CHAPTER III

EXPERIMENTAL

3.1 Home-made IR card from polytetrafluoroethylene (PTFE) film

3.1.1 Preparation of the home-made IR card

3.1.1.1 Materials and equipment

1. Polytetrafluoroethylene (PTFE) or Teflon tape
   The Teflon tape used in this research was purchased from local hardware stores (PTFE Thread Seal Tape, distributed by Pioneer Company). The width of Teflon tape is 12 mm.
2. Polyvinyl chloride (PVC) sheet with a dimension of 53 x 67 x 4.5 x 10^{-2} cm^3
3. Commercial PTFE IR card (Spectra-Tech)
4. Adhesive tape
5. Mirror with a dimension of 15 x 25 x 0.2 cm^3
6. Polarizer (Jasco, Japan Spectroscopic Co., Ltd.)
7. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.1.1.2 Methodology

On side of the PVC sheet was fastened with the double coated tissue paper tape and then cut the PVC sheet to be a circular ring with inner and outer diameter of 1 and 2 cm, respectively by the mould. The mould was invented to be about the double circular hole for cutting PVC sheet.

The Teflon tape was fastened to the miror. A 8 cm length of Teflon tape was secured with adhesive tape placed so that it covers a 3 mm strip along the top of the Teflon (see Figure 3.1A). A second piece of adhesive tape was then attached along the bottom of the Teflon leaving a strip about *x* mm wide uncovered. The Teflon tape was then stretched by pulling it down with heating from a drier, as shown in Figure 3.1B. The botom piece of adhesive tape was pressed down onto the mirror to hold the Teflon in place. Subsequently, two pieces of adhesive tapes were attacthed to

remained sides of Teflon tape, and then the both sides were pulled to be about 8 cm. Place a circular PVC ring onto the stretched PTFE film and cut it from a mirror. Finally, the home-made IR card with different thicknesses were achieved for sample analysis. Stretching ratio (S) was defined as the width of uncovered Teflon tape after stretching (y), divide by the width of uncovered tape before stretching (x) in the follow equation: $S = y/x$

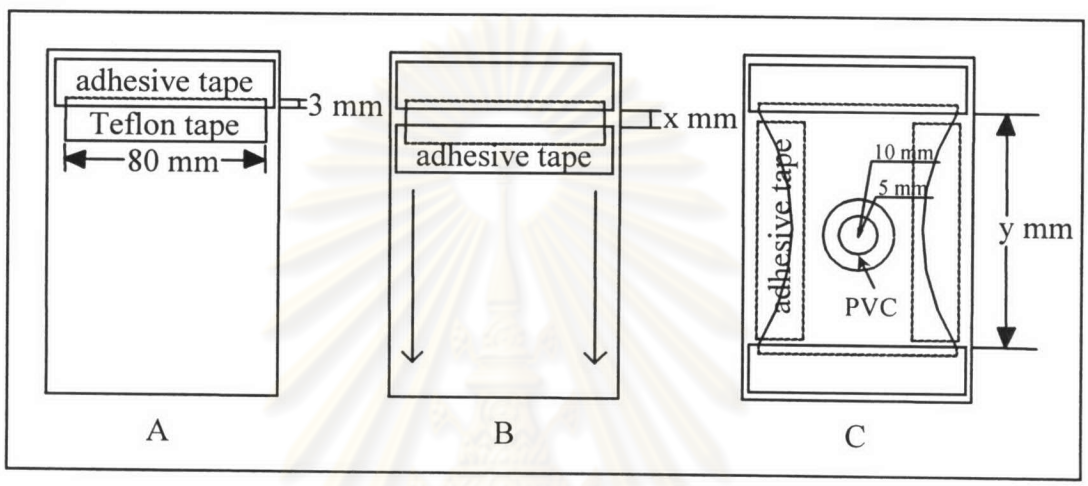

Figure 3.1 Preparation of the Teflon tape as IR card. Marks indicating the position of the Teflon tape can be etched permanently on the backing to obtain consistent stretching ratios.

