CHAPTER 2
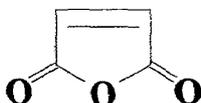
EXPERIMENTAL

EXPERIMENTAL

2.1 RAW MATERIALS

2.1.1 Acids and Acid Anhydrides

a- Maleic anhydride (MA)

Systematic name: cis-butenedioic anhydride

Formula:

This dibasic acid was obtained from Aldrich Chemical Co. Ltd. (UK). It is a white powder, having molecular weight 98.06, melting point 54-56°C and boiling point 200°C.

b- Acrylic acid (AA)

Formula:

$$H_2C=CH-COOH$$

It was obtained from Aldrich Chemical Co. Ltd. (UK) with the following specifications:

molecular mass 72.06 g/mol, boiling point 139°C, melting point 13°C, and density 1.051g/cm³.

c- Methacrylic acid (MA)

Formula:

$$H_2C=C-(CH_3)-COOH$$

It was purchased from Aldrich Chemical Co. Ltd. (UK) with the following specification:

Molecular mass 86.09 g/mol, boiling point 163°C, melting point 16°C, and density 1.015g/cm^3.

2.1.2 Glycols

a- Ethylene glycol (EG)

Systematic name: 1, 2-dihydroxy ethylene.
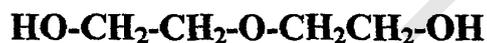
Formula:

$$HO-CH_2-CH_2-OH$$

EG was obtained from Aldrich Chemical Co. Ltd. (UK). It is a colorless, liquid, and miscible with water having molecular mass 62.07 g/mol, density 1.113 g/cm^3, melting point -13°C, boiling point 196-198°C.

b- Diethylene glycol (DEG)

Systematic name: 2-hydroxyethylether.

Formula:

$$HO-CH_2-CH_2-O-CH_2CH_2-OH$$

DEG was obtained from Aldrich Chemical Co. Ltd. (UK). It is a colorless, liquid, and miscible with water having molecular mass 106.12 g/mol, density 1.118 g/cm^3, melting point -10°C, and boiling point 245°C.

c- Tetraethylene glycol (TEG)

Systematic name: 2, (2-hydroxyethylether).

Formula:
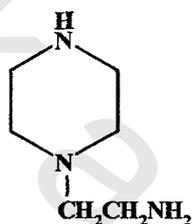
$$O-(CH_2-CH_2-O-CH_2CH_2-OH)_2$$

TEG was obtained from Aldrich Chemical Co. Ltd. (UK). It is a colorless, liquid, and miscible with water having molecular mass 194.23 g/mol, density 1.125 g/cm^3, melting point -6°C, boiling point 314°C.

2.1.3 Hardener, Catalysts, Inhibitor, Activator and Initiator

1- Hardener

1-(2-Amino ethyl) piprazine (AEP)

Formula:

It was obtained from Aldrich Chemical Co. Ltd. (UK). It has molecular mass 129.21 g/mol, melting point 93°C, boiling point 218-222°C, density 0.985 g/cm^3 and n_D^{20} 1.5.

2- Catalyst

a-Triethylamine (ET$_3$N)

Formula:

$$(C_2H_5)_3\,N$$

It was obtained from Aldrich Chemical Co. Ltd. (UK) with the molecular weight 101.19 g/mol, melting point -115°C, boiling point 88.8°C and density 0.726.

### b- Manganese acetate (Mn-acetatae)

Formula:

$$Mn\ (CH_3CO_2)_2$$

It was obtained from Sigma Chemical Co. Ltd. (UK) with the molecular weight 173.03 g/mol. Used as Trans – esterification catalyst for glycolsis of PET.

c- Triphenyl phosphite (Ph₃P)

Formula:

$$(C_6H_5O)_3P$$

It was obtained from Aldrich Chemical Co. Ltd. (UK) with the molecular mass 310.29 g/mol. milting point 22-24°C, boiling point 360°C and density 1.184.

