CHAPTER III

EXPERIMENTAL PROCEDURE AND DEVELOPMENT

This chapter describes the design of experiments on producing proton tracks in polymer as well as related experiments and associated measurements to study the properties of track-tech polymer on light transmission. Parameters that might affect the density, shape and size of the tracks are also experimentally investigated.

3.1 Equipment design and construction

3.1.1 Polymer

It is important to note that polymer is often specific to a particular formulation and particles which form tracks in Table 2.4 in Chapter II shows examples of some commonly used solid state nuclear track detectors. A list of chemical etchants generally used along with the etching conditions for different detectors, particle sensitivity and the critical angle of etching are also given. Table 2.4 shows that polycarbonate plastics (Lexan, Makrofol, Mylar) are sensitive to alpha particles of energy $\leq$ 0.3 MeV while cellulose nitrate (Diacell, LR-115, CA-80-15) and allyldiglycol polycarbonate (CR-39) are sensitive to proton particle of energy $\leq$ 0.5 and 1.0 MeV, respectively.

Fleischer et al. mentioned that cellulose nitrate, cellulose acetate and UV-sensitized Lexan are sensitive to protons of energy up to $\sim$100 keV. The fraction of atoms that are hydrogen is 32 % in cellulose nitrate and 45 % in Lexan. In a neutron energy interval from perhaps 20 to 100 keV, tracks of recoiled protons produced in elastic collisions with hydrogen in the plastic would be detectable [3].

In this study, many types of polymer such as cellulose nitrate, CR-39, CR-39 optical lens and sunshade polycarbonate plastic were first tested by irradiating with neutrons from a Cf-252 source submerged in water to produce recoil proton tracks.The

Cf-252 source emitted neutrons at the rate of 4.54 x 10^7 n/s on 28^{th} of June 2002 as showed in part I of appendix A. In June 2010, when the irradiation was conducted, the neutron emission rate was about 5 x 10^6 n/s. The result showed that after etching proton tracks could be seen in CR-39, CR-39 optical lens and sunshade polycarbonate plastic. Then a locally available polycarbonate plastic for sunshade, 1.5 mm thick was used as a commercial PC because it is inexpensive and easily available.

Polycarbonate (PC) plastic, known by the trademarked names Lexan, Makrolon, Makroclear and others, are a particular group of thermoplastic polymers. The main polycarbonate material is produced by the reaction of bisphenol A and Phosgene ($COCl_2$). The chemical structure is showed as Figure 3.1 and the physical and thermal properties are showed in Table 3.1 [25].

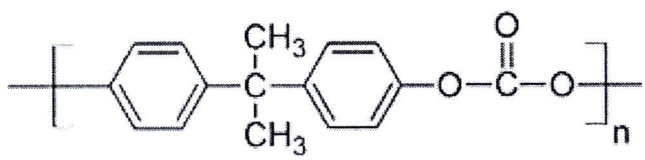

Figure 3,1 The structure of Polycarbonate polymer. [25]

Table 3.1 Physical and thermal properties of Polycarbonate

Property	
Density (ρ)	1.20-1.22 g/cm^3
Refractive index (n)	1.584-1.586
Melting temperature (Tm)	267°C

3.1.2 Neutron Irradiation

The scope of this study mentioned that track formation process on films used neutrons from radioisotope source via $^1H(n, n)^1H$ and $^1H(n, n')^1H$ reactions. The study was designed to use neutron radioisotope source because it was easy to operate when compare to reactor and the maintenance cost is almost zero. The possible choices for radioisotope neutron source are much more limited and are based on either spontaneous fission or on nuclear reactions. The most common spontaneous fission source is ^{252}Cf. Its half life is 2.65 years is long enough to be reasonably convenient, and the isotope is one of the most widely produced of all the transuranics. The dominant decay mechanism is alpha decay, and the alpha emission rate is about 32 times that for spontaneous fission. The neutron yield is 0.116 n/s per Bq, where the activity is the combined alpha and spontaneous fission decay rate. On a unit mass basis, 2.30 x 10^6 n/s are produced per microgram of ^{252}Cf. The average neutron energy is about 2.1 MeV, and the predominant energy is about 0.7 MeV. The energy spectrum of the neutron is showed as Figure 3.2 [Appendix A].

