# CHAPTER 3

# EXPERIMENTAL PROCEDURES

## 3.1 Raw Materials

The polymer used in this study are three commercial polyolefin films supplied by Thai Polyethylene Co., Ltd. (TPE) as follow

- Low Density Polyethylene ( LDPE ) grade no. LD 1905 F
- High Density Polyethylene ( HDPE) grade no. H 5604 F
- Polypropylene ( PP ) grade no. P 600 F

The characteristics of raw materials are shown in Table 3.1

Table 3.1 Physical properties of polyolefin films.

| Type of polyolefins / Properties | LDPE | HDPE | PP |
|---|---|---|---|
| - Melt Flow Index (g/10 min) | 5.00 | 0.04 | 10.00 |
| -Density ($g/cm^3$) | 0.919 | 0.956 | 0.901 |
| -Thickness (mm) | 0.070 | 0.020 | 0.040 |

### 3.2 Exposure Conditions

Exposure Condition for the testing method of photooxidative is based on the results of Thailand weathering climate (at Bangkok Metropolis). Table 3.4 and Figure 3.1-3.5 show the weathering climate at Bangkok Metropolis, recorded by Meteorological Department.

Table 3.2 Data of Thailand weathering climate.

| Year | Radiation $MJ.m^{-2}$ | UV $MJ.m^{-2}$ | Temp °C | RH % | Rain (mm) | Wind (knots) |
|---|---|---|---|---|---|---|
| 1986 | 6186.39 | 309.32 | 27.8 | 73 | 1807.5 | 4.2 |
| 1987 | 5840.07 | 292.00 | 28.4 | 73 | 1370.3 | 4.0 |
| 1988 | 6523.64 | 326.18 | 28.1 | 74 | 2097.3 | 3.9 |
| 1989 | 7049.40 | 352.47 | 28.2 | 73 | 1496.4 | 4.4 |
| 1990 | 6948.53 | 347.43 | 28.5 | 72 | 1362.9 | 3.1 |
| 1991 | 6697.98 | 334.90 | - | 75 | - | 3.4 |
| 1992 | - | - | 28.3 | 73 | 1442.9 | 2.6 |
| 1993 | 6786.74 | 339.34 | 28.1 | 73 | 1543.6 | 2.5 |
| 1994 | 6460.33 | 323.02 | 28.3 | 74 | 1596.4 | 2.3 |
| 1995 | - | - | 28.2 | 66 | - | 2.9 |
| Average | 6561.64 | 328.08 | 28.21 | 72.6 | 1589.66 | 3.33 |