3- Inhibitor

Hydroquinone (HQ)

Formula:

It is a white crystal obtained from Aldrich Chemical Co. Ltd. (UK) with the molecular mass 326.44 g/mol., melting point 173-175. HQ used as an inhibitor to avoid the polymerizable unsaturated polyester obtained.

4- Initiator

### Methyl Ethyl Keton Peroxide (MEKP)

Formula:
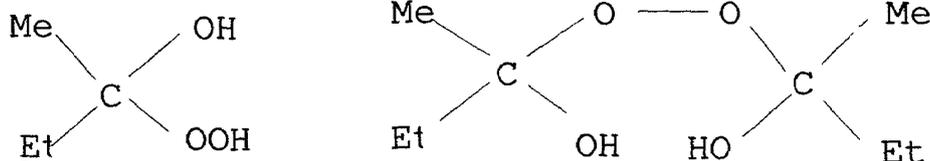
It is a colorless liquid obtained from SIR Company. MEKP was used as initiator for curing of unsaturated polyester.

5- Activator

Cobalt Octoate (CO)

It is a violet liquid obtained from SIR Company. It is a 10% solution of cobalt octoate in styrene and used as an activator for curing of unsaturated polyester.

2.1.4 Vinyl Monomer (Styrene)

Systematic name: phenylethylene or vinylbenzene.
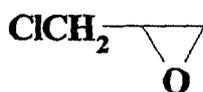
Formula:
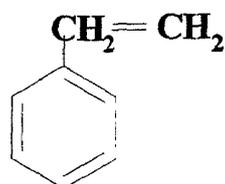
It is a colorless, liquid, containing hydroquinone as an inhibitor. It is immiscible with water but miscible with benzene, toluene and carbon tetrachloride.

Quoted specifications of styrene monomer, which was obtained from SIR Company. It has molecular mass 104.15 g/mol, m. p. -310°C, b. p.145°C, and n_D^{20} 1.5458.

2.1.5 Epichlorohydrin (EC)

Systematic name: (1- chloro-2, 3- epoxy propane)

Formula:

$$ClCH_2 \overset{}{\underset{O}{\triangledown}}$$

It was obtained from Aldrich Chemical Co. Ltd. (UK) with the molecular weight 92.53 g/mol. m. p. -57°C, b. p. 115-117°C and density 1.183.

2.1.6 Poly (ethylene terephthalate), PET

Formula:

$$\left[\overset{O}{\underset{}{\overset{\|}{C}}} - \bigcirc - \overset{O}{\underset{}{\overset{\|}{C}O}} \ CH_2 CH_2 O \right]_n$$
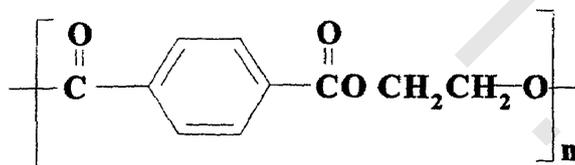
PET waste was obtained from collected beverage bottles. The intrinsic viscosity of PET in m-chlorophenol at 25°C was found to be equal 0.015dm^{3}Kg^{-1}. The weight average molecular weight was determined by gel permeation chromatography (GPC) was close to 63,200 g mol $^{-1}$.

2.1.7 Commercial Unsaturated Polyester Resin (UP1)

Unsaturated polyester resin (UP1) obtained from SIR Company. The resin is based on (isophthalic acid with propylene glycol and maleic anhydride) as a commercial source in this study.

2.1.8 Solvents

High purity grade toluene, ethanol, acetone, pyridine, acetic Acid & its anhydride, and methylene chloride (purchased from Aldrich) were used as received.

2.2 TECHNIQUES

2.2.1 DEPOLYMERIZATION OF PET

The PET waste (beverage bottles) was depolymerized using (TEG or DEG) with weight ratio (1:1 wt %) in the presence of 0.5% (by weight) of manganese acetate based on the weight of the PET. The reaction carried out in four necked flask fitted with mechanical stirrer, nitrogen inlet, reflux condenser and thermometer. The reaction was completed under nitrogen atmosphere at temperature $200^{\circ}C$ for 4 hour, and at 210-$230^{\circ}C$ for 3 hour. After completion of the glycolysis, the product cooled to room temperature, dissolved in suitable amount of methylene chloride (CH_2Cl_2) and shaken vigorously with an equal amount of 5% aqueous sodium chloride solution to remove any unreacted glycol and remaining catalyst. The organic layer was collected and washed several times with water. Finally, purified oligomers were obtained after removal of the solvent by rotary evaporation.

