

Etching and technical parameter of polymer film for manufacture of nuclear track membrane

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Abstract

The purpose of the research was to investigate the etching and the technical parameter of polymer films for the manufacture of nuclear track membrane. The polycarbonate film was stored in the thermal column tube and the irradiated neutron at different time. The result shown the optimized time to irradiate neutron of polycarbonate was 20 seconds. Two types of polymer (polycarbonate and polyethylene terephthalate) were the irradiated neutron and the irradiated film which were etched in the chemical etchant by the sodium hydroxide (NaOH) and the potassium hydroxide (KOH) respectively. The technical parameters etched track all polymer films which were investigated by the optical microscopy. Besides that, it was found that the optimized time, the temperature of chemical and the concentration of chemical etched track on two types of polymer film that were 60 and 60 minutes, 60 and 60 °C and 6 and 4 mol/L respectively. At this condition, two types of polymer film were measured by track size. The results found that track size of polymer had about 1 to 2 micrometer. The experimental results indicated that the nuclear track could be used in the filter membrane industries.

Keywords : etching track, nuclear fission, sodium hydroxide, potassium hydroxide

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Introduction

Membrane filters were widely used in the filtration manufacture. The polymer filters were the interested membrane which produced membrane filter. The membrane filter could be produced through many techniques (Husaini *et al.*, 2008); the stretch membrane, the phase inversion, the skin casting, and the nuclear track etc. The nuclear track was the popular technique in developing new techniques of material surfaces for changing their surface properties according to requirement applications (Awasthi *et al.*, 2014). Track or pore size on the membrane was more difficult to control and was affected for mass separation. This technique was extremely interesting among researches due to controlling of track size on membrane filter (Vijay, 2008). One of membrane filters was polymer which had found a broad range of applications in the fields (Kaur *et al.*, 2012). Polycarbonate (PC) and polyethylene terephthalate (PET) were the most commercially available resistant polymers because of they had the chemical inertness, the thermal stability and the good mechanical properties (Klintberg *et al.*, 2001). In the production process of polymer filter, it was much chemical solution etching. Therefore, technical parameters of etching were important to control track and pore size on polymer. In the research, we investigated the technical parameters of polymer films for manufacturing

of the nuclear track membrane. Technical parameters were etching time, temperature and concentration of chemical solution. The characteristic of track and pore size on polymer could be applied for the filtration manufacture.

Materials

In the research, two types of polymer film were selected and studied; the polycarbonate film and the polyethylene terephthalate film. The polycarbonate film of thickness 6 μm was purchased from the Good fellow Cambridge limited and the polyethylene terephthalate film of thickness 100 μm was purchased from the SPEX Sample Prep Company. The sample size, 1.40x6.50 cm^2 , pasted on the Uranium screen and be inputted in rabbit holder.

Sample preparation

The polycarbonate films were irradiated by the neutron from U^{235} of a specific energy at $1.0 \times 10^9 \text{ n/cm}^2 \text{ s}^{-1}$ of the Office of atomic energy for peace (OAEP), Thailand. The samples were irradiated at different times from 10 to 50 s with an increment of 10 s. The samples were etched by 6 mol/L of sodium hydroxide (NaOH) solution for investigating on the optimum time of irradiation. The optimum parameter, such as etching time, solution temperature and solution concentration were investigated on etching

track polymer membrane. Polycarbonate and polyethylene terephthalate film were irradiated with U^{235} and were etched by 6 mol/L sodium hydroxide (NaOH) and 4 mol/L of potassium hydroxide (KOH), respectively in the water bath. The solution etching was used dependent on the thickness of polymer. The time for etching and temperature of solution etching were varied from 30 to 80 min and 40 to 80 °C, respectively. The concentration of NaOH and KOH solution were investigated from 4 to 8 mol/L and 2 to 6 mol/L, respectively. All samples were washed with distilled water, 5% of ethanol, soaping water and taped water. The optimum parameters (etching time, solution temperature and solution concentration) of samples were characterized from micrograph of track etched on polymer by the optical microscopy (OM).

