

CHAPTER 2

EXPERIMENTAL TECHNIQUES

2.1. Chemicals and Instruments

In this chapter, the solvents mixed with the paints to reduce viscosity of the paints are to be mentioned. They were selected according to the principles stated in CHAPTER 1. Apparatus and instruments used in this research such as gas chromatography, film applicator and viscosity cup and experimental methods will also be presented.

2.1.1 Chemicals

2.1.1.1 Solvents

The solvents, source and price used in this research (compared interval time an experiment), are shown in Table 2.1.

Table 2.1 Solvents used with sources and prices. (Explored 24/02/48)

Entry	Solvents	Grade	Price (Bahts)	Source
1	1-Butanol	A.R.	1,010.-/2.5 l	Mallinckrodt, USA
2	n-Butyl acetate	A.R.	640.-/500 ml	Analyticals Carlo Erba, France
3	Ethanol	A.R.	890.-/2.5 l	Carlo Erba Reagenti, France
4	Ethyl acetate	A.R.	560.-/2.5 l	Lab Scan, Thailand
5	Methanol	A.R.	980.-/2.5 l	Lab Scan, Thailand
6	1-Propanol	A.R.	1,100.-/4 l	Lab Scan, Thailand
7	2-Propanol	A.R.	465.-/2.5 l	BDH, England
8	Propyl acetate	A.R.	900.-/250 ml	Fluka, Switzerland
9	Toluene	A.R.	540.-/2.5 l	Lab Scan, Thailand
10	Xylene	A.R.	540.-/2.5 l	Merck, Germany
11	o-Xylene	A.R.	7,650.-/2.5 l	Merck, Germany
12	p-Xylene	A.R.	4,680.-/2.5 l	Merck, Germany
13	m-Xylene	A.R.	3,231.-/2.5 l	Fluka, Switzerland
14	Thinners (Toluene:2-propanol:Ethyl acetate; 6:3:1)			Siam Sindee Co., Ltd.

2.1.1.2 Paints

All samples of paints were obtained from Siam Sindee Co., Ltd., as shown in Table 2.2.

Table 2.2 Paint samples for plastic printing under investigations.

Entry	Paints	Code	Source	Remarks
1	Brown	NEH 7494	Hsin Mei Kuang Co., Ltd.	Supported by
2	Orange	NEH 3528	(Samutsakhon, Thailand)	Siam Sindee Co., Ltd.
3	Green	NEH 6202		(Songkla, Thailand)
4	Red	NEH 3519		

2.1.2 Instruments

2.1.2.1 Gas Chromatography The studies of volatility of solvents were performed with a Hewlett-Packard Model 5890 Series II GC terminal equipped with an HP-1 capillary column [cross-linked methyl silicone gum phase (25 m x 0.32 mm i.d.)] and a flame ionization detector (FID) at 250 °C. Sample aliquots of 10 µl were injected (split-less)(injector temperature 250 °C) by using a gas-tight syringe (Hamilton Company, USA). The column temperature was programmed: 50 °C to 180 °C, at 20 °C/min and dried nitrogen of high purity with a flow rate 35 cm³/min was used as carrier gas. The addition gases for the flame ionization detector were hydrogen (40 cm³/min) and air (400 cm³/min).

2.1.2.2 Film applicator is used for film coating to measure the adhesion on plastic. Film application was made from materials resistant to corrosion and solution. A shape of this equipment is a rectangular prism in which the lower part was made to have a gap with the width of approximately 100±2 µm, from the floor that this equipment was placed to lower its ridge (TIS 285 Part 16, 1981), as shown in Figure 2.1.

