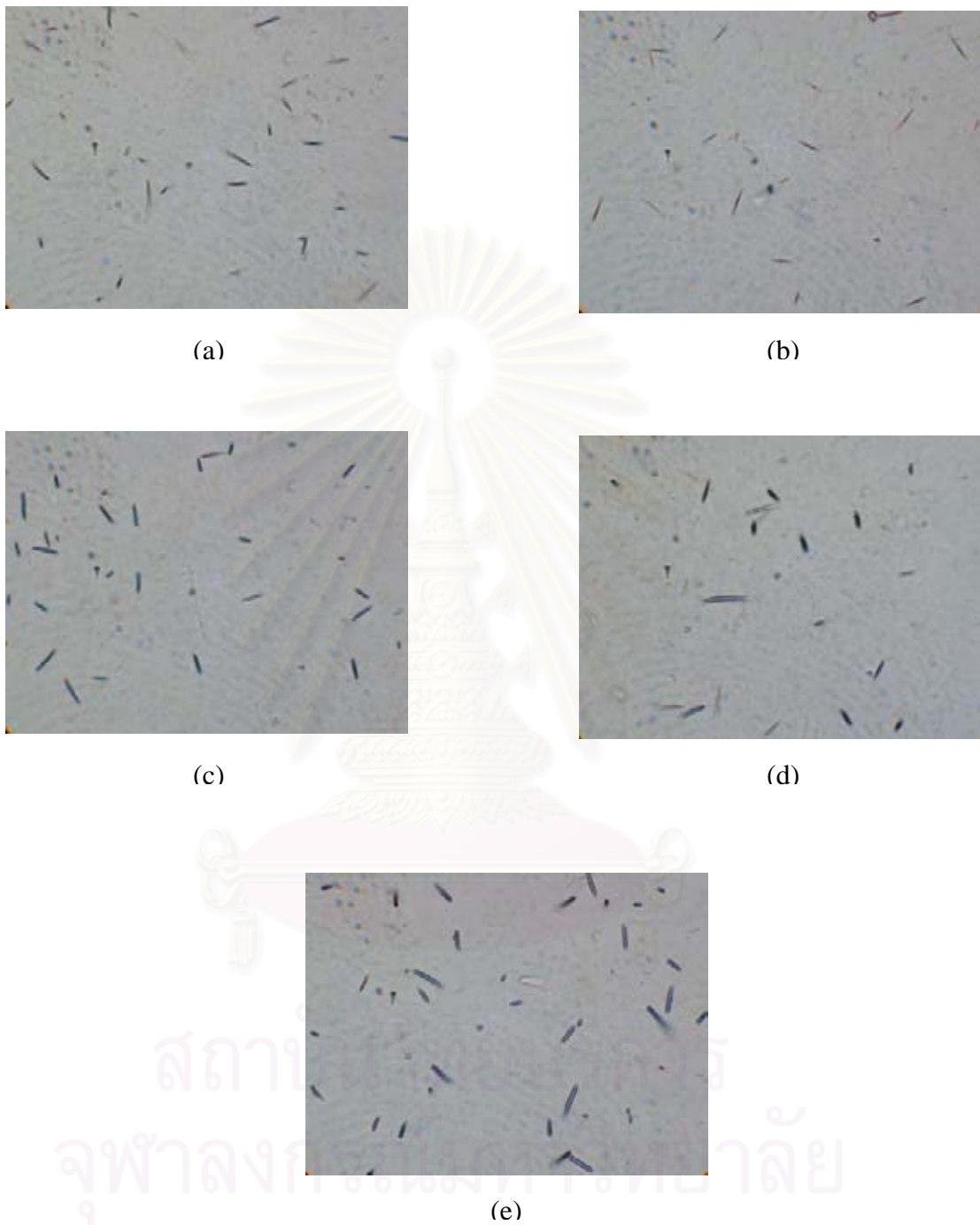
Figure B.5 Optical micrographs of PC microfilters in the power of 100 times at

(a) 4N NaOH 60 °C with 30 minutes
(b) 4N NaOH 60 °C with 60 minutes
(c) 4N NaOH 60 °C with 90 minutes
(d) 4N NaOH 60 °C with 120 minutes
(e) 4N NaOH 60 °C with 150 minutes.

(a)   (b)
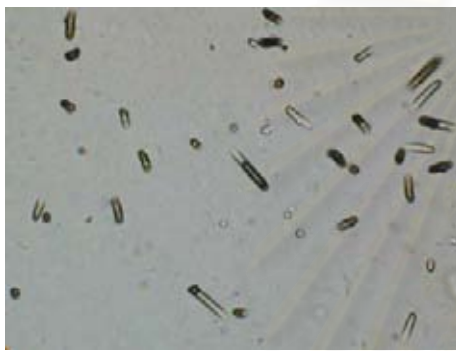
(c)   (d)
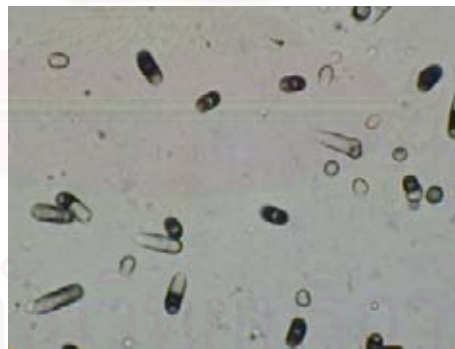
(e)