Results and discussion

Optimization irradiation time

The polycarbonate films were irradiated neutron at different times shown in (Figure 1). (Figure 1a) showed the OM micrograph of polycarbonate film before bombardment with the neutron and etching with the NaOH solution. The surface of polycarbonate film was smooth and disappeared on any track and pore. After bombardment with neutron, tracks and pores were appeared on the surface of polycarbonate film as

presented in (Figure 1 b-f). The relationships between number of track and irradiation time were shown in (Figure 2). The track density on the polycarbonate film was increased with the increasing irradiation time. Over the long period of time of irradiation, small tracks were connected and due to the large track as shown in (Figure 1e) and (Figure 1f). The track density in the (Figure 1b) had weakness. The number of track per 0.01053 mm^3 in the (Figure 1) were determined by image J program as shown in Table 1 and (Figure 2). The different number of track between 20 s and 30 s was 29 that were small increased and no significant. From the results indicated that the optimum irradiation time was 20 s.

Optimization parameter etching:

Etching time

The OM micrographs of both samples were irradiated the neutron at 20 s and etched at different times from 30 to 80 min with an increment of 10 min presented in (Figure 3). The concentration of NaOH and KOH solution were controlled by 6 mol/L and 4 mol/L at 60 °C, respectively. The numbers and sizes of tracks on both polymers were determined and shown in (Figure 4). The results were shown that the size of track was increased with the increasing etching time as a linear. This indicated that

etching solution could be etched track on the polymer film (Traumann *et al.*, 1996). In the microfiltration manufacture of bacteria and colloid emulsion, track size of filter membrane was ranged 1.00 to 2.00 μm (Apel, 2001). The track size was more than 1.00 μm , had to be etching time that was higher than 50 min as shown in (Figure 4). The polycarbonate had track size 1.29 and 1.46 μm and the polyethylene

terephthalate had track size 1.30 and 1.49 μm for etching time at 60 and 70 min, respectively. The both etching time had different track sizes which no significantly. Therefore, 60 min was optimum for etching time because track size of both polymer films was around 1.30 μm . These results were confirmed with the distribution track on both polymers as shown in (Figure 3)

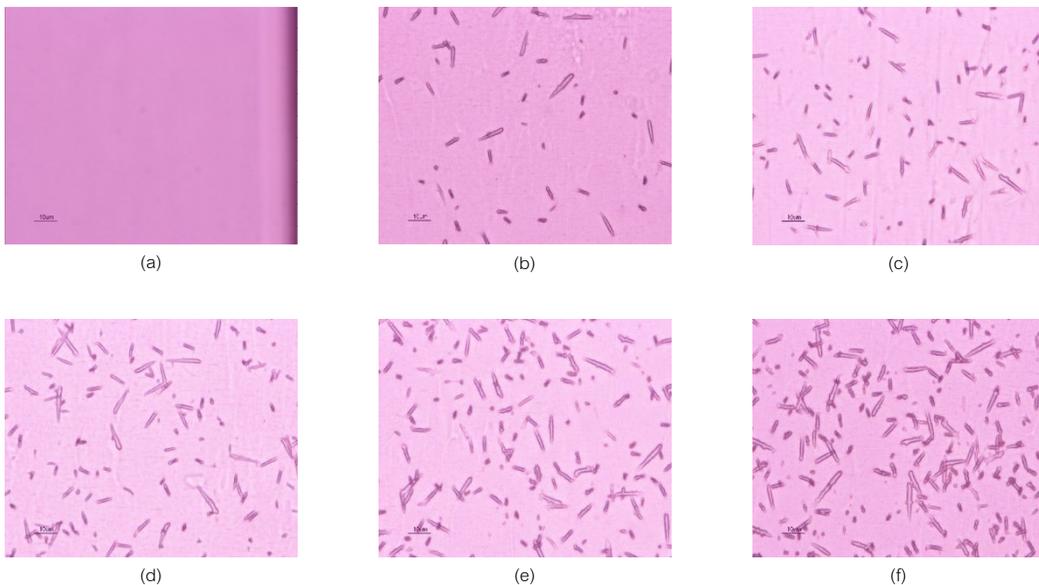
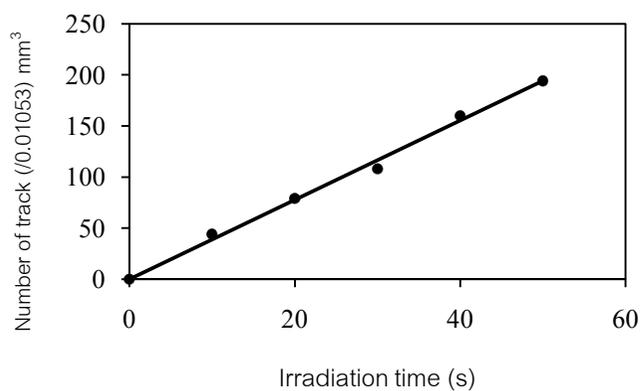