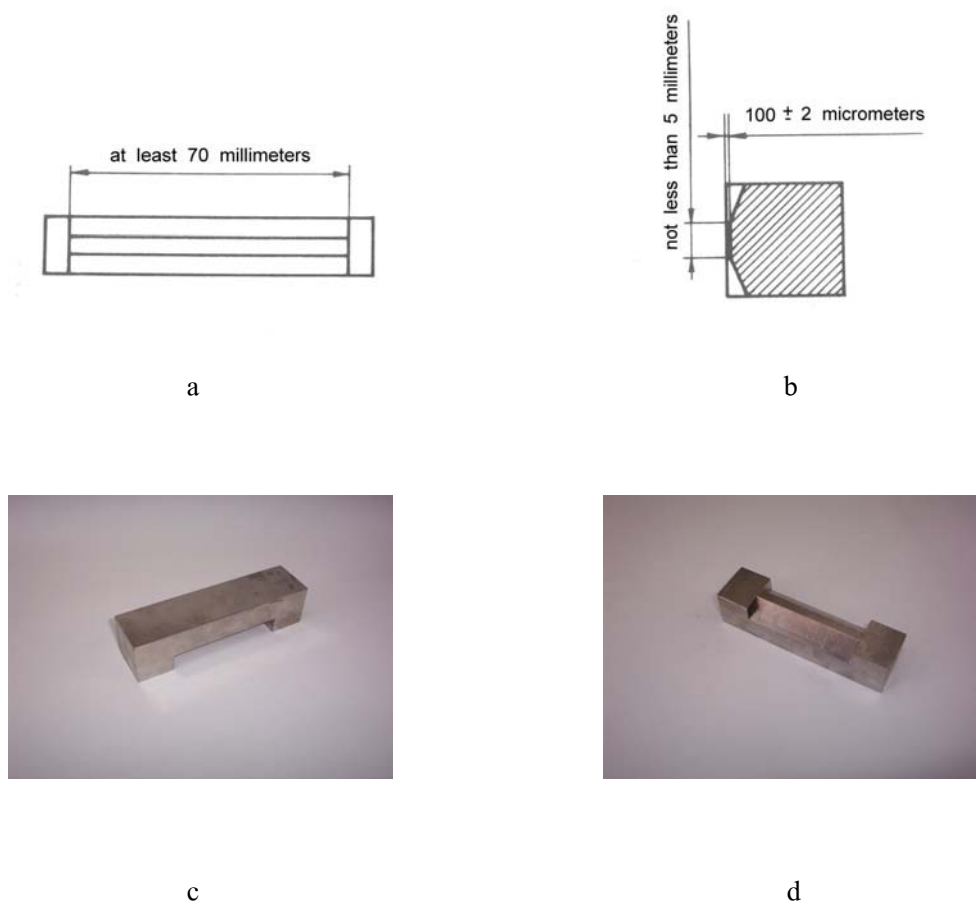


Figure 2.1 A film applicator; a) and b) Drawing showing dimensions of a film applicator.

c) and d) Film applicator when placed and turned over, respectively (TIS 285 Part 16, 1981).

2.1.2.3 Viscosity cup is the tool to determine viscosity of paints. Viscosity cup No. 4 was used to measure viscosity, purchased from Obrig Co., Ltd. (Thailand), as shown in Figure 2.2 to be corresponding to that of Siam Sinee Co., Ltd. This equipment was made from materials resistant to corrosion and solution.



Figure 2.2 Viscosity cup No. 4.

2.1.2.4 Thermometer was used together with viscosity cup to measure temperature during viscosity measurement and preparation of static headspace (E-mil P10116, Lloyd's Register Quality Company, UK).

2.1.2.5 Stopwatch was used to determine flow time of sample during viscosity measurement (Alba, Japan).

2.1.2.6 Tape was used to test adhesion of film on plastic after film coating [cellulose tape (Panfix), Nichiban Co., Ltd., Japan].

2.2. Procedures

2.2.1 Solvent selections

First of all, the solvents for an experiment are the mixed solvent currently used in Siam Sindee Co., Ltd. It consists of toluene, 2-propanol and ethyl acetate with the ratio of 6:3:1 by volume, respectively. It was used in 2 preliminary studies as follows:

1. The study of the same solvents as the currently used solvents, toluene:2-propanol:ethyl acetate, with different ratios of mixing totally 36 samples (Table 2.3).

Table 2.3 Various ratios of solvent mixtures of toluene, 2-propanol and ethyl acetate, respectively.

Entry	Ratios of solvent mixtures (v/v)		
	toluene	2-propanol	ethyl acetate
1	1	8	1
2	1	7	2
3	1	6	3
4	1	5	4
5	1	4	5
6	1	3	6
7	1	2	7
8	1	1	8
9	2	7	1
10	2	6	2
11	2	5	3
12	2	4	4
13	2	3	5
14	2	2	6
15	2	1	7
16	3	6	1
17	3	5	2
18	3	4	3
19	3	3	4
20	3	2	5
21	3	1	6
22	4	5	1
23	4	4	2
24	4	3	3
25	4	2	4

Table 2.3 (Continued)

Entry	Ratios of solvent mixtures (v/v)		
	toluene	2-propanol	ethyl acetate
26	4	1	5
27	5	4	1
28	5	3	2
29	5	2	3
30	5	1	4
31	6	3	1
32	6	1	3
33	6	2	2
34	7	2	1
35	7	1	2
36	8	1	1

2. The study of the same ratio of solvent mixing 6:3:1 by volume, with different kinds of solvent with similar polarities to toluene, 2-propanol and ethyl acetate, respectively, totally 45 samples (Table 2.4).

Table 2.4 Various kinds of solvent with the ratio of 6:3:1 by volume.