3.1.2 Spectral acquisition of different in PTFE film thicknesses

1. An open-beam through the aperture of the circular PVC ring was recorded in order to use as a background.

2. Place the commercial PTFE IR card in the sample beam and then spectrum was collected.

3. Repeat the above two steps by using the unstretched Teflon tape and the home-made IR cards at different stretching ratios.

4. At the end of the experiment, plot the relationship between absorption intensity at 1228 cm^{-1} (*i.e.*, C-F stretching vibration) and stretching ratios (S).

3.1.3 Spectral subtraction of absorption bands of PTFE

1. Place the home-made IR card from thin PTFE film in the sample beam and then record the spectrum of PTFE.
2. Smear moisturizing cream (Garnier, distributed by Loreal Co., Ltd.) onto the surface of thin PTFE film.
3. Place the home-made IR card with sample in the sample beam and collect the spectrum.
4. Subtract the spectrum of PTFE out of the mixture spectra of PTFE and moisturizing cream.

3.1.4 Spectral acquisition of sensitivity determination of the thin PTFE film

1. Place the home-made IR card from thin PTFE film in the sample beam and then record the spectrum to subtract the absorption bands of PTFE after sampling sample.
2. Attach this home-made IR card onto the face of human and then carefully pulled it.
3. Place the home-made IR card with sample in the sample beam and scan the sample.

3.1.5 Spectral acquisition of orientation of PTFE chain

1. Put the polarizer into the sample holder of FT-IR spectrometer.
2. Rotate the polarizer at 90° to obtain the perpendicular polarized light and then record the spectrum to be a background.
3. Place the commercial IR card in front of the polarizer and collect the spectrum.
4. Rotate the polarizer at 0° to obtain the parallel polarized light and then record the spectrum to be a background.
5. Place the commercial IR card in front of the polarizer and collect the spectrum.
6. Repeat steps 2 to 5 by using the home-made IR cards with different stretching ratios of 4.0, 12.8 and 53 instead of the commercial IR card and observed

the different of the absorption intensity between perpendicular and parallel polarized light to compare the occurring orientation of polymer chain.

3.2 Acquisition parameters for FT-IR experiment

Nicolet Magna 750 Series II FT-IR spectrometer

Experimental setup	
Resolution	4.0 cm^{-1}
Number of scans	16
Result spectrum	Absorbance
Spectrometer parameters	
Aperture	open
Gain Amp	1
Source	Globar
Detector	DTGS
Beam splitter	KBr

3.3 Efficiency study of the home-made IR card for various types of samples

3.3.1 Preparation of solids

3.3.1.1 Preparation of solid samples by grinding

3.3.1.1.1 Materials and equipment

1. Solid samples
   - Clay; from Thailand Institute of Scientific and Technological Research
   - Zeolite Y; from Thai Silicate Chemical Company
   - Chromium benzoate; from Natural Product Research Unit Chulalongkorn University
   - *p*-Nitrobenzoic acid; analytical grade from Merck
2. Potassium bromide (KBr); analytical grade from Merck
3. Agate mortar and pestle
4. Die set presses with 10 mm diameter pellet
5. Home-made IR card

6. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.1.1.2 Methodology and spectral acquisition

Home-made IR card; steps in preparating a home-made IR card:

1. Put ~100 mg of sample in an agate mortar and grind it to fine powder for 3-5 minutes.
2. Transfer a small amount of finely powdered sample with pestle onto the surface of home-made IR card.
3. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and then record the spectrum. Before transfer sample to the home-made IR card, record the spectrum of home-made IR card as a reference spectrum for subtraction process.

KBr pellet; steps in preparating a KBr pellet:

1. Put ~30 mg of sample and ~300 mg of KBr in a mortar. Grind well with a pestle (~1 minute).
2. Assemble the die set press and add ~100 mg of the mixture.
3. Press the mixture into a pellet.
4. Remove a pellet from a die, place in the pellet holder, place a pellet holder in the FT-IR spectrometer and record the spectrum.