Figure B.6   Optical micrographs of PC microfilters in the power of 100 times at

(a) 4N NaOH 70 °C with 30 minutes
(b) 4N NaOH 70 °C with 60 minutes
(c) 4N NaOH 70 °C with 90 minutes
(d) 4N NaOH 70 °C with 120 minutes
(e) 4N NaOH 70 °C with 150 minutes.
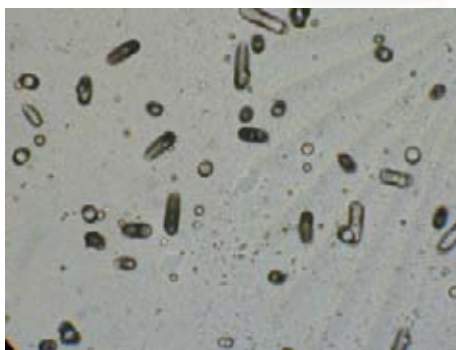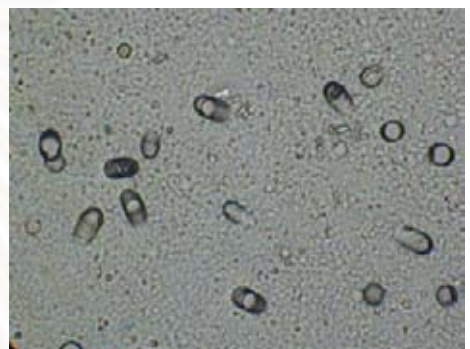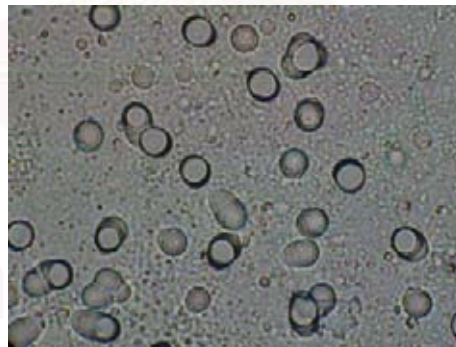
Figure B.7   Optical micrographs of PC microfilters in the power of 100 times at

(a) 4N NaOH 80 °C with 30 minutes
(b) 4N NaOH 80 °C with 60 minutes
(c) 4N NaOH 80 °C with 90 minutes
(d) 4N NaOH 80 °C with 120 minutes
(e) 4N NaOH 80 °C with 150 minutes.

(a)

(b)

(c)

(d)

(e)

Figure B.8 Optical micrographs of PC microfilters in the power of 100 times at

     (a) 6N NaOH 50 °C with 30 minutes
     (b) 6N NaOH 50 °C with 60 minutes
     (c) 6N NaOH 50 °C with 90 minutes
     (d) 6N NaOH 50 °C with 120 minutes
     (e) 6N NaOH 50 °C with 150 minutes.

(a)

(b)

(c)
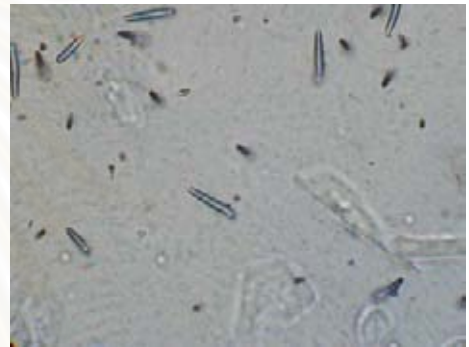
(d)

(e)

Figure B.9 Optical micrographs of PC microfilters in the power of 100 times at

(a) 6N NaOH 60 °C with 30 minutes
(b) 6N NaOH 60 °C with 60 minutes
(c) 6N NaOH 60 °C with 90 minutes
(d) 6N NaOH 60 °C with 120 minutes
(e) 6N NaOH 60 °C with 150 minutes.

(a)

(b)

(c)

(d)