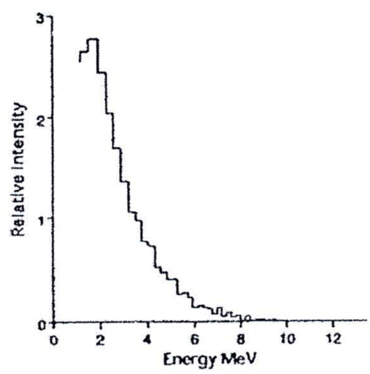

Figure 3.2 Neutron energy spectrums from the spontaneous fission of Cf-252.

Nuclear reaction radioisotope source generates neutron via (α, n) reaction. Because energetic alpha particles are available from the direct decay of a number of convenient radionuclides, it is possible to fabricate a small self-contained neutron source by mixing an alpha-emitting isotope with a suitable target material. Several different target materials can lead to (α, n) reactions for the alpha particle energies that are readily available in radioactive decay. The maximum neutron yield is obtained when beryllium is chosen as the target, and neutrons are produced through the reaction:

$$_2\alpha^4 + {}_4Be^9 \rightarrow {}_6C^{12} + {}_0n^1$$

which has a Q-value of + 5.71 MeV.

Some of radioisotope (α, n) sources are ^{238}Pu-Be, ^{239}Pu-Be, ^{241}Am-Be, ^{244}Cm-Be, etc. The ^{239}Pu-Be source was the most widely used several years ago. However, because about 16 g of the material is required for 1 Ci (3.7×10^{10}Bq) of activity, sources of this type of a few centimeters in dimension are limited to about 10^7 n/s [6]. Nowadays, ^{241}Am-Be is most widely used. Half-life of ^{241}Am is 433 years. The energetic neutrons are generated following an interaction between the alpha particle and the target material's nucleus and possess energies up to 10 MeV, with an average value of ~4 MeV.

In this study, californium-252 (Cf-252) radioisotope source was used to be neutron source due to its high neutron emission rate and its average neutron energy was less than ^{241}Am-Be radioisotope source. Fleischer et al. mentioned that a neutron energy interval from perhaps 20 to 100 keV, tracks of recoiled protons produced in elastic collisions with hydrogen in the plastic would be detectable [3].

Cf-252 neutron source used in this research was originally 20 μg or 10.7 mCi on 28th of June 2002. The neutron emission rate was 4.54×10^7 n/s [26]. Each of spontaneous fission emitted 3.76 neutrons. The calculated neutron emission on June 2010 was approximately 5×10^6 n/s which gave the maximum neutron flux in water about 5×10^4 n/cm^2.s. The specification of Cf-252 is showed in Appendix A. Figure 3.3 illustrates a drawing of Cf-252 source.

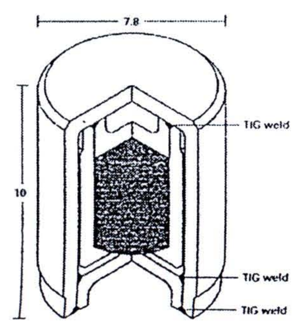

Figure 3.3 Illustration of Cf-252 neutron source.

In this study, the Cf-252 neutron source was placed at the end of L shape aluminum tube and sealed the end with aluminum plate. The diameter of tube was 0.8 cm, 0.2 cm thickness and 40 cm length. Inside of aluminum tube inserted with acrylic tube in order to fix Cf-252 source. (Shown as Figure 3.4)

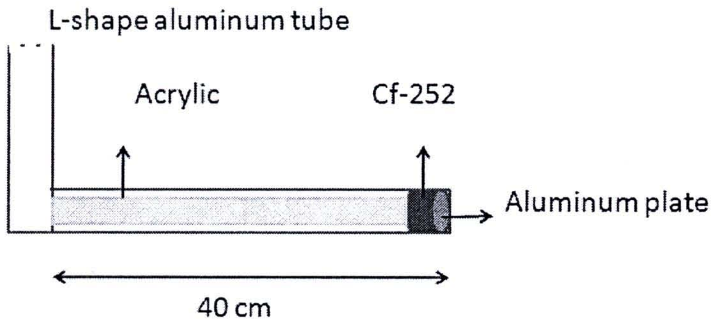

Figure 3.4 the designed envelop of Cf-252 source.

