



Chapter 3

Apparatus And Experimental Method

The photodegradation of injection grade high density polyethylene with and without anthraquinone and benzophenone photosensitizers was studied. Natural weathering and accelerated weathering tests were carried out in two sections by using four tubular low-pressure mercury vapour fluorescent lamps and medium pressure mercury lamp. Testing methods used for the natural weathering, accelerated weathering and evaluation of characters were the same.

3.1 Reagent and Materials

3.1.1 Injection grade high density polyethylene supplied by Thai Petrochemical Industry Co., Ltd. The mechanical properties of the original samples before exposure are shown in table 3.1

3.1.2 Photosensitizers

3.1.2.1 Anthraquinone (yellow needles, melting point 286°C) having an ultraviolet absorption maximum between 280 and 330 millimicrons.

3.1.2.2 Benzophenone (white prisms, melting point 47.5°C) having an ultraviolet absorption maximum between 330 and 400 millimicrons.

3.1.3 Solvent - Decahydronaphthalene ; >98 %

3.1.4 Heat transfer medium - paraffin Liquid

Table 3.1 Original properties of test sample

property	test method	unit	value
melt index[2.16 kg/190°C]	ASTM D1238	g/10 min	15.00
melt index[5 kg/190°C]	ASTM D1238	g/10 min	45.00
density	ASTM D1505	g/cm ³	0.957±0.001
tensile strength	ASTM D 638	N/mm ²	>13
yield strength	ASTM D 638	N/mm ²	>30
ultimate elongation	ASTM D 638	%	>100
ball indentation hardness	DIN S3456	N/mm ²	>54
notched impact strength	DIN S3453	MJ/mm ²	>1.0
vicat softening temperature	ASTM D1525	°C	-
haze	ASTM D1003	% max	-
gloss	ASTM D2453	% min	-
impact strength	ASTM D17038	g	-

3.2 Apparatus

3.2.1 Extruder

3.2.2 Cutting Machine

3.2.3 Hot Pressing

3.2.4 Punching Machine

3.2.5 Exposed equipment made from steel and porcelain. The dimension of exposed equipment are 1.2 x 2.4 meters about 1 meter above ground.

3.2.6 Weathering cabinet

3.2.7 Fume cupboard

3.2.8 Lamp

3.2.8.1 Medium-pressure mercury vapour lamp

(Philips 125 W) emits a high degree of ultraviolet radiation.

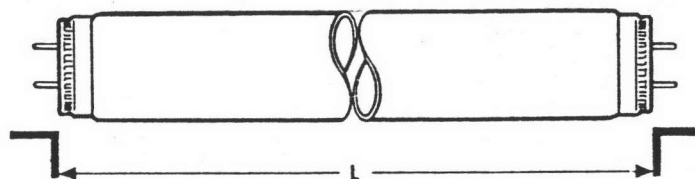


3.2.8.2 Tubular low-pressure mercury vapour fluorescent lamps (Philips 40 W) emits UV radiation between 300 and 460 nm with a maximum at 365 nm.

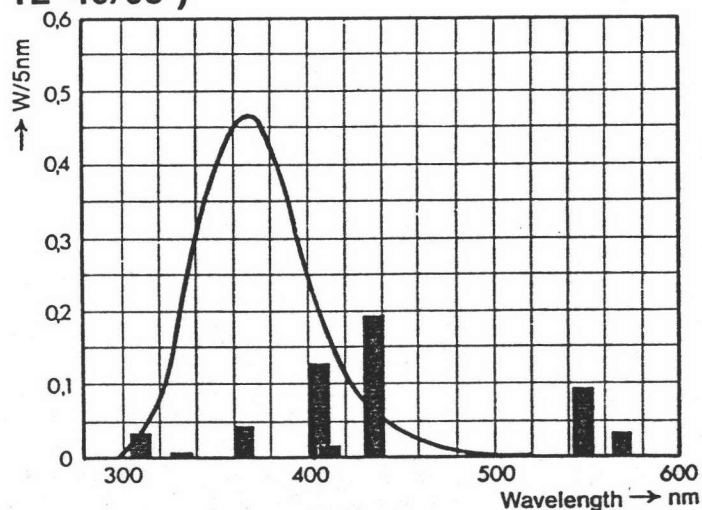
Table 3.2 Electrical data

Type	Nom. length cm	Nom. diameter mm	Lamp watts w	Lamp voltage v	Lamp current A	Useful life h
'TL' 40W/05	122	38	40	103	0.43	3000

Dimensions



Absolute spectral power distribution for 'TL' 40/05*)