(e)
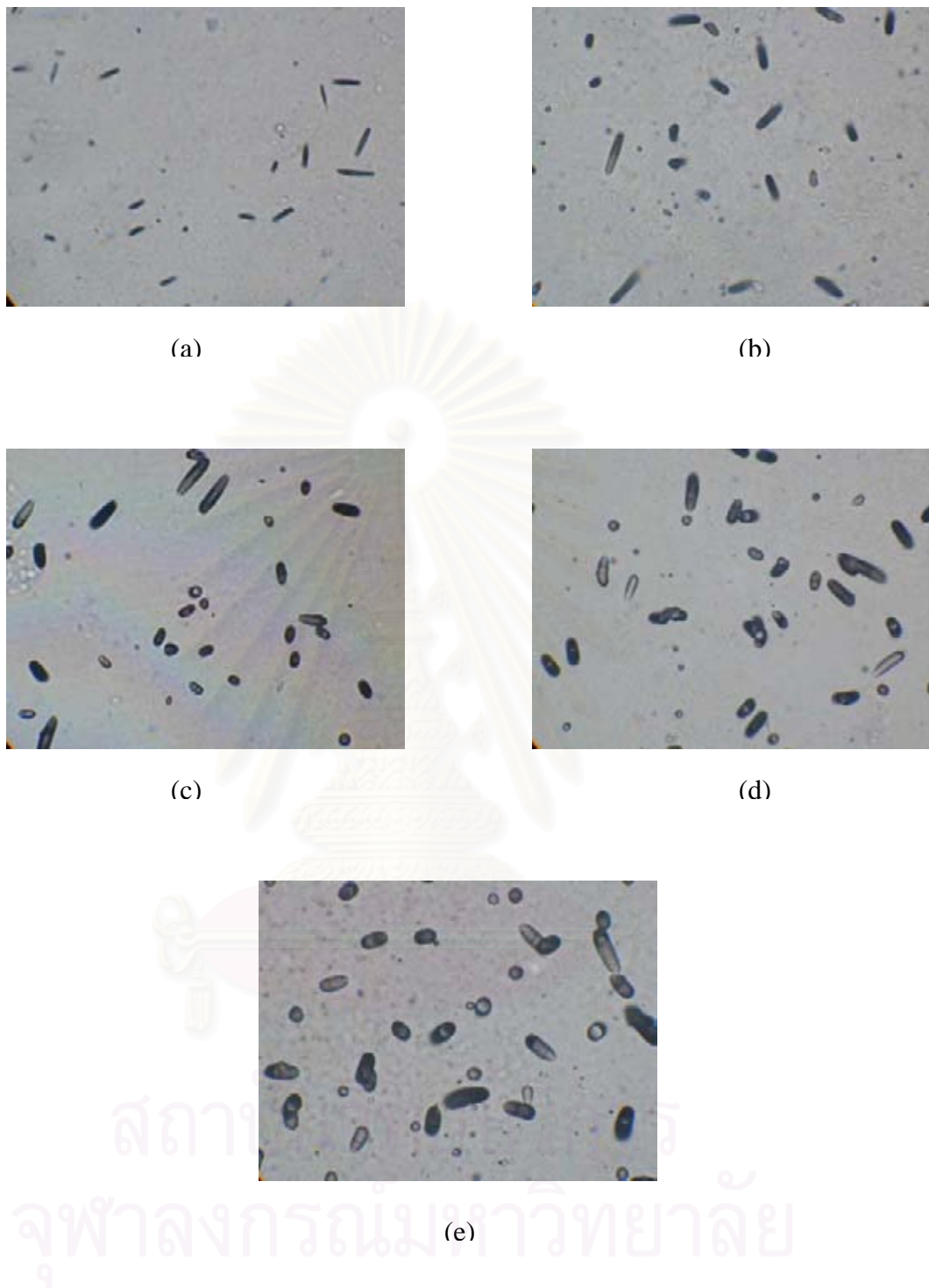
Figure B.10 Optical micrographs of PC microfilters in the power of 100 times at

(a) 6N NaOH 70 °C with 30 minutes
(b) 6N NaOH 70 °C with 60 minutes
(c) 6N NaOH 70 °C with 90 minutes
(d) 6N NaOH 70 °C with 120 minutes
(e) 6N NaOH 70 °C with 150 minutes.
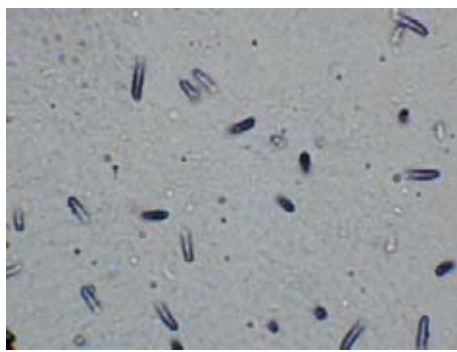
(a)
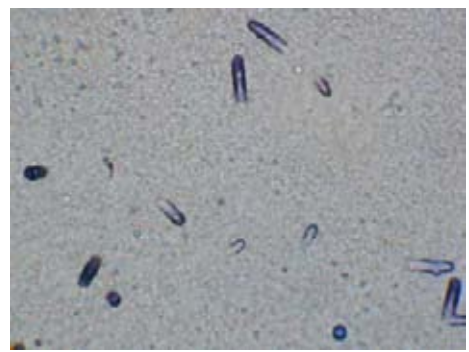
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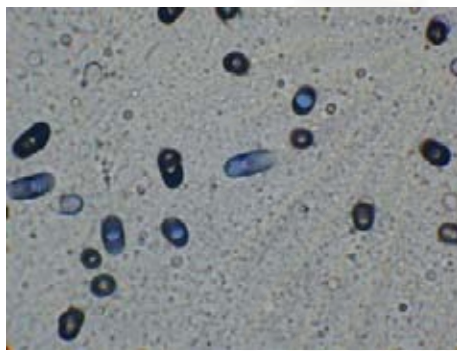
(c)
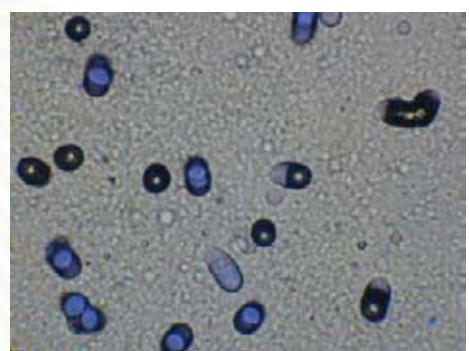
(d)
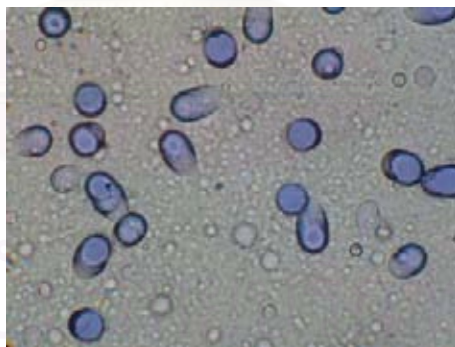
(e)