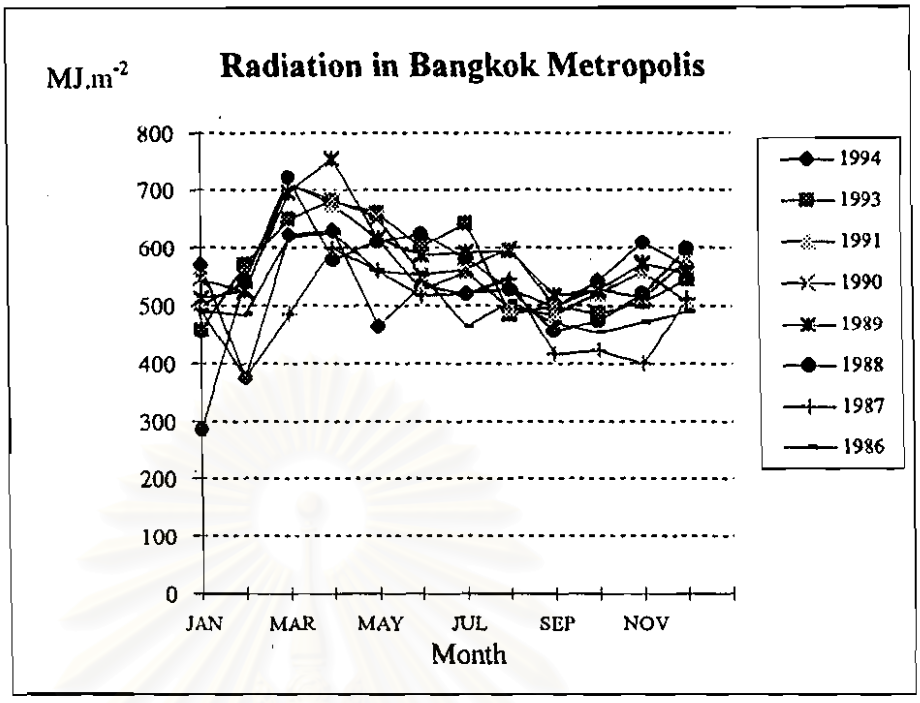


Figure 3.1 Average radiation exposure at Bangkok Metropolis during 1986-1994

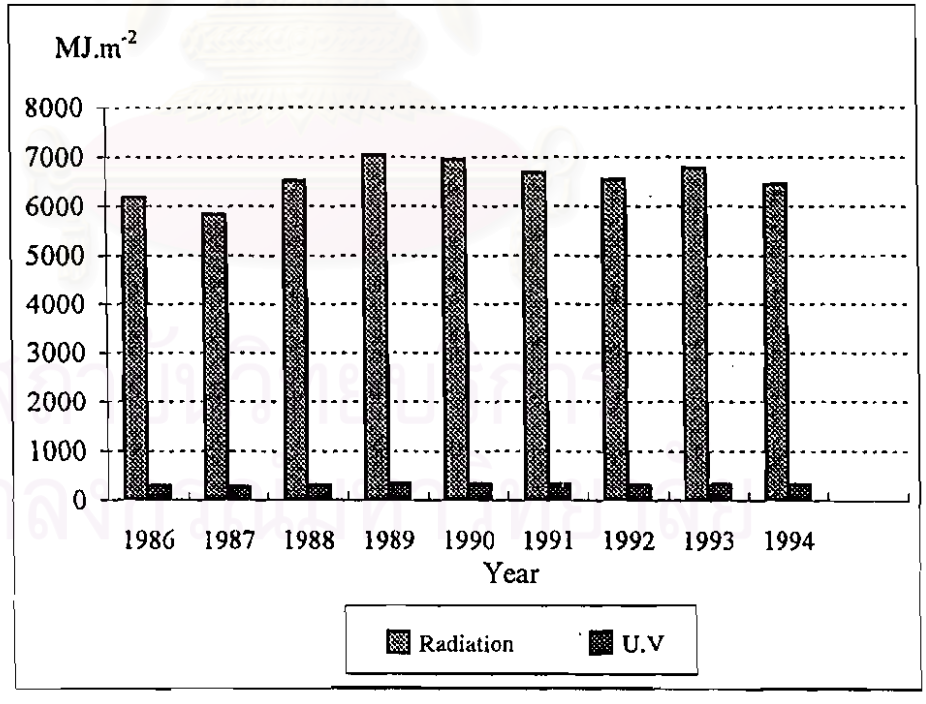


Figure 3.2 The average of UV and total radiation exposure at Bangkok Metropolis during 1986-1994

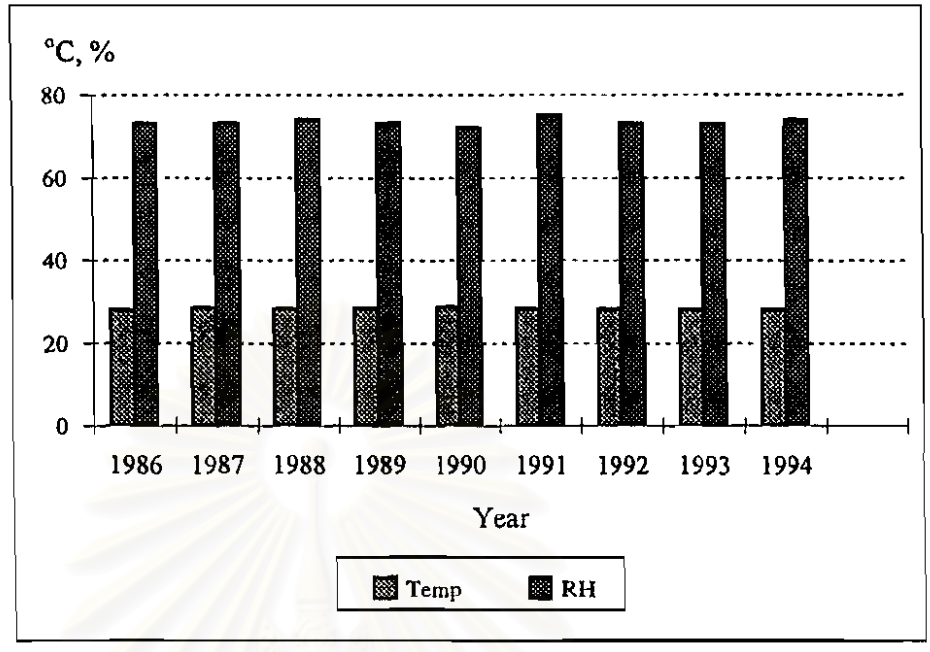


Figure 3.3 The average temperature and relative humidity at Bangkok Metropolis during 1986-1994