The purified glycolysates of PET with DEG and TEG are designated as GD and GT, respectively. The product was subjected to analyze the

hydroxyl value, which was determined by the conventional acetic anhydride pyridine method **[Sorenson et al. 1968]**.

2.2.2 SYNTHESIS OF UNSATURATED POLYESTER (UP) RESIN

The UP resin was prepared by reacting the glycolyzed products with glycol (EG) and with MA. The unsaturated polyester (UP2) having different chemical structure was obtained. MA was added at the ratio of OH/COOH 1.1/1 (mol/mol). The reactions were carried out in an esterification reactor (as represented in *Figure 2.1*) equipped with stirrer, thermometer, Dean-Stark side arm, and nitrogen inlet. The reactants were heated from room temperature to 180°C in nitrogen atmosphere; in a period equals 1.5h and held at this temperature for 4hr. Finally, the temperature was raised and kept at 200°C for 1hr. Then the reaction product was permitted to cool to 60°C and hydroquinone (0.1 wt %) was added as inhibitor to avoid the polymerization of the product.

2.2.3 PURIFICATION OF UNSATURATED POLYESTER (UP) RESINS

The unsaturated polyester resin (UP2) was dissolved in adequate amount of CH_2Cl_2 and then an equal volume of distilled water was added to reaction products. The mixture was shacked by means of a flask shaker for half an hour and it was then left to separate. The organic layer was separated and the solvent was evaporated by means of a rotary evaporator. The purified samples were placed in a vacuum oven and weighed at intervals of time until constant weight was attained for the sample. The polyester was viscous liquids having dark yellow to pale brownish color was diluted by a sufficient quantity of styrene monomer to give 60% solution of polyester.

1. Condenser.
2. Stirrer.
3. Thermometer.
4. Adapter (Trap).
5. Double Jacket Reactor.
6. Measuring Cylinder.
7. Contact Thermometer.
8. Stand.
9. Thermostat.
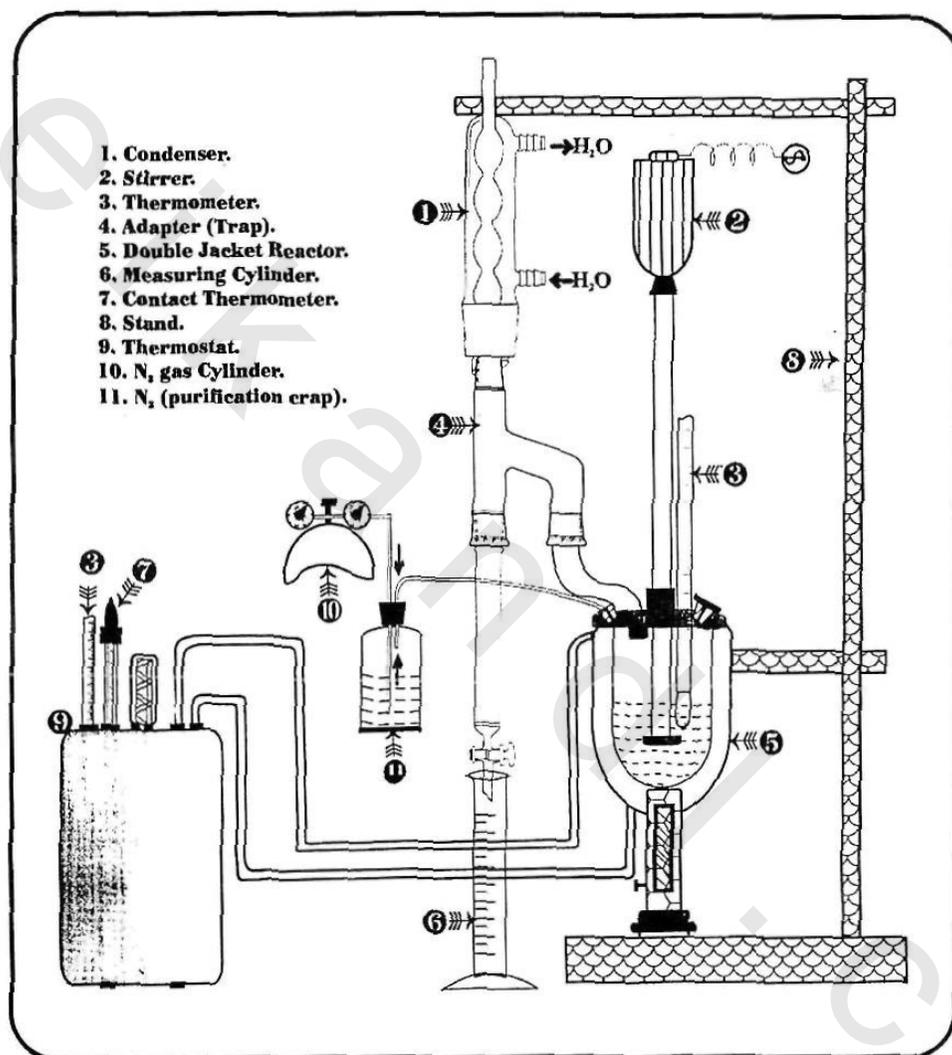10. N₂ gas Cylinder.
11. N₂ (purification crap).