Figure B.11 Optical micrographs of PC microfilters in the power of 100 times at

(a) 6N NaOH 80 °C with 30 minutes
(b) 6N NaOH 80 °C with 60 minutes
(c) 6N NaOH 80 °C with 90 minutes
(d) 6N NaOH 80 °C with 120 minutes
(e) 6N NaOH 80 °C with 150 minutes.

(a)

(b)

(c)

(d)

(e)

Figure B.12 Optical micrographs of PC microfilters in the power of 100 times at

(a) 8N NaOH 50 °C with 30 minutes
(b) 8N NaOH 50 °C with 60 minutes
(c) 8N NaOH 50 °C with 90 minutes
(d) 8N NaOH 50 °C with 120 minutes
(e) 8N NaOH 50 °C with 150 minutes.

(a)

(b)

(c)

(d)

(e)

Figure B.13 Optical micrographs of PC microfilters in the power of 100 times at

    (a) 8N NaOH 60 °C with 30 minutes
    (b) 8N NaOH 60 °C with 60 minutes
    (c) 8N NaOH 60 °C with 90 minutes
    (d) 8N NaOH 60 °C with 120 minutes
    (e) 8N NaOH 60 °C with 150 minutes.
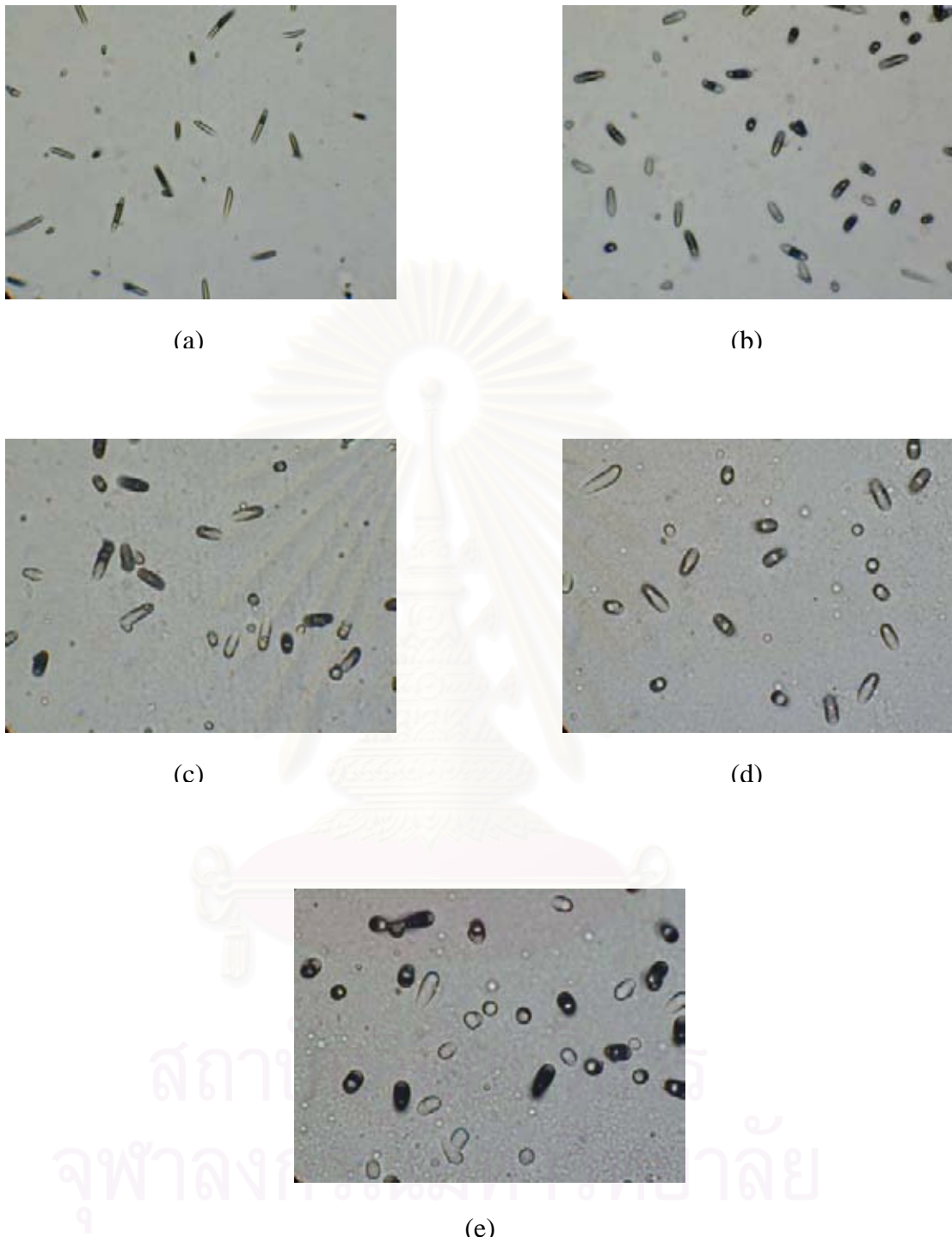
Figure B.14 Optical micrographs of PC microfilters in the power of 100 times at