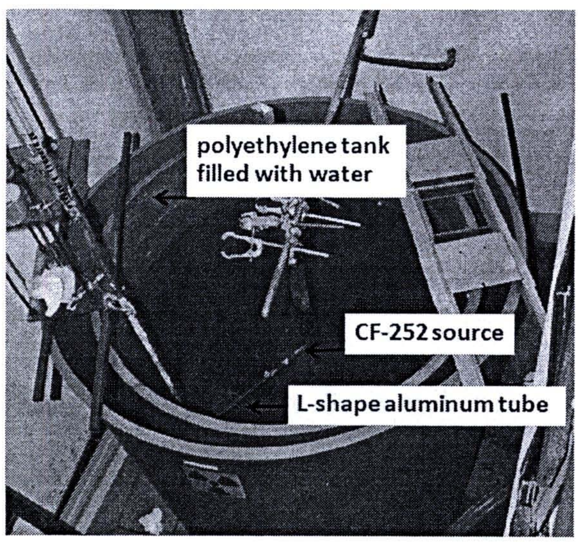

Figure 3.5 Showing Cf-252 source contained in an L shape aluminum tube submerged in a polyethylene tank filled with water.

The aluminum tube with Cf-252 source was placed under water in a polyethylene tank which was filled 1,000 liter of water. The diameter of inner tank was 105 cm and height was 105 cm (Figure 3.5). PC chip was put in an aluminum box and placed the box close to Cf-252 source in order to fix the distance of PC from the source. The proton track density in PC was varied by changing the irradiation time.

3.1.3 Etching conditions

After neutron irradiation, PC was etched to enlarge the latent tracks. From the literature review, Table 2.4 in Chapter II showed that the general etching condition of PC for alpha track is 6 N NaOH at 60°C, 60 min. Some researchers [25, 26, and 27] used PEW solution as the etchant for alpha track on PC. The PEW solution composed of 15% KOH, 40% ethanol and 45% H_2O.

The result from previous study [in part I, Appendix B] showed that the proper etchant for proton track on PC in this study was PEW solution. The etching time of PEW solution at 70°C was shorter than NaOH 6.25N at 60°C. The etching time for PEW and NaOH were 60 and 100 min, respectively.

The properties of track depend on the condition of etchant. In this study, the etching time and temperature were varied to create the different track sizes and shapes. Two etching conditions were studied:

3.1.3.1 The temperature of PEW solution was varied from 65, 70 and 75°C. The temperature of etchant was controlled with water bath (Figure 3.6)

Figure 3.6 Water bath with thermometer.

3.1.3.2 The etching time of PC was increased 15 minutes in each step from 15 to 90 minutes at the temperature 65, 70 and 75 ^{o}C in order to investigate track shape and size.

3.1.4 Observation and measurement of proton tracks

Proton track on PC can be observed under an optical microscope equipped with Motic Image Plus version 2.0 digital microscopy software (User's Manual is showed in Appendix C). Figure 3.7 demonstrates the optical microscope equipped with Motic Image software. This software was used to capture the track image and saved in jpeg format. The magnification of optical microscope was approximately x100. Then the captured track image was used to characterize properties of the tracks by using the ImageJ software which was an image processing and analysis program (User's Manual showed in Appendix D). In this study, the parameter settings of the software in analysis of the tracks were showed in Figure 3.8. The range of particle size (μm^2) was 1 to infinity and circularity of track was 0.5 to 1.0.