Figure (2.1): Polyesterification Reaction Kettle.

2.2.4 SYNTHESIS OF GLYCIDYL ETHER OF RECYCLED PET

In a humidity-protected 500ml glass reactor equipped with a mechanical stirrer, dropping funnel and condenser, the purified glycolysate GD or GT (0.11 mol) was mixed with 1% (wt% reacted to weight of GT) of $BF_3.ET_2O$ and heated at 40°C. EC (10 mol) was gradually added to the reaction mixture during 2 hour. The reaction mixture was heated at 25°C for 30 min and after cooling to 40°C, 0.22 mol of NaOH (as 50% of aqueous solution) was added to the reaction mixture (*figure 2.1*). Temperature of the reaction mixture was raised up to 80°C for 30 min. After cooling, the mixture was neutralized with 50 ml of acetic acid (50%); and 150 ml of toluene was added and the reaction mixture was filtered to remove sodium salts. The filtrate washed four times with 150ml of hot water (60-80°C), toluene evaporated off from organic layer by rotary evaporator.

The glycidyl ether of GT and GD are designated as GTE and GDE, respectively. The products were obtained as viscous products and the epoxy content, EC, was determined as reported method [Dobinson et al. 1996].

2.2.5 SYNTHESIS OF ACRYLATE AND METHACRYLATE VINYL ESTERS OF "GTE"

GTE (0.11 mol) was mixed with (0.2 mol) of acrylic acid, AA or methacrylic acid, MA, 1% of triphenyl phosphite, 0.5% of hydroquinone and diluted with 5% of toluene into reaction reactor (*figure 2.1*). The reaction temperature rose up to 95°C and the reaction was preceded until 0.2mol of water was separated using Dean–Stark separator. The synthesized acrylate and methacrylate oligomers of GTE

were purified by extraction with ethanol, distilling off the solvent under reduced pressure. The viscous products were washed with sodium bicarbonate, Na_2CO_3 aqueous solution to remove unreacted acid until the washings become neutral. The products were dried over anhydrous sodium sulphate, $Na_2SO_4.2H_2O$, filtered and vacuum–distilled. They had a yellow oily liquid appearance.

The diacrylate and dimethacrylate oligomers of GTE are designated here as GTA and GTM, respectively.

2.3 PHYSICAL MEASUREMENTS

2.3.1 ^{1}HNMR-SPECTROSCOPY

The prepared compounds were dissolved in $CDCl_3$ and analyzed using Varian ^{1}HNMR spectrometer model JNM-EX (300 MHz) for determining their chemical structures.