(a) 8N NaOH 70 °C with 30 minutes
(b) 8N NaOH 70 °C with 60 minutes
(c) 8N NaOH 70 °C with 90 minutes
(d) 8N NaOH 70 °C with 120 minutes
(e) 8N NaOH 70 °C with 150 minutes.

(a)

(b)

(c)

(d)

(e)
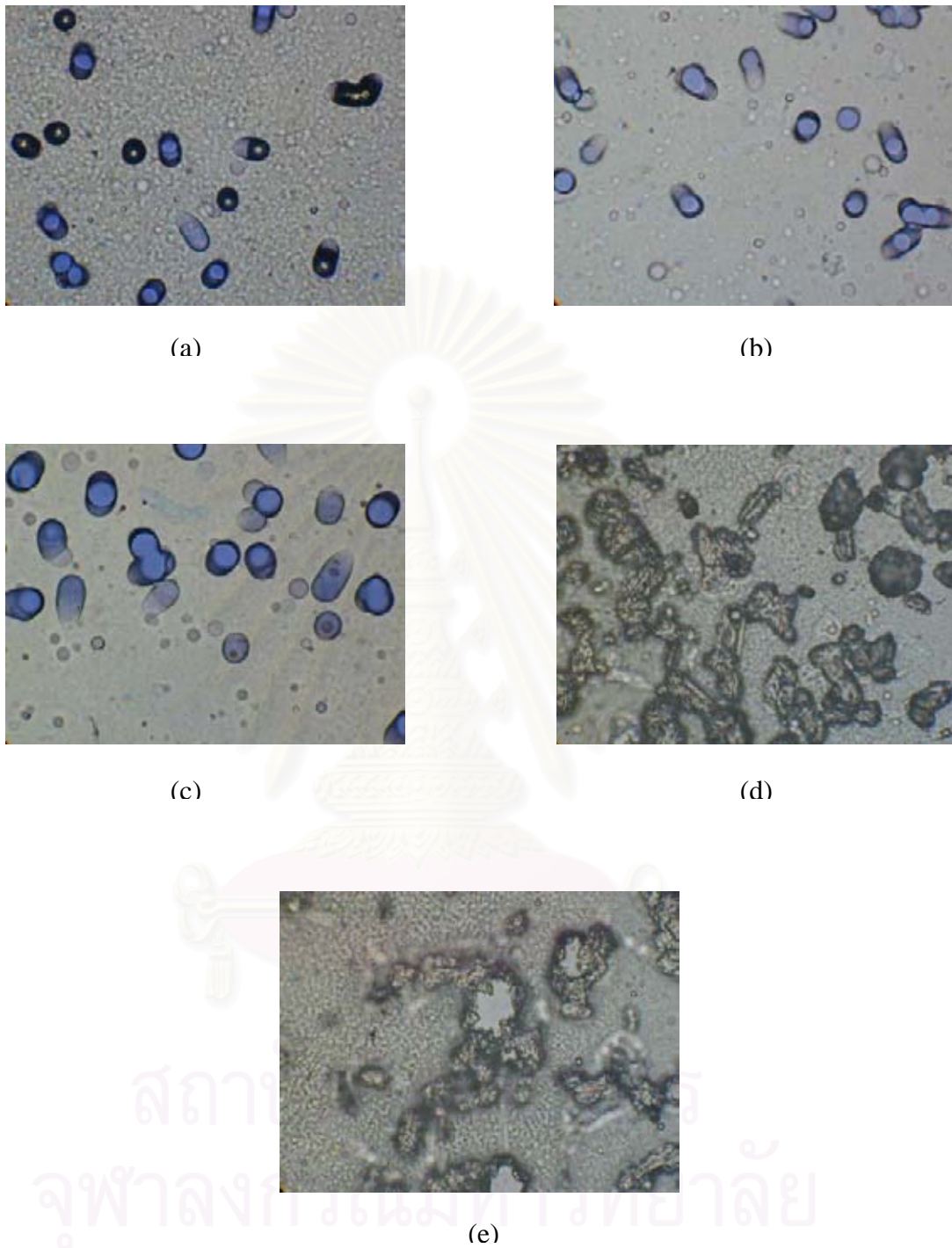
Figure B.15 Optical micrographs of PC microfilters in the power of 100 times at

(a) 8N NaOH 80 °C with 30 minutes
(b) 8N NaOH 80 °C with 60 minutes
(c) 8N NaOH 80 °C with 90 minutes
(d) 8N NaOH 80 °C with 120 minutes
(e) 8N NaOH 80 °C with 150 minutes.