3.3.1.2 Preparation of solid by dissolving with organic solvent

3.3.1.2.1 Materials and equipment

1. *p*-Nitrobenzoic acid; analytical grade from Merck
2. Methanol; analytical grade from Merck
3. Home-made IR card
4. Beaker 5 mL
5. Volumetric flask 5 mL
6. Dropper
7. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.1.2.2 Methodology and spectral acquisition

1. Dissolve 0.0845 g of *p*-nitrobenzoic acid with a small volume of methanol in a beaker.
2. Pour *p*-nitrobenzoic acid solution into a volumetric flask and make volume to be 5 mL with methanol.
3. Add a drop of *p*-nitrobenzoic acid solution onto the surface of home-made IR card. Allow the methanol to evaporate (~30s).
4. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before transfer sample to the home-made IR card, record the spectrum of home-made IR card as a reference spectrum for subtraction process.

3.3.1.3 Effect study of the high molecular weight paraffinic hydrocarbon liquids

3.3.1.3.1 Materials and equipment

1. Tablet samples
   - Chlorphenylamine hydrochloride; from Government Pharmaceutical Organization Thailand
   - Paracetamol; from Nam Kok Dispensary Co., Ltd.
2. Potassium bromide; analytical grade from Merck
3. Nujol; gas chromatography grade from Perkin-Elmer
4. Fluorolube; from Spectra-Tech Inc.
5. Agate mortar and pestle
6. Die set presses with 10 mm diameter pellet
7. Home-made IR card
8. Glass slide
9. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.1.3.2 Methodology and spectral acquisition

Home-made IR card; steps in preparating a home-made IR card:

1. Two glass slides were clean with acetone.

2. Add a drop of Fluorolube onto the surface of the first glass slide.

3. Place the second glass slide on the top of the first one and then slightly squeeze to make as possible a thin film of Fluorolube.

4. Move the second glass slide off the first one. The both glass slides were coated with thin Fluorolube film.

5. Attach the home-made IR card onto the coated glass slide and slightly pulled off and then place the coated home-made IR card into the sample compartment of FT-IR spectrometer and record the spectrum. The coated home-made IR card is kept in order to employ as a substrate for sample analysis.

6. Repeat above five steps by using Nujol instead of Fluorolube.

7. Put ~100 mg of sample in an agate mortar and grind it to fine powder for 3-5 minutes.

8. Transfer a small amount of finely powdered sample with pestle onto the surface of home-made IR card which was coated with Fluorolube.

9. Place the coated home-made IR card with sample into the sample compartment of FT-IR spectrometer and then record the spectrum. Before transfer sample to the coated home-made IR card, record the spectrum of coated home-made IR card as a reference spectrum for subtraction process.

KBr pellet; steps in preparating a KBr pellet:

1. Put ~30 mg of Chlorphenylamine hydrochloride and ~300 mg of KBr in a mortar. Grind well with a pestle (~1 minute).

2. Assemble the die set press and add ~100 mg of the mixture.

3. Press the mixture into a pellet.

4. Remove a pellet from a die, place in the pellet holder, place a pellet holder in the FT-IR spectrometer and record the spectrum.

5. Repeat the above four steps by using paracetamol instead of Chlorphenylamine hydrochloride.

3.3.2 Preparation of viscous liquids

3.3.2.1 Materials and equipment

1. Viscous liquid samples
   - Hair treatment (Oriental Princess, manufactured by K. M. Interlab Co., Ltd.)
   - Gel (Eucerin, manufactured by Beiersdort AG Hamburg)
   - Cream (La Roche, distributed by Loreal Co., Ltd.)
2. Zinc selenide (ZnSe) window
3. Home-made IR card
4. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.2.2 Methodology and spectral acquisition

Home-made IR card; steps in preparating a home-made IR card:

1. Smear the small amount of sample onto the surface of home-made IR card.
2. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before smear sample to the home-made IR card, record the spectrum of home-made IR card as a reference spectrum for subtraction process.