2.3.2 IR-SPECTROSCOPY

The unsaturated polyesters, glycolyzed monomer, acrylates and methacrylate esters were characterized by FTIR-spectroscopy using FTIR-spectrophotometer type [Mattson-Infinity series bench to P 961]. The wave number and intensities of IR bands of the different types of the function groups were determined a between of 500-4000 cm^{-1}.

2.3.3 GPC TECHNIQUE

The molecular weights of modified products of PET "GTA and GTM", "GTE and GDE" and the oligomers "GT and GD" were measured by gel permeation chromatography (GPC) water model 600E. The measurements were recorded at 30°C, under UV-visible

spectrophotometer water, mobile phase toluene HPLC grade, column, styragel, columns and injection volume chart [Iglesias et al. 1994, 1995, 1997].

2.3.4 DETERMINATION OF MOLECULAR MASS OF GT, GD AND (UP2) RESIN

The molecular weight of glycolyzed (GT and GD) and unsaturated polyester resin (UP2) was determined by the end-group analysis. UP2 was analyzed for both acid number (A) and hydroxyl value (B) in (mg KOH/g of resin). But, the GT and GD were analyzed for hydroxyl value (B) only.

The hydroxyl value was determined by weighing 2.0 g of UP2 to the nearest milligram into a 100 ml round –bottom flask. To dissolve the sample, 25 ml of a mixture of (5 ml acetic anhydride and 25 ml pyridine) was added by pipette. The solution was refluxed for 1h. the condenser was then washed out by the addition of 5 ml water through the top, and the heating was continued for 5 min. The heat was then removed and the condenser tube and tip washed with 25 ml methanol. When the mixture has cooled to room temperature, it was titrated with approximately 0.5N standard potassium hydroxide solution to phenolphthalein end-point. This was value "a", taken as mg KOH/g. a blank is then run on a mixture of the same volumes used above of acetic anhydride-pyridine reagent and water, this value is "b'.

$$OH_{no.} = \frac{0.5N \times 56.1 \times (a-b)}{Wt \ of \ sample} \qquad (2.1)$$

The acid value was determined by titrating the solution of the weighed quantity of resin in acetone, with a standard alcoholic KOH solution ($\simeq 0.5N$) using phenolphthalein as an indicator.

$$COOH_{no.} = \frac{0.5N \times 56.1 \times (a-b)}{Wt \text{ of sample}} \qquad (2.2)$$

The number average molecular weight M_n was then, calculated from the following equation [Sorenson et al. 2001].

$$\langle Mn \rangle = \frac{2 \times 56.1 \times 1000}{B} \qquad (2.3)$$

$$\langle M_n \rangle = \frac{2 \times 56.1 \times 1000}{A + B} \qquad (2.4)$$

2.3.5 VISCOSITY MEASUREMENTS

Dynamic viscosity of resins was determined with a Brookfield, Model DV-111 viscometer at different temperatures and, can be determine the gel time of the cured resin by measuring the change of the viscosity of mixture as function of time at constant temperature and shear rate.

Using the initial viscosity with time, the critical value for the viscosity (η_c) and time (t_c) corresponding to the gel point were determined. This procedure was repeated at different temperatures 298, 308, 318, 328 K. The apparent activation energy of the curing process can be calculated from the slope of the lines using logarithmic graph ln t_c versus 1/T (K).

On the other hand, the curing activation energy can be determined from relation between critical viscosity (ln η_c) and 1/T (K) [Rai et al. 1993].

2.3.6 EPOXY GROUP CONTENT

The epoxy content of resins (GDE and GTE) is frequently expressed as weight per epoxide or epoxide equivalent weight (EEW), which is defined as the weight in grams, which contains one-gram equivalent epoxide. In this respect, 0.5 g of the sample was dissolved in 20 ml/pyridine solution (1L pyridine + 16 ml concentrated HCl) and boiling in flask with reflux condenser for 30 min.