Thickness of Track-etched PC Microfilter in Difference Etching Condition

Table C.1   Thickness of PC microfilters in difference etching concentration.

Etching condition*	Pore diameter (micron)	Pore density (pore/cm^2)	Thickness (micron)			SD
			Max	Min	Mean	
Virgin-PC	-	-	13	13	13	0.00
Tracking-PC	-	-	13	13	13	0.00
2N 60^{0}C 60 min	0.486	5.19x10^4	17	13	15	2.08
2N 70^{0}C 60 min	0.798	1.37x10^5	15	14	15	0.58
2N 80^{0}C 60 min	1.157	1.48x10^5	14	13	13	1.53
6N 60^{0}C 60 min	1.953	1.49x10^5	15	7	12	4.00
6N 70^{0}C 60 min	3.125	1.54x10^5	14	11	11	1.53
6N 80^{0}C 60 min	3.889	1.54x10^5	14	8	11	3.06
6N 80^{0}C 75 min	4.479	1.49x10^5	12	8	10	2.00
6N 80^{0}C 90 min	6.423	1.52x10^5	12	6	9	3.05
6N 80^{0}C 105min	7.813	1.55x10^5	9	6	8	2.08
6N 80^{0}C 120 min	9.514	1.54x10^5	8	4	7	2.31

*   PC films were irradiated at BT source for 180 minutes at the placement distance of 70 cm.

APPENDIX D

Chemical Modifications Induced in PC Microfilters

Table D.1 FTIR spectra of irradiated PC films in difference irradiation time.

Number	Irradiation time (min)	Length of BT(cm2)	Neutron flux (ion/s-1/cm-2)	Poredensity (pore/cm2)	Wave length (cm^{-1})	Normalized absorbances (A/A0)			
						Max	Min	Mean	SD
1	60	70	1.09×10^6	48,591	1012	0.972	0.962	0.969	0.005
					1510	0.975	0.945	0.958	0.014
					1782	0.879	0.846	0.861	0.017
					3500	1.183	1.148	1.169	0.018
2	120	70	1.09×10^6	101,513	1012	0.972	0.944	0.953	0.016
					1510	0.915	0.882	0.904	0.019
					1782	0.671	0.657	0.669	0.010
					3500	1.363	1.311	1.328	0.029
3	180	70	1.09×10^6	154,195	1012	0.897	0.878	0.885	0.010
					1510	0.857	0.821	0.844	0.019
					1782	0.504	0.477	0.492	0.014
					3500	1.541	1.496	1.519	0.022
4	240	70	1.09×10^6	195,136	1012	0.841	0.832	0.835	0.005
					1510	0.786	0.714	0.762	0.041
					1782	0.421	0.409	0.414	0.006
					3500	1.668	1.648	1.656	0.111

Table D.2 FTIR spectra of PC microfiters in difference etching condition.

Etching condition	Pore diameter (micron)	Pore density (pore/cm^2)	Wave number (cm^{-1})	Normalized absorbances (A/A_0)			SD
				Max	Min	Mean	
2N 60^{0}C 60 min	0.486	5.19x10^4	1012	0.6	0.596	0.598	0.002
			1782	0.407	0.392	0.399	0.007
			3500	1.663	1.637	1.647	0.014
2N 70^{0}C 60 min	0.798	1.04x10^5	1012	0.595	0.558	0.58	0.019
			1782	0.393	0.385	0.388	0.004
			3500	1.697	1.653	1.671	0.023
2N 80^{0}C 60 min	1.157	1.48x10^5	1012	0.553	0.544	0.548	0.004
			1782	0.376	0.358	0.369	0.009
			3500	1.771	1.75	1.763	0.011
6N 60^{0}C 60 min	1.953	1.50x10^5	1012	0.503	0.497	0.5	0.003
			1782	0.364	0.354	0.358	0.005
			3500	1.814	1.77	1.791	0.022
6N 70^{0}C 60 min	3.125	1.54x10^5	1012	0.479	0.457	0.468	0.011
			1782	0.349	0.333	0.341	0.008
			3500	1.878	1.832	1.857	0.023
6N 80^{0}C 60 min	3.889	1.54x10^5	1012	0.459	0.442	0.45	0.008
			1782	0.329	0.317	0.325	0.007
			3500	1.971	1.929	1.949	0.021
6N 80^{0}C 75 min	4.479	1.49x10^3	1012	0.436	0.42	0.427	0.007
			1782	0.329	0.318	0.324	0.005
			3500	2.001	1.977	1.998	0.018
6N 80^{0}C 90 min	6.423	1.52x10^5	1012	0.397	0.381	0.391	0.008
			1782	0.297	0.272	0.285	0.012
			3500	2.066	2.044	2.055	0.018
6N 80^{0}C 105min	7.813	1.55x10^5	1012	0.335	0.32	0.326	0.007
			1782	0.277	0.246	0.261	0.016
			3500	2.071	2.042	2.061	0.017
6N 80^{0}C 120 min	9.514	1.54x10^5	1012	0.286	0.257	0.273	0.014
			1782	0.163	0.152	0.156	0.006
			3500	2.114	2.089	2.101	0.012

APPENDIX E

Water Permeability Tester of PC Microfilter

Table E.1 Water flux of PC microfiters in difference etching condition.