ZnSe window; steps in preparating a ZnSe window:

1. Place a ZnSe window into the sample compartment of FT-IR spectrometer and record the spectrum as a background.
2. Smear the small amount of sample onto the surface of ZnSe window.
3. Place a ZnSe window with sample into the sample compartment of FT-IR spectrometer and record the spectrum

3.3.3 Preparation of volatile liquids

3.3.3.1 Effect of sample volume

3.3.3.1.1 Materials and equipment

1. Toluene; analytical grade from Merck

In this section, toluene was used as a volume-test analyte in order to investigate the suitable volume of volatile liquid that analyzed by the IR card window.

2. Home-made IR card
3. Microsyringe
4. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.3.1.2 Methodology and spectral acquisition

1. Add 1 μL of toluene onto the middle surface of the first home-made IR card from thin PTFE film and then cover it with another IR card.
2. Place the home-made IR card window with sample into the sample compartment of FT-IR spectrometer and record the spectrum
3. Repeat the two steps by using different volumes of toluene at 2, 3, 4, 5, 6, 7 and 8 μL instead of 1 μL of toluene.

3.3.3.2 Time-dependent phenomena

3.3.3.2.1 Materials and equipment

1. Toluene; analytical grade from Merck

From investigation of suitable volume of toluene, 5 μL of toluene was the suitable volume for determination characteristic of toluene. The time-dependent effect on the capillary thin film was investigated by mean of changing in spectral feature. Therefore, in order to determine the efficiency of the home-made IR card, a 5 μL toluene was used for data collection automated by Macro-Nicolet program.

2. Home-made IR card
3. Microsyringe
4. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.3.2.2 Methodology and spectral acquisition

1. Add 5 μL of toluene onto the middle surface of the first home-made IR card from thin PTFE film and then cover it with another IR card.

2. Place the home-made IR card window with sample into the sample compartment of FT-IR spectrometer and record the spectrum for 1 minute. Take a series of spectra to observe the changes in absorption intensity as a function of time.

3.3.3.3 Effect of polarity of volatile liquids

3.3.3.3.1 Materials and equipment

1. Non-polar volatile liquids
- Hexane; analytical grade from Merck
- Cyclohexane; analytical grade from Carlo Erba
- Heptane; analytical grade from Merck
2. Low-polar volatile liquids
- Benzene; analytical grade from Merck
- Toluene; analytical grade from Merck
- *p*-Xylene; gas chromatography grade from Fluka
3. Moderate-polar volatile liquids
- Ethanol; analytical grade from Merck
- Isopropanol; analytical grade from Merck
- Acetone; analytical grade from Merck
4. High-polar volatile liquids
- Methanol; analytical grade from Merck
- Diethylene glycol; analytical grade from BDH Chemistry Ltd.
5. Home-made IR card
6. Microsyringe
7. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.3.3.2 Methodology and acquisition

1. Add 5 μL of hexane onto the middle surface of the first home-made IR card from thin PTFE film and then cover it with another IR card.

2. Place the home-made IR card window with sample into the sample compartment of FT-IR spectrometer and record the spectrum

3. Repeat the above two steps by varying the types of volatile liquids.

3.3.3.4 Application of the home-made IR card with liquid mixture

3.3.3.4.1 Materials and equipment

1. A fraction of ring-opening reaction at 2 hours of styrene oxide; from Natural Products Research Unit, Chulalongkorn University
2. Home-made IR card
3. Microsyringe
4. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.3.4.2 Methodology and spectral acquisition

1. Add 5 μL of fraction of ring-opening reaction of styrene oxide onto the middle surface of the first home-made IR card from thin PTFE film and then cover it with another IR card.

2. Place the home-made IR card window with sample into the sample compartment of FT-IR spectrometer and record the spectrum for 2 minutes. Take the series of spectra to investigate the spectral changes as a function of time.