The excess of HCl was titrated against 0.5N KOH. In the same, time a control (blank) experimental carried out [Dobinson et al. 1996].

$$\text{Epoxy group content} = \frac{(a - b) \times K \times 0.028 \times 100}{g} \qquad (2.5)$$

Where, **a:** is quantity of KOH in ml taken for titration the acid in the control experiment (blank).

b: quantity of KOH in ml taken from titrating the acid in sample.

K: constant = 0.998.

g: weight of the sample.

2.3.7 DETERMINATION of PEAK EXOTHERM OF CURING

The curing exotherm of unsaturated polyesters with styrene and cross-linkers and the prepared epoxy with hardener amine curing agent were measured by Digitron digital differential thermometer, type K, model 3202 with a resolution 0.1°C, as described in the previous work

[Abdel-Azim A.A. 1995 and 1996]. The reaction mixture of (UP & cross-linkers) was thermostated at (298, 308, 318, 328K) while, the reaction mixture of (epoxy & amine) was thermostated at (313, 333K) for about 5min. Then the appropriate amount of accelerator and initiator were added successively and mixed thoroughly for 1 min in case of UP. While (1wt %) of triethylamine was added in epoxy case. The reaction mixture was poured into a test tube and a glass bottle of 5 ml. To achieve the reproducibility of the measurements and affording a comparative study, the volume and the shape of the curing glass bottles, used in all experiments were kept constant. The curing zero time was considered of the moment at which the initiator is added to the reaction mixture of UP. The maximum curing temperature (T_{max}) was taken as the highest value in the temperature / time curve, whereas t_{max} is the time required acquiring T_{max}. The concentration of methyl ethyl keton peroxide, MEKP (initiator) and cobalt octoate, CO (accelerator) was kept constant all over the curing reactions as 2 and 0.2 (wt %), respectively. The concentration of GTA and GTM was varied from 5 to 20 (wt %) based on weight of UP and styrene mixtures. While, GTE and GDE were mixed with hardener amine and different weight ratios from 1-4 (wt %).

2.3.8 DETERMINATION OF MECHANICAL PROPERTIES OF CURED UP/VE RESINS

The following section is mainly concerned with measuring the comparative properties of the prepared polyester in the form of test specimens of standard shape. The cured rods were immersed in acetone

solution for 24h to extract soluble materials and then dried in oven at temperature of curing 25, 35, 45 and $55^{\circ}C$.

Compression is the change in length produced in a longitudinal section of the test specimen by a compressive load. It is expressed in centimeter. The following parameters were measured

- ### COMPRESSIVE STRENGTH (σ_Y)

Compressive strength may be defined as the maximum compressive stress (nominal) carried by a test specimen at the moment of rupture. It is expressed in N/m^2

- ### YOUNG'S MODULUS (E_Y)

It is the elastic modulus at (σ_y).

Shimadzu Universal Testing Machine measured the compressive stress-strain curves for all samples under investigation; UH-A series model 2000 KN Computer-Controlled Hydraulic Servo System. Testing of mechanical properties was carried out according to [**ASTM method, D695 – 44T**].

This universal testing machine performs tension, compression, transverse, and bending tests. The machine's hydraulic loading system has a wide control range of loading speeds and permits smooth loading. For every material, the stress-strain curve was given as an average of five specimens. The test specimen was in the form of a right cylinder whose length was twice its principal width or diameter and whose ends are parallel. These cylinders were prepared by casting the resin in test

tubes having the same diameter. The produced rods cut to pieces of appropriate length. Then they examined for their mechanical properties.

2. 3.9 TESTING OF THE EPOXY COATINGS

2. 3. 9.1 Preparation of steel panels for testing
[ASTM Method, D609-95]

Before blast cleaning and application of the coating system, the panel shall be free of all surface contaminates. Oil, grease, and other contaminates were removed by suitable solvent, detergent or potable water. The treatment involved the following steps:

- Remove the rust by hand tool cleaning (wire brush or sanding paper) to achieve the requirements of **St2, SIS 05590 and BS 7079.**

- The rust free panels wiped vigorously on both sides with a cloth wet with methanol.

- The panels were allowed to dry at room temperature (15 minutes). Then rubbed briskly with clean, lint less cloth to remove any adhering dirt.