Entry	Solvents		
	6	3	1
1	o-Xylene	Ethanol	Butyl acetate
2	o-Xylene	Ethanol	Propyl acetate
3	o-Xylene	Ethanol	Ethyl acetate
4	o-Xylene	1-Butanol	Butyl acetate
5	o-Xylene	1-Butanol	Propyl acetate
6	o-Xylene	1-Butanol	Ethyl acetate
7	o-Xylene	1-Propanol	Butyl acetate
8	o-Xylene	1-Propanol	Propyl acetate
9	o-Xylene	1-Propanol	Ethyl acetate
10	p-Xylene	Ethanol	Butyl acetate
11	p-Xylene	Ethanol	Propyl acetate
12	p-Xylene	Ethanol	Ethyl acetate
13	p-Xylene	1-Butanol	Butyl acetate
14	p-Xylene	1-Butanol	Propyl acetate
15	p-Xylene	1-Butanol	Ethyl acetate
16	p-Xylene	1-Propanol	Butyl acetate
17	p-Xylene	1-Propanol	Propyl acetate
18	p-Xylene	1-Propanol	Ethyl acetate
19	m-Xylene	Ethanol	Butyl acetate
20	m-Xylene	Ethanol	Propyl acetate
21	m-Xylene	Ethanol	Ethyl acetate

Table 2.4 (Continued)

Entry	Solvents		
	6	3	1
22	m-Xylene	1-Butanol	Butyl acetate
23	m-Xylene	1-Butanol	Propyl acetate
24	m-Xylene	1-Butanol	Ethyl acetate
25	m-Xylene	1-Propanol	Butyl acetate
26	m-Xylene	1-Propanol	Propyl acetate
27	m-Xylene	1-Propanol	Ethyl acetate
28	Toluene	Ethanol	Butyl acetate
29	Toluene	Ethanol	Propyl acetate
30	Toluene	Ethanol	Ethyl acetate
31	Toluene	1-Butanol	Butyl acetate
32	Toluene	1-Butanol	Propyl acetate
33	Toluene	1-Butanol	Ethyl acetate
34	Toluene	1-Propanol	Butyl acetate
35	Toluene	1-Propanol	Propyl acetate
36	Toluene	1-Propanol	Ethyl acetate
37	Xylene	Ethanol	Butyl acetate
38	Xylene	Ethanol	Propyl acetate
39	Xylene	Ethanol	Ethyl acetate
40	Xylene	1-Butanol	Butyl acetate
41	Xylene	1-Butanol	Propyl acetate
42	Xylene	1-Butanol	Ethyl acetate
43	Xylene	1-Propanol	Butyl acetate
44	Xylene	1-Propanol	Propyl acetate
45	Xylene	1-Propanol	Ethyl acetate

2.2.2 The determination of solvent evaporation

After selecting solvents to be suitable for film coating, the evaporation rate was determined by using gas chromatography. The sample was taken from headspace vial and then introduced into column by using gas-tight syringe and external standard method was used to calculate solvent quantity.

2.2.2.1 Effect of time to the amount evaporating of the mixture

To understand the effect of time to the amount evaporating, the experiment was performed by using toluene, xylene, 1-propanol, ethyl acetate and n-butyl acetate with the concentration of 10% by volume, which is the representative of the solvents. The substances analyzed were prepared in headspace bottles and left at the temperature 25 ± 3 °C. The vapor was taken from the headspace with the volume of 10 μ l at the time of 15, 25, 35, and 65 min.

2.2.2.2 The effect of stirring to the amount evaporating

The representative for studying the effect of stirring rate is toluene, xylene, 1-propanol, 2-propanol, ethyl acetate and n-butyl acetate with the concentration of 10% by volume. The 10 μ l of the vapor from headspace were injected into the column with the stirring rates of 0, 600, 900 and 1200 rpm for 25 min.

2.2.2.3 The effect of temperature to the amount evaporating

The experiment was performed by using toluene, xylene, ethyl acetate, n-butyl acetate, 1-propanol and 2-propanol as a representative with the concentration of 10% by volume. The vapor was taken from the gaseous part of the headspace with the volume of 10 μ l and injected into the column. The temperatures of the headspace were set at 40, 50 and 60 °C for a period of 25 min.

2.2.2.4 The determination of the evaporation rate of solvent mixtures

To measure the evaporation rate of the solvent mixture, the samples were prepared in headspace bottles. Then they were dipped into the water bath at the temperature of 40 °C. 10 µl of the vapor was taken from the bottle and injected into the column periodically at the time of 5, 15, 25 and 35 min.

The signals from the detection were taken to calculate the quantity of each solvent by making use of the standard curve. The amount of solvent was then used to plot the graph as a function of time. The evaporation can then be determined from the slope (Glavchev *et al.*, 2003).