3.2.9 Tensile Tester : Shimadzu AUTOGRAPH model S-100-C ;
test speed 200 mm/min , load select 20 kg.

3.2.10 Fourier Transform Infrared Spectroscopy :
Perkin-Elmer model 1720.

3.2.11 Viscosity analysis apparatus

- Ubbelohde viscometer
- Beaker 5 liters
- Transfer pipet 5 ml
- Volumetric flask 50 ml , 25 ml
- Thermometer 0 - 200 °c
- Hot plate stirrer
- Timer
- Vacuum oven
- Cylinder
- Erlenmayer flask 125 ml

3.2.12 Vernier

3.3 Sample preparation

The power photosensitizers , anthraquinone and benzophenone in a ratio of 1:1 , were mixed with HDPE in a hopper and were then homogenized by an extruder. The sheet , thickness about 0.4 mm , containing 2 photosensitizers in a concentration of 0 % , 1.0 % , 1.5 % and 2.0 % were compression-molded using stainless steel glazing plates. The plates were maintained at 453 K under a pressure of 158 kg/cm² for 15 minutes and then cooled to about 300 K by running cold water while maintaining the same pressure , then the square sheets (18 x 18 cm) were further machined into flat dumbbell shapes (International Standard ISO 1184).

3.4 Sample irradiation

The location of the exposure sites was downtown Bangkok (latitude 13 44 N longitude 100 34 E). The meteorological data for the exposure period are shown in table 3.4. All data were obtained from "The Monthly Report of the Climatology Division , Meteorological Department.

Table 3.3 Meteorological data

site	Avgtemp, °C	Annual humit, %R.H.	Annual rainfall, mm	Annual solar rad 10 ² .MJ/M ²
Bangkok	28.7	73	1496.4	1938.92

- value for January 1989 to December 1989.

3.4.1 Natural weathering

The dumbbells were set on the sample holder with wire. The tests were started in October 1989 to April 1990 for 6 months by the arrangement of the samples on the exposed equipment as shown in figure 3.3.

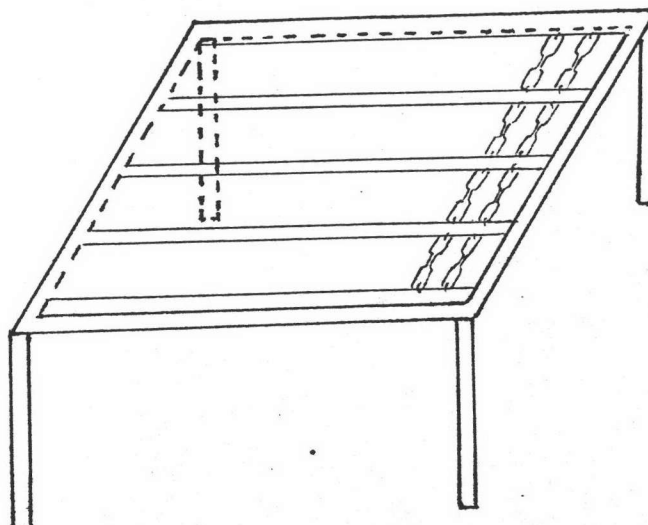


Figure 3.3 Exposed equipment

3.4.2 Artificial weathering

3.4.2.1 The artificial test by 4 fluorescent lamps (160 W). The irradiation samples were of the same shape and the same arrangement of samples used for the outdoor exposure test were used. The distance between samples and the light source was about 10 centimeters. The temperature in a cabinet was about 305 K. The irradiation period was 33 days.

3.4.2.2 The artificial test by medium pressure mercury vapour lamp (125 W). The same shape of samples used for the outdoor exposure test were used and were set on the sample holders (four dumbbells for each holder) around the light source as shown in figure 3.5. The irradiation period was 240 hours.

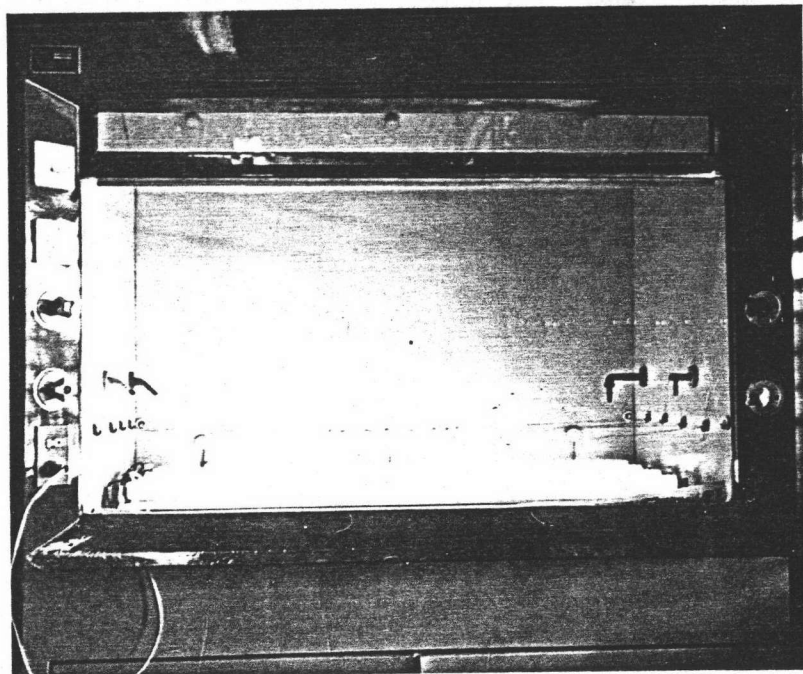


Figure 3.4 Accelerated cupboard with four fluorescent lamps.