- Then, the panels were dipped in phosphoric acid (1:1) at 25°C for one minute and rinsed immediately by distilled water for a minimum 15sec.

- The panels were dried at a temperature between 50°–100°C and coated immediately or stored in desiccators for future use.

- The panels should not be touched with the fingers during the process of preparation.

- The coated panels should be scribed to bare steel; the scribe line was approximately 10cm.

2. 3.9.2 Measurements of film thickness
[ASTM Method, D1005-95]

There are four methods of determining the dry film thickness (DFT) of paint:

a- *Non-destructive test gauge*

b- *Destructive test gauge*

c- *Test panels*

d- *Calculation*

Measuring DFT directly with non-destructive test is the most widely used method: The instrument used for measuring the dry film thickness (DFT) was the *"Minitest microprocessor coating thickness gauge, Model 2100"*, manufactured by ELECTRO – PHYSIK West Germany. The measuring range of the instrument is 0-1.25mm and its accuracy +/-5 μm (1mm =1000 micron).

The instrument should be calibrated before use as per ISO 2808 as follow:

- Mount the probe to the instrument and turn on the instrument

- Place the probe onto unpainted smooth steel and hold it there

The instrument shall display 0μm. If the instrument displays other values than 0, the instrument must be adjusted. Do this while you hold the probe onto the steel.

- Take a non- magnetic shim with a defined thickness e.g. 100μm and place it into the smooth steel surface. Place the probe onto the shim and hold it there. (The instrument shall display 100μm. If the instrument displays other values than 100, the instrument must be adjusted. Do this while you hold the probe onto the steel).

- You have now adjusted the instrument for measurements in the area of 0-100 μm. But for your own sake check that you actually get 0 μm on smooth steel and 100 μm on the shim. If you still do not have, the correct values displayed do step 2 and 3 again until you do.

2.3.9.3 Film hardness by pencil test

[ASTM Method, D3363-92a]

The purpose of this test is to assess the ability of the surface coating to withstand scratching. A coated panel is placed on a firm horizontal surface. The pencil is held firmly against the film at a 45° angle (point away from the operator) and pushed away from the operator in a ¼-inch (6.5-mm) stroke. The process was started with the hardest pencil 6H and continued down the scale of hardness until a pencil found that for scratch hardness (the hardest pencil that will not scratch the surface of the film). Make a minimum of two determinations scratch hardness for each pencil.

2.3.9.4 Pull – Off strength of coating using portable adhesion testers [ASTM D4541 – 95]

This test method covers a procedure for evaluating the pull – off strength (commonly referred to as adhesion).

Generally, there are two field tests for the adhesion of coatings, the first is pull – off adhesion and the second is Hydraulic adhesion test, which is carried out as follow:

- Sand rough the dollies and area of the painted surface with a sandpaper
- Degease the dollies and the painted surface using a suitable solvent and dry them before use.
- Mix the 2 pack solvent less epoxy in the correct ratio before applying it on to the dollies, press the dollies to the surface and turn them 360° in order to ensure that most of the glue is squeezed out.
- Before the pull – off test is accomplished cut through the cured adhesive and the paint coating to the substrate , round the circumference of the test cylinder using a cutting device , extreme care is required to prevent micro – cracking in the coating, since such crack may cause failure at diminished strengths.
- The instrument measures the force in Kg / cm^2 , PSI or MPa

2.3.9.5 Flexibility (BEND) test
[ASTM Method, D522-93a]

The test was performed to determine the adhesion power of the varnish to the substrate by the bending apparatus. 5x15 cm tin panel was coated

by the varnish, and dried. The panel was placed into the bending apparatus (0.3 cm diameter) in such away that the surface of the panel was directed out side. The apparatus was then opened and the panel was examined carefully to find out any cracking or dislodging of the film as criterion of loss of consistency of the film. The varnish was considered satisfactory if no marks for cracking or dislodging are observed after bending.

2.3.9.6   Resistance   to   mechanical   damage   (IMPACT RESISTANCE) [ASTM