2.2.3 The measurement of viscosity

The ink and solvent mixtures were mixed in 500 ml beaker with the ratio of 1:0.5*. 150 ml of the solvent mixtures was added into 300 ml of the paint. The sample was blended homogeneously without air bubbles or contaminants. Then, the sample was stirred for approximately 10 min at constant speed by using magnetic stirrer and left for 10 min. After that the viscosity was measured by immersing the viscosity cup No. 4 into the sample until the sample was filled in the cup. Then, the viscosity cup was lifted together with the triggering of the stopwatch when the sample begins to exit from the orifice of cup until the line from the orifice breaks (TIS 285 Part 14, 1981).

* The mixing ratios between paint and solvent mixtures (Thinners) is 1:0.5. This ratio is corresponding to that used in Siam Sindee Co., Ltd.



Figure 2.3 Viscosity measurements by using viscosity cup No. 4.

2.2.4 Film Coating on plastic

The plastic (HDPE plastic) was cut to obtain the size of 100 x 200 mm and put on a smooth glass plate before coating the film. Then, the sample was stirred until homogeneously blended. After that the sample was dropped onto plastic flange (approximately 15-20 drops) without any air bubbles. The film was coated on plastic by the film applicator and left to obtain a dry film. The sample was then taken to measure the adhesion.

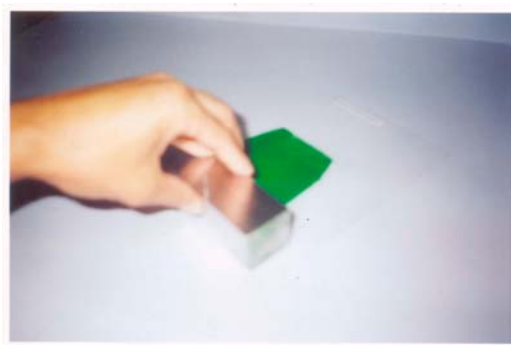


Figure 2.4 Coating the paint onto plastic by film applicator.

2.2.5 The measurement of adhesion of film on plastic

A sharp edge of a knife was used to scratch onto a dry film. The assay was performed by grid cut: 20 mm long, 2 mm apart and six cuts were made. Then, the center of the tape was placed over the grid and the area of the grid was smoothed with a finger. The tape was removed by seizing the free end and pulling rapidly. The adhesion was evaluated as follows (ASTM D 3359, 1979):

Classification	A characteristic of adhesion for coating film on substrate
5	None of the squares of the lattice is detached.
4	Less than 5% of the area of the squarely intersections of film coating on substrate is detached.
3	5 to 15% of the area of the squarely intersections of film coating on substrate is detached.
2	15 to 35% of the area of the squarely intersections of film coating on substrate is detached.
1	35 to 65% of the area of the squarely intersections of film coating on substrate is detached.
0	More than 65% of the area of the squarely intersections of film coating on substrate is detached.

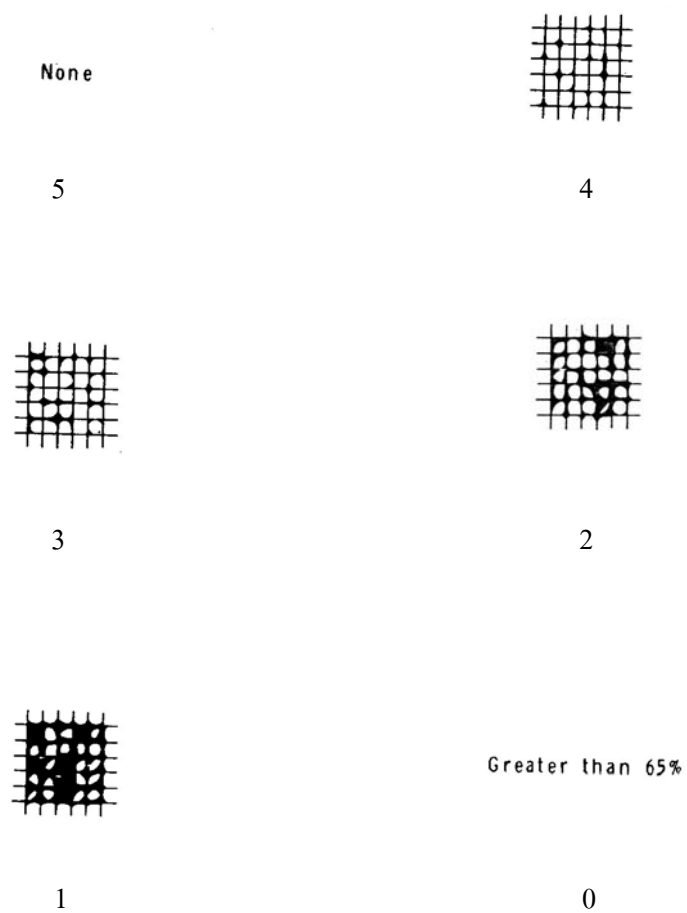


Figure 2.5 Classification of adhesion test results. (ASTM D 3359, 1979)