Etching condition*	Pore diameter (micron)	Pore density (pore/cm^2)	Water flux (ml/min/cm^2)			SD
			Max	Min	Mean	
Virgin-PC	-	-	-	-	-	-
Tracking-PC	-	-	-	-	-	-
2N 60^{0}C 60 min	0.486	5.19x10^4	-	-	-	-
2N 70^{0}C 60 min	0.798	1.37x10^5	-	-	-	-
2N 80^{0}C 60 min	1.157	1.48x10^5	-	-	-	-
6N 60^{0}C 60 min	1.953	1.49x10^5	2.411x10^{-4}	1.447x10^{-4}	1.875x10^{-4}	4.911x10^{-5}
6N 70^{0}C 60 min	3.125	1.54x10^5	1.857x10^{-3}	1.326x10^{-3}	1.591x10^{-3}	2.652x10^{-4}
6N 80^{0}C 60 min	3.889	1.54x10^5	5.305x10^{-3}	4.243x10^{-3}	4.951x10^{-3}	6.125x10^{-4}
6N 80^{0}C 75 min	4.479	1.49x10^5	1.087x10^{-2}	5.434x10^{-3}	8.089x10^{-3}	2.721x10^{-3}
6N 80^{0}C 90 min	6.423	1.52x10^5	1.379x10^{-2}	1.220x10^{-2}	1.305x10^{-2}	8.010x10^{-4}
6N 80^{0}C 105min	7.813	1.55x10^5	2.122x10^{-2}	1.867x10^{-2}	1.966x10^{-2}	1.364x10^{-3}
6N 80^{0}C 120 min	9.514	1.54x10^5	3.045x10^{-2}	2.896x10^{-2}	3.055x10^{-2}	1.647x10^{-3}

*   PC films were irradiated at BT source for 180 minutes at the placement distance of 70 cm.

APPENDIX F

Mechanical Properties of PC Microfilter in Difference Etching Condition

Table F.1 Tensile strength of PC microfilters in difference etching concentration.

Etching condition*	Pore diameter (micron)	Pore density (pore/cm^2)	Tensile strength (Mpa)			SD
			Max	Min	Mean	
Virgin-PC	-	-	14.92	12.13	13.37	1.04
Tracking-PC	-	-	12.27	11.34	11.86	0.378
2N 60^{0}C 60 min	0.486	51,943	11.34	10.43	11.05	0.37
2N 70^{0}C 60 min	0.798	103,670	11.50	10.32	10.88	0.53
2N 80^{0}C 60 min	1.157	148,212	10.62	8.52	9.62	0.78
6N 60^{0}C 60 min	1.953	149,576	9.23	8.65	8.96	0.25
6N 70^{0}C 60 min	3.125	154,195	8.27	7.37	7.85	0.41
6N 80^{0}C 60 min	3.889	154,337	6.45	6.03	6.24	0.16
6N 80^{0}C 75 min	4.479	149,103	6.29	5.56	5.91	0.27
6N 80^{0}C 90 min	6.423	151,580	5.30	4.78	4.90	0.22
6N 80^{0}C 105min	7.813	155,469	4.65	3.22	4.07	0.61
6N 80^{0}C 120 min	9.514	154,025	3.22	1.56	2.58	0.64

*   PC films were irradiated at BT source for 180 minutes at the placement distance of 70 cm.

Table F.2 Yield strength of PC microfilters in difference etching concentration.

Etching condition	Pore diameter (micron)	Pore density (pore/cm^2)	Yield strength (Mpa)			SD
			Max	Min	Mean	
Virgin-PC	-	-	68.58	58.48	62.69	4.64
Tracking-PC	-	-	61.86	57.02	59.00	2.23
2N 60^{0}C 60 min	0.486	5.19x10^4	60.85	55.87	57.92	1.88
2N 70^{0}C 60 min	0.798	1.37x10^5	55.23	52.16	54.05	1.17
2N 80^{0}C 60 min	1.157	1.48x10^5	51.02	47.55	48.94	1.32
6N 60^{0}C 60 min	1.953	1.49x10^5	47.02	45.24	45.93	0.69
6N 70^{0}C 60 min	3.125	1.54x10^5	45.78	40.11	43.94	2.29
6N 80^{0}C 60 min	3.889	1.54x10^5	37.98	35.21	36.82	2.08
6N 80^{0}C 75 min	4.479	1.49x10^5	31.18	29.41	30.62	0.72
6N 80^{0}C 90 min	6.423	1.52x10^5	30.21	25.43	27.94	1.79
6N 80^{0}C 105min	7.813	1.55x10^5	24.26	21.03	22.85	1.16
6N 80^{0}C 120 min	9.514	1.54x10^5	23.56	17.24	20.47	2.76

*   PC films were irradiated at BT source for 180 minutes at the placement distance of 70 cm.