Figure 1 The optical microscope (Nikon 80i) X1000 of polycarbonate thin film surface irradiated neutron at different times;(a) before irradiate neutron, (b) 10 s, (c) 20 s (d), 30 s (e), 40 s, (f) 50 s.

Table 1 The number of track and track density of polycarbonate film irradiated neutron at different times.

polycarbonate films	irradiation time (s)	number of track (per 0.01053 mm ³)	the track density (track/mm ³)
1	10.00	44	4178.54
2	20.00	79	7502.37
3	30.00	108	10256.41
4	40.00	160	15194.68
5	50.00	194	18423.55

**Figure 2** The relationships between numbers of track and the irradiation time.

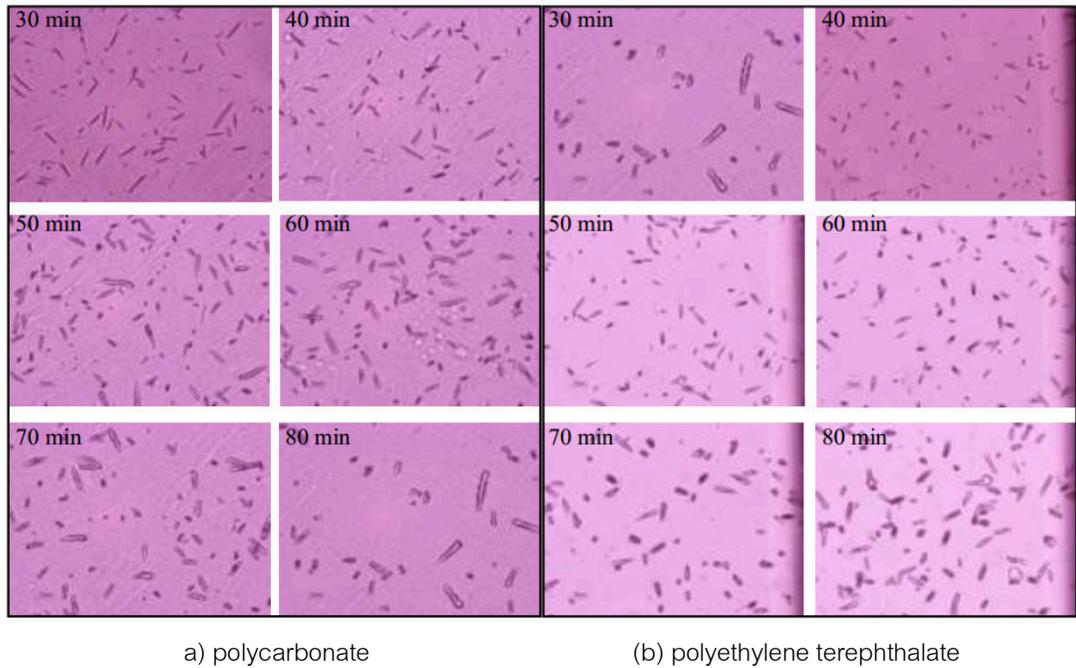


Figure 3 The comparison of the optical microscope (Nikon 80i) X1000 of the polycarbonate and the polyethylene terephthalate thin film surface at different times of etching.