3.3.4 Preparation of thin films

3.3.4.1 Determination of the thin film of lipstick on the glass

3.3.4.1.1 Materials and equipment

1. Lipstick (Cute Press, manufacture by K. M. Interlab Co., Ltd.)
2. Home-made IR card
3. ZnSe window
4. Glass
5. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.4.1.2 Methodology and spectral acquisition

Home-made IR card; steps in preparating a home-made IR card:

1. Smear the lipstick onto the surface of glass to be a thin film and carefully attach the home-made IR card to the coated surface of glass with a slightly press.
2. Carefully pull off the home-made IR card from the coated surface of glass.
3. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before attach the home-made IR card to the sample, record the spectrum of the home-made IR card as a reference spectrum for subtraction process.

ZnSe window; steps in preparating a ZnSe window:

1. Place a ZnSe window into the sample compartment of FT-IR spectrometer and record the spectrum as a background.
2. Smear the small amount of lipstick onto the surface of ZnSe window.
3. Place a ZnSe window with sample into the sample compartment of FT-IR spectrometer and record the spectrum. The obtained spectrum was compared with the spectrum from the home-made IR card.

3.3.4.2 The determination of the deposited molecules on a dried fish

3.3.4.2.1 Materials and equipment

1. Dried fish

The dried fish in this research purchased from the TaoPoon food market, Bangkok.

2. Home-made IR card
3. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.4.2.2. Methodology and spectral acquisition

1. Attach the home-made IR card onto the surface of dried fish with a slightly press.

2. Carefully pull off the home-made IR card from the surface of dried fish.

3. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before attaching the home-made IR card onto the sample, record the spectrum of the home-made IR card as a reference spectrum for further subtraction process.

3.3.4.3 The determination of dust particles deposited on the glass fiber paper

3.3.4.3.1 Materials and equipment

1. Glass fiber paper that was employed to collect dust particles

Glass fiber paper used in this section received from Safety and Environmental Control Technologies.

2. Home-made IR card
3. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.4.3.2. Methodology and spectral acquisition

1. Attach the home-made IR card onto the surface of the glass fiber paper with a slightly press.

2. Carefully pull off the home-made IR card from the surface of the glass fiber paper.

3. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before attach the home-made IR card onto the sample, record the spectrum of the home-made IR card as a reference spectrum for subtraction process.

3.3.4.4 The determination of released organic compounds in the plastic packaging

3.3.4.4.1. Materials and equipment

1. Packaging made from poly(ethylene terephthalate, (PET))
2. Home-made IR card
3. FT-IR spectrometer (Nicolet Magna 750 Series II)

4. Oven

3.3.4.4.2 Methodology and spectral acquisition

1. Heat the piece of PETE in the oven at 65 °C for 15 minutes.
2. Remove PETE piece and attach the home-made IR card onto the surface of PETE piece with a slightly press.
3. Carefully pull off the home-made IR card from the surface of PETE .
4. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before attach the home-made IR card onto the sample, record the spectrum of the home-made IR card as a reference spectrum for subtraction process.

3.3.5 Preparation of Gases

3.3.5.1 Materials and equipment

1. Clay BNH (Bentonite H are kindly donated by Ceramic "R" US)
2. Benzene; analytical grade from Merck
3. Agate mortar and pestle
4. Home-made IR card
5. Beaker 5 mL
6. Vacuum chamber
7. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.3.5.2 Methodology and spectral acquisition

1. Put ~100 mg of clay BNH in an agate mortar and grind it to fine powder for 3-5 minutes.
2. Transfer a small amount of finely powdered clay BNH with pestle onto the surface of home-made IR card.
3. Place the home-made IR card with clay BNH into the sample compartment of FT-IR spectrometer and record the spectrum as a reference spectrum.
4. Put the home-made IR card with clay BNH into the vacuum chamber for 2 days in order to allow clay to absorb vapor of benzene. The vacuum chamber was saturated with vapor of benzene.