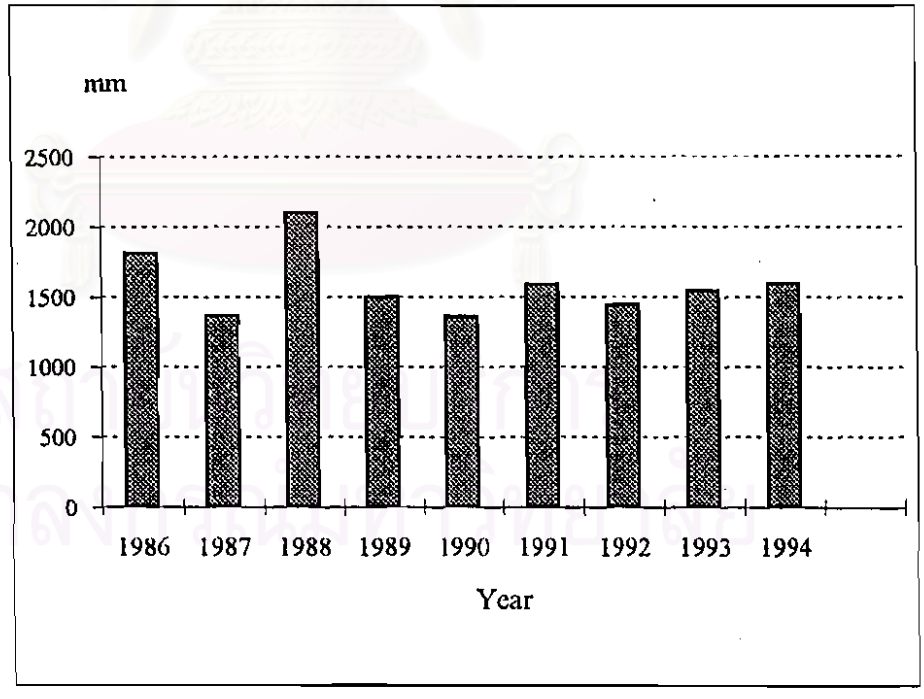


Figure 3.4 Average rainfall at Bangkok Metropolis during 1986-1994

The information will be used to examine outdoor exposure and Xenotest machine.

### 3.2.1 Outdoor Exposure

Outdoor exposure, carried out under a tropical climate at Bangkok Metropolis (Thailand) , was started in August, 1995 . The samples were prepared in rectangular shape, cut to size 25cm x 30 cm, and then fixed on the exposure racks with 45 degree angle to the horizontal. The racks were designed in accordance with ASTM D 1435-85 as shown in Figure 3.5. It was placed on the top of the building at Chulalongkron University. This building height is about 40 m. above ground level. The samples were exposed to sunlight until the films are entirely brittle. During the experiment, in every week the films were collected for chemical characterization and mechanical property testing.



Figure 3.5 The exposure racks for outdoor exposure study.

### 3.2.2 Accelerated UV exposure ( in Xenotest)

Accelerated UV exposure was carried out by using a xenon arc lamp in Xenotest Beta Lamp machine which shown in Figure 3.6. This practice may be used for evaluating the behavior of plastics exposed in a machine that produces simulated sunlight irradiance and controlled temperature and relative humidity.

The Xenotest operation program is based on standard method ISO 4892/2 under the following parameters:

| | | |
|---|---|---|
| Filter system | : | Xenochrome 320* |
| Program number | : | 2 |
| Number of phase | : | 1 |
| Irradiance (E) | : | 100 $W/m^2$ |
| Temperature | : | 28 °C (chamber temperature) |
| Relative Humidity | : | 73% |
| Phase time | : | 60 min. |
| Switch off criterion | : | Total time |
| Total time | : | 76 hrs.** |

* wavelength ($\lambda$)

** Total time was calculated as follow:

1 year Thailand sunlight (UV300-400nm) = 328 $MJ/m^2$

= $328 \times 10^5$ Watts $sec/m^2$

Irradiance Xenotest Beta UV = 100 $Watts/m^2$

Therefore, 1 year of outdoor equivalent to = $\frac{328 \times 10^5 \text{ Watts sec/m}^2}{100 \text{ Watts/m}^2}$

= $3.28 \times 10^6$ sec. in Xenotest

= 911 hrs. in Xenotest

≈ 38 days in Xenotest

(1 MJ = $10^6$ Watt sec.)