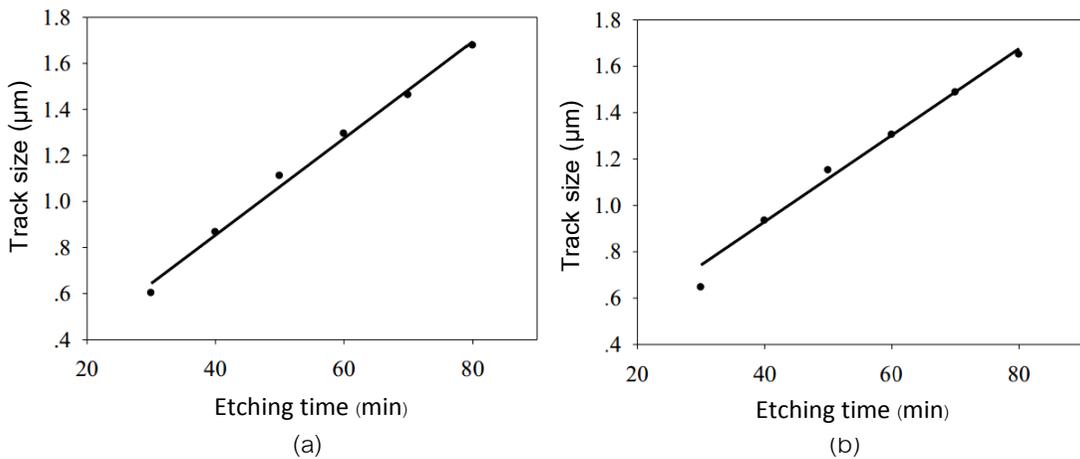


Figure 4 The relationships between track size and etching time; (a) polycarbonate, (b) polyethylene terephthalate.

Temperature of solution

The polycarbonate and polyethylene terephthalate films were bombardment with the neutron at 20 s and etched by the 6 mol/L of NaOH and 4 mol/L of KOH solution for 60 min, respectively. The temperatures of solution were investigated from 40 to 80 °C with an increment of 10 °C. The OM micrographs and track sizes of both polymer films at different temperatures of solution as shown in (Figure 5) and (Figure 6).

The results were shown that track sizes of both polymer films were extended rapidly. On the other hand, both polymer films were damaged when the samples were etched at the high temperature of solution. Moreover, the optimum temperature of solution of etching the polycarbonate and polyethylene terephthalate films was 60 °C while track sizes of polymer films were about 1.24 and 1.64 μm respectively.

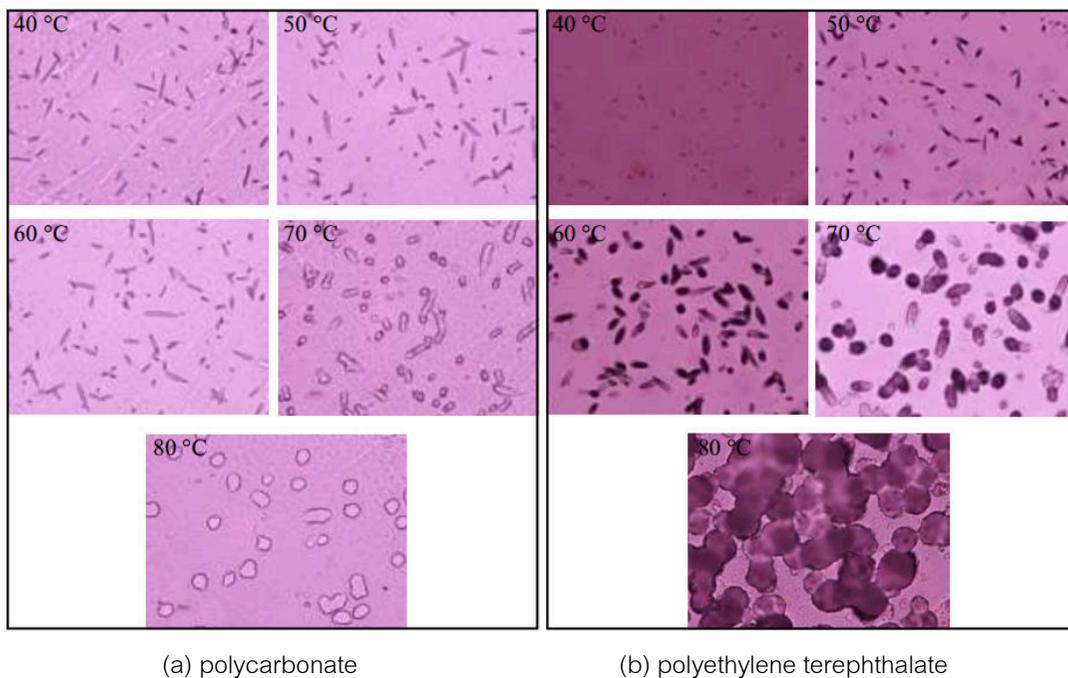


Figure 5 The comparison of optical microscope (Nikon 80i) X1000 of the polycarbonate and the polyethylene terephthalate thin film surface at different temperatures of etching.