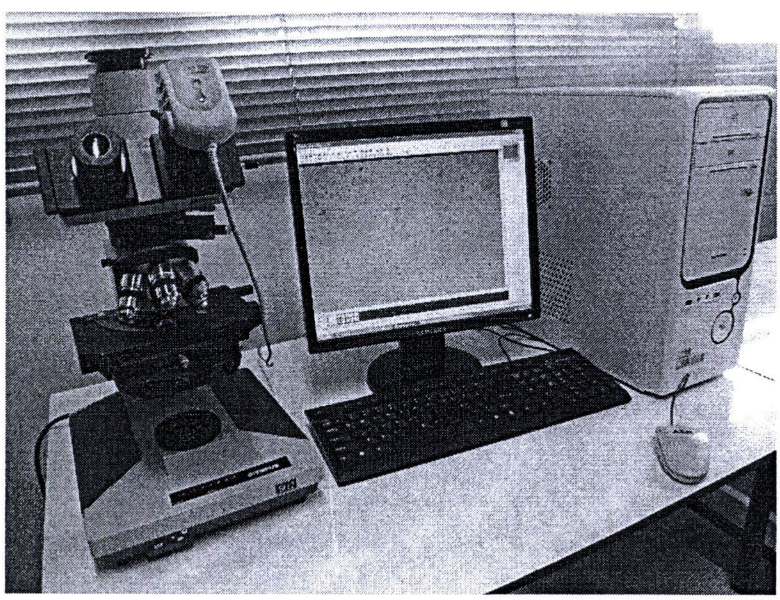

Figure 3.7 Microscope with Motic image capture software.

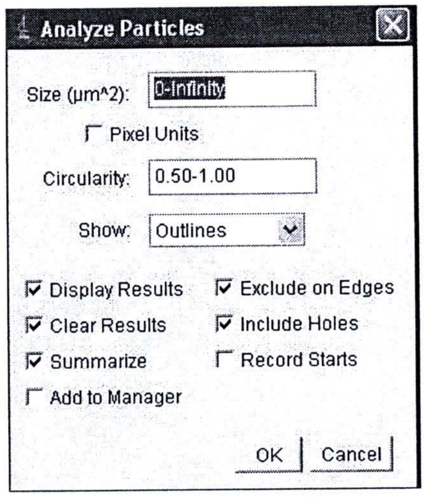

Figure 3.8 Parameter settings in analysis of proton tracks as seen on the screen of the ImageJ software.

3.1.5 Light transmission measurement

Investigation of properties of track-etch polymer films on transmission of visible light, ultraviolet (UV) and infrared (IR) was conducted by using a SPECTRUM DETECTIVE Transmission Meter model SD2400. The transmission meter displays energy transmission values in three regions. The light sources used for the three regions have a peak response at the following wavelengths: UV 365 nm, visible light with full weighted spectrum and infrared 950 nm (Figure 3.9). The user's manual is showed in Appendix E.

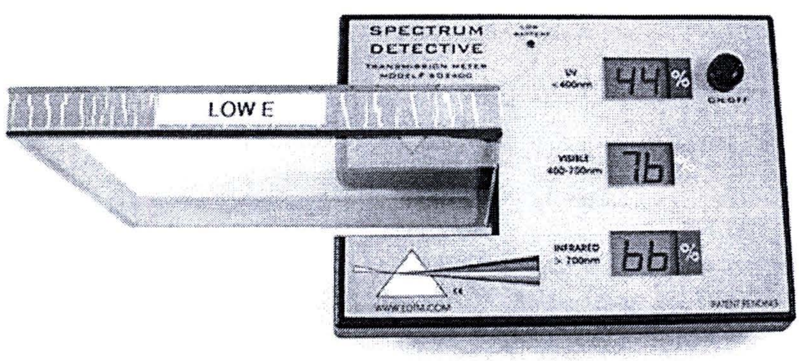

Figure 3.9 SPECTRUM DETECTIVE Transmission Meter SD2400.