The specimen size was (5 cm x 9 cm). Irradiation source, used in the Xenotest apparatus, was xenon arc lamp. It shall be capable of closely simulating the spectral power distribution of sunlight (Global Solar Irradiance).

Figure 3.6 The Xenotest Beta Lamp machine for accelerated UV exposure.

## 3.4 Analytical Measurements

### 3.4.1 Infrared absorption

After different exposures, films were removed for the analysis to estimate the effects of outdoor exposure and accelerated UV exposure. Fourier Transform Infrared Spectroscopy model Impact 400D (FT-IR) was equipped with air dry-system with a frequency range from 4000 to 400 $cm^{-1}$ The resolution was chosen to be 2 $cm^{-1}$ for 36 scans. It was used to monitor the damage caused by UV irradiation on the chemical structural changes in the films. Figure 3.7 showed the FT-IR spectrometer. The variation in absorbance peak heights at selected bands, bands for carbonyl and aliphatic hydrocarbon were taken:

| Bands | Functional group | Wave number ($cm^{-1}$) |
|---|---|---|
| Carbonyl compounds | (-C-) with O | 1715±10 |
| Aliphatic hydrocarbon | ($-CH_2-$) | 1467±10 |

-A peak at 1467 $cm^{-1}$ is the characteristic of the polyolefins that was used as a reference peak or internal standard to minimize error due to radiation.

The equation for the carbonyl index (I) is

$$I = \frac{\text{Absorbance peak height at } 1715\ cm^{-1}}{\text{Absorbance peak height at } 1467\ cm^{-1}}$$

Figure 3.7 FTIR spectrometer model Impact 400D.

### 3.4.2 Tensile testing

Elongation at break and initial modulus were chosen to represent mechanical properties during the degradation of the films. The testing method carried out according to ASTM D 882 using Lloyd Universal Testing Machine (as shown in Figure 3.8). The parameters of tensile testing machine were:

| | |
|---|---|
| Load cell | 100 N |
| Cross head speed | 500 mm/min for PE |
| Cross head speed | 100 mm/min for PP |
| Sample strips | |
| width | 5 mm |
| length | 100 mm |
| Gauge length | 50 mm |
| Number of specimen | 5 |
| Conditions | |
| Temperature | 23 ± 2 °C |
| Relative Humidity | 50 ± 5% |

Elongation at break in percent was expressed by the following equation:

$$\% \text{ Elongation at break} = \frac{\Delta L}{L} \times 100$$

when;

$\Delta L$ = The extension at the moment of rupture.

$L$ = Gauge length of specimen.

and initial modulus was obtained from the initial slope of stress-strain curve which can be expressed by the following equation:

$$\text{Initial modulus} = \frac{\sigma}{\varepsilon}$$

when,

$\sigma$ = Stress in linear portion of stress-strain curve = F/A

$\varepsilon$ = Strain in linear portion of stress-strain curve = $\Delta L/L$

F = Force required to stretch the specimen.

A = Original minimum cross-sectional area of specimen.

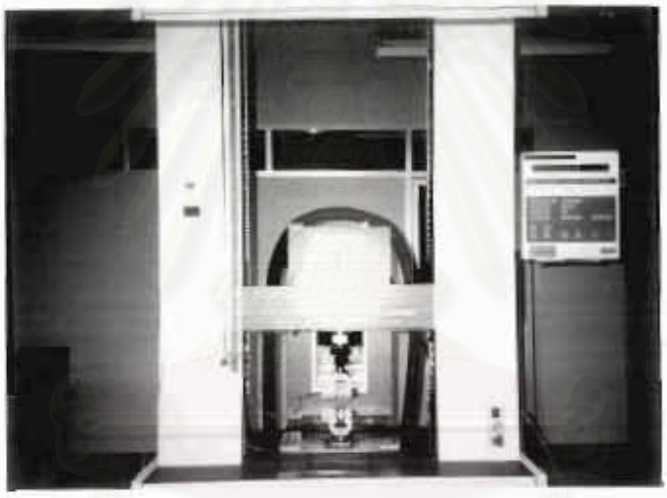

Figure 3.8 The LR 100K Lloyd Universal Testing machine.