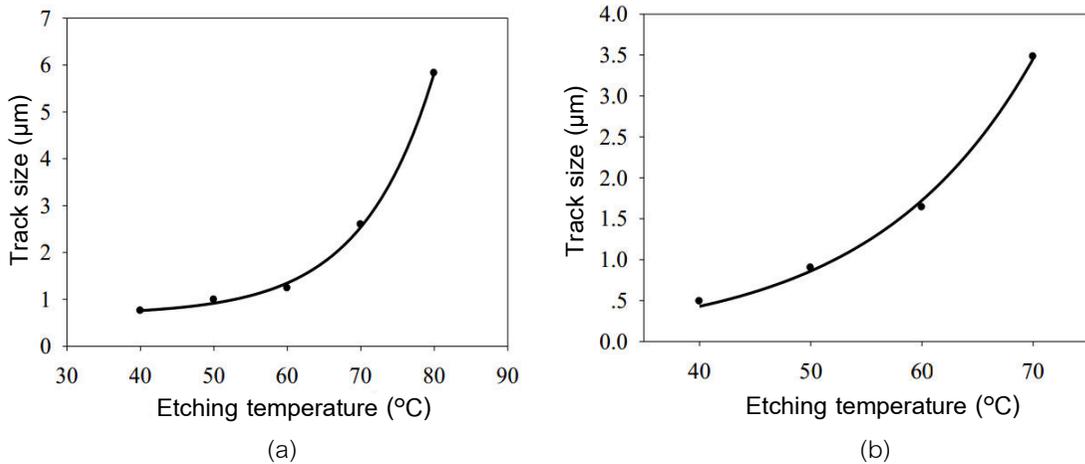


Figure 6 The relationships between track size and etching temperature; (a) polycarbonate, (b) polyethylene terephthalate.

Concentration of solution

Polycarbonate and polyethylene terephthalate films were bombardment with the neutron 20 s and etched by the NaOH and KOH solution at different concentrations from 4.0 to 8.0 mol/L and 2.0 to 6.0 mol/L with an increment of 1.0 mol/L, respectively. The both solution were controlled temperature at 60 °C and etched time for 60 min. The OM micrographs of both polymer films were shown in (Figure 7). It was observed that track size of both polymer films etched at the low concentration of solution was

smaller than others while both polymer films were damaged at the high concentration of solution. From (Figure 7), it could be determined track size as presented in (Figure 8). The track sizes of both polymer films were increased with increasing concentration of solution. Track size was about 1.48 µm which the good track size could be applied to use in microfiltration industries (Adach *et al.*, 2002). Moreover, concentration of solution etched the polycarbonate and the polyethylene terephthalate films were 6.0 and 4.0 mol/L respectively.