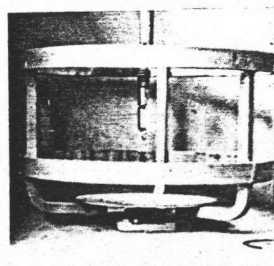
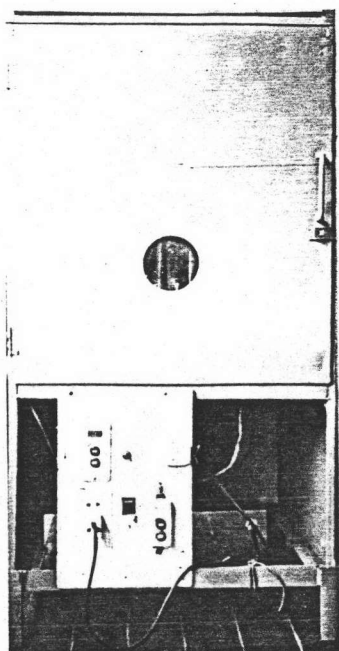


Figure 3.5 Accelerated cabinet with medium pressure mercury lamp

3.5 Mechanical measurements

The samples for the mechanical tests were strips of 25 mm width with a gauge marks of 25 mm. Tensile strength measurements were made using an instron tensile tester with a test speed of 200 mm/min in air at room temperature according to the ISO 1184. The test were repeated under identical conditions five times for all samples. The resulting tensile strengths were then determined as an average.

3.6 FTIR measurements

FTIR absorption spectra were recorded using a FTIR spectrophotometer and were used for estimating the changes in carbonyl absorbance peak at about 1715 , 3370 , 1677 , 909 cm^{-1} during irradiation. The results , for example carbonyl absorbance peak , were expressed by a carbonyl index I_{co} defined as

$$I_{\text{co}} = A_{1715} / A_{2019}$$

where

A_{1715} = absorbance of carbonyl group

A_{2019} = internal standard band in PE chain

3.7 Average molecular weight measurements

Average molecular weights of all samples were determined in the viscosity method, using decahydronaphthalene solutions and calculated from the Mark-Houwink-Sakurada equation.

$$[\eta] = kM^a$$

where

- $[\eta]$ = viscosity number of a solution
- k = constant value = 62×10^{-3}
- a = constant value = 0.7
- M = molecular weight

3.7.1 Typical procedure for viscosity determination

Approximately 15 ml of decahydronaphthalene was transferred by syringe into an Ubbelohde viscosity which was permanently positioned in an oil bath which was kept constant at 135 °c until the solution attained thermal equilibrium (about 5 minutes). The liquid level was brought to approximately 10 mm above the upper graduation mark on the viscometer capillary. As the meniscus passed this point, the timer was started and the interval for the solution to drain to the lower mark on the capillary was timed. (See figure 3.6) The effect time of the solution was measured at least three times. Three consecutive readings should agree within 0.2 second. The solution was then removed from viscometer.

One PE sample of 0.4 + 0.0002 grams was weighted and transferred quantitatively to a 100 ml volumetric flask. Approximately 50 ml of solution was added to the sample flasks and placed in an oil bath maintained at 140 °c. The flasks were shaken once every 10 min. until the solution was completed and then the solution was made up to the 100 ml mark with the solvent.

maintained at 135°C by means of a syringe and the flasks were shaken once again. In the same manner as decahydronaphthalene, three consecutive effect times of the first PE solution in the viscometer were recorded. The first solution was then diluted to the concentration of 0.24 , 0.16 , 0.08 g/dl^{-1} by adding 15 , 10 and 5 ml of the first solution into the three 25 ml volumetric flasks, respectively and then all solutions were made up to the 25 ml mark with the solvent maintained at 135°C . The effect time of each times and the average effect time of undoped HDPE sample and HDPE samples doped with 2-photosensitizers in a concentration of 1.0% , 1.5% and 2.0% by weight before and after exposure to the light source were obtained.

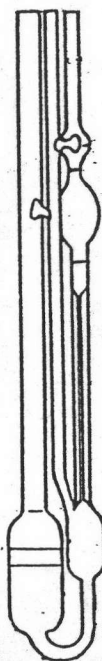


Figure 3.6 Ubbelohde viscometer