Table F.3 Modulus of PC microfilters in difference etching concentration.

Etching condition*	Pore diameter (micron)	Pore density (pore/cm^2)	Modulus (Mpa)			SD
			Max	Min	Mean	
Virgin-PC	-	-	1,594.23	1,374.70	1,490.29	78.13
Tracking-PC	-	-	1,488.9	1,394.71	1,447.86	45.67
2N 60^{0}C 60 min	0.486	5.19x10^4	1,258.45	1,198.56	1,231.49	25.13
2N 70^{0}C 60 min	0.798	1.37x10^5	1,168.26	1,058.45	1,111.77	39.85
2N 80^{0}C 60 min	1.157	1.48x10^5	1,104.28	1,016.15	1,062.41	37.91
6N 60^{0}C 60 min	1.953	1.49x10^5	9,25.63	878.69	906.19	19.85
6N 70^{0}C 60 min	3.125	1.54x10^5	840.39	789.49	809.53	21.95
6N 80^{0}C 60 min	3.889	1.54x10^5	634.95	583.40	608.50	37.61
6N 80^{0}C 75 min	4.479	1.49x10^5	613.86	541.83	569.39	28.18
6N 80^{0}C 90 min	6.423	1.52x10^5	533.93	495.23	512.58	14.03
6N 80^{0}C 105min	7.813	1.55x10^5	489.78	381.23	446.87	37.75
6N 80^{0}C 120 min	9.514	1.54x10^5	415.91	339.65	385.24	28.79

*   PC films were irradiated at BT source for 180 minutes at the placement distance of 70 cm.
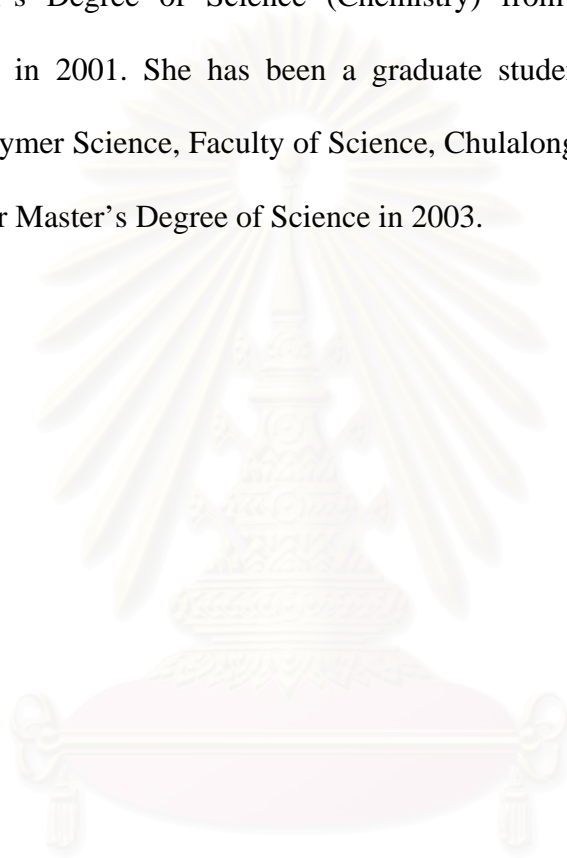
Table F.4 % Elongation of PC microfilters in difference etching concentration.

Etching condition*	Pore diameter (micron)	Pore density (pore/cm^2)	% Elongation			SD
			Max	Min	Mean	
Virgin-PC	-	-	7.81	6.85	7.13	0.39
Tracking-PC	-	-	8.16	6.98	7.73	0.47
2N 60^{0}C 60 min	0.486	5.19x10^4	11.59	10.43	11.23	0.48
2N 70^{0}C 60 min	0.798	1.37x10^5	13.48	12.36	12.98	0.47
2N 80^{0}C 60 min	1.157	1.48x10^5	15.57	14.75	15.18	0.36
6N 60^{0}C 60 min	1.953	1.49x10^5	15.93	15.05	17.47	0.37
6N 70^{0}C 60 min	3.125	1.54x10^5	23.16	22.56	22.89	0.24
6N 80^{0}C 60 min	3.889	1.54x10^5	24.38	23.84	24.15	0.24
6N 80^{0}C 75 min	4.479	1.49x10^5	26.98	26.13	26.68	0.52
6N 80^{0}C 90 min	6.423	1.52x10^5	29.04	28.38	28.83	0.26
6N 80^{0}C 105min	7.813	1.55x10^5	32.12	32.65	32.68	0.57
6N 80^{0}C 120 min	9.514	1.54x10^5	36.51	35.12	35.73	0.52

  *   PC films were irradiated at BT source for 180 minutes at the placement distance of 70 cm.

VITAE

Pornphan Makphon was born on August 2, 1979 in Khon Kaen, Thailand. She received the Bachelor's Degree of Science (Chemistry) from Faculty of Science, Thammasat University in 2001. She has been a graduate student in the Program of Petrochemistry and Polymer Science, Faculty of Science, Chulalongkorn University since 2001 and completed her Master's Degree of Science in 2003.