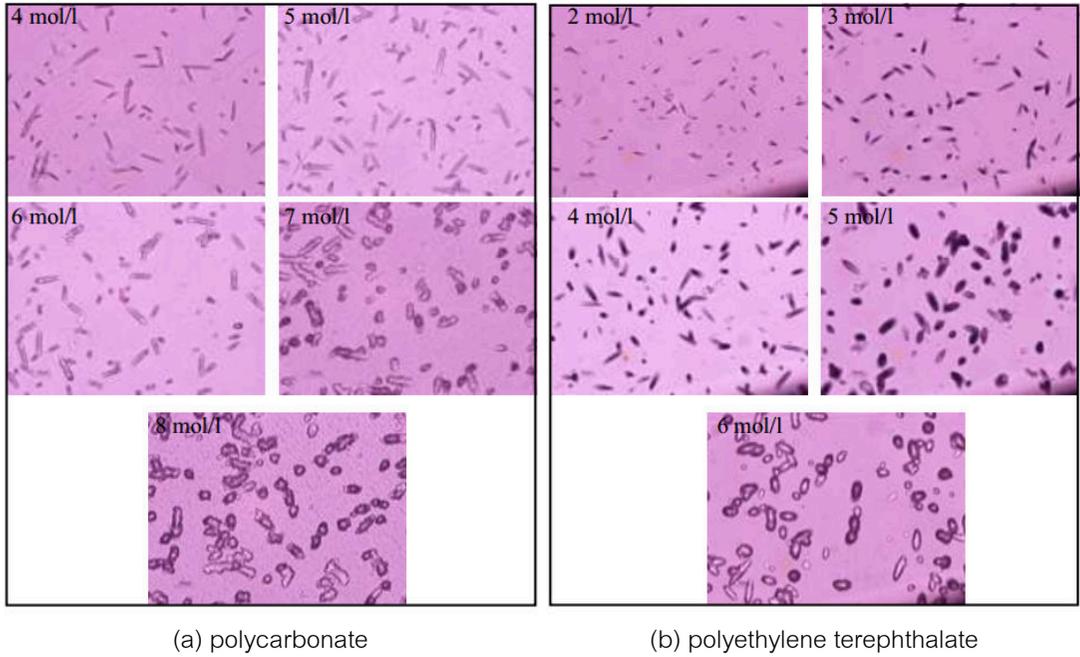


Figure 7 The comparison of optical microscope (Nikon 80i) X1000 of the polycarbonate and the polyethylene terephthalate thin film surface at different concentrations of etching.

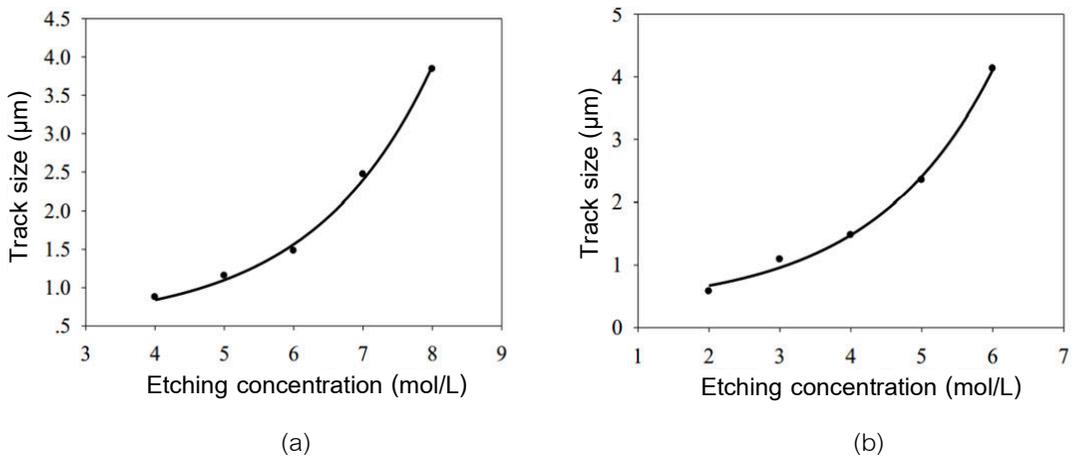


Figure 8 The relationships between track size and etching concentration; (a) polycarbonate, (b) polyethylene terephthalate.

From the experiment, the results were shown which were affected on track size of polymer the etching time, temperature and concentration film. The etching parameter of etching track on of the chemical solution for etching polymer film both polymer films which were shown in Table 2.

Table 2 The optimum parameter (time, temperature and concentration) etching track on both polymers.

type of polymer	chemical solution	time (min)	temperature (°C)	concentration (mol/L)
polycarbonate	NaOH	60.00	60.00	6.00
polyethylene terephthalate	KOH	60.00	60.00	4.00

Conclusion

Track on polycarbonate and polyethylene terephthalate films was produced by using the neutron bombardment. It had been found that etching time, temperature and concentration of chemical solution were affected on track size of polymer film. However, track size on the polymer film could be used at different industries.

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