5. Place the adsorbed home-made IR card into the sample compartment of FT-IR spectrometer and record the spectrum.

3.4 Application of the home-made IR card for various fields of research works

3.4.1 Determination of water in zeolite Y

3.4.1.1 Materials and equipment

1. Zeolite Y; from Thai Silicate Chemical Company
2. Home-made IR card
3. Agate mortar and pestle
4. Hot cell
5. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.4.1.2 Methodology and spectral acquisition

1. Put ~100 mg of zeolite Y in an agate mortar and grind it to fine powder for 3-5 minutes.
2. Transfer a small amount of finely powdered zeolite Y with pestle onto the surface of home-made IR card.
3. Place the home-made IR card with zeolite Y into the hot cell and record the spectrum as a reference spectrum.
4. Heat the hot cell from room temperature to 100°C for 6 minutes and then take the series of spectra to observe the changes in absorption intensity of water as a function of time.

3.4.2 The determination of organic compounds suspended in waste water

3.4.2.1 Materials and equipment

1. Waste water

Waste water sample used in this research was collected from Klong Saen Saeb, the sampling point area is in front of Thep Lee La temple.

2. Home-made IR card
3. Beaker 100 mL
4. Stirring rod

5. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.4.2.2 Methodology and spectral acquisition

1. Pour 50 mL of waste water into a beaker and stand it at room temperature for 3 hours in order to organic compounds floated to be the thin film on the surface of water.

2. Take up the thin film of organic compound with stirring rod.

3. Attach the stirring rod onto the surface of home-made IR card.

4. Carefully pull off the stirring rod from the surface of home-made IR card.

5. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum. Before attach the home-made IR card onto the sample, record the spectrum of the home-made IR card as a reference spectrum for subtraction process.

3.4.3 The determination of atmospheric dust particles from the heavy road traffic

3.4.3.1 Materials and equipment

1. Home-made IR card and coated home-made IR card with Fluorolube (coating preparation was described in the title 3.3.1.3.2)

2. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.4.3.2 Methodology and spectral acquisition

1. Record the spectrum of home-made IR card and coated home-made IR card to collect the reference spectrum.

2. Place the home-made IR card and coated home-made IR card at the heavy road traffic (Pathumwan Road in front of Chulalongkorn University, about 1 m above the ground situated of road, 4 m from the gate Faculty of Science) for 7 days.

3. Take spectrum of the home-made and coated home-made IR cards when collecting time of dust particles at 1, 3, 5 and 7 days.

3.5 Surface-enhanced home-made IR card

3.5.1 Materials and equipment

1. *p*-Nitrobebzoic acid; analytical grade from Merck
2. Methanol; analytical grade from Merck
3. Home-made IR card
4. Beaker 5 mL
5. Volumetric flask 5 mL
6. Microsyringe
7. FT-IR spectrometer (Nicolet Magna 750 Series II)

3.5.2 Methodology

3.5.2.1 Preparation of *p*-nitrobenzoic acid and spectral acquisition

1. Dissolve 0.0845 g of *p*-nitrobenzoic acid with a small volume of methanol in a beaker.
2. Pour *p*-nitrobenzoic acid solution into a volumetric flask and make volume to be 5 mL with methanol.
3. Add a drop of *p*-nitrobenzoic acid solution onto the surface of home-made IR card. Allow the methanol to evaporate (~30s).
4. Place the home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum.

3.5.2.2 Preparation of surface-enhanced home-made IR card

The PTFE film was sputter coated with gold (Au) by using IB 3 ion coater system (Eiko). The sputtering rate was controlled at 3 mA/min to give a gold thickness of approximately 50 ^{o}A.

3.5.3 SEIRA spectral acquisition

1. Record the spectrum of coated PTFE film as a reference spectrum
2. Drop 1 drop of solution of *p*-nitrobenzoic acid onto the coated PTFE film with use of a microsyringe, the solvent was allowed to evaporate in the air.

3. Place the coated home-made IR card with sample into the sample compartment of FT-IR spectrometer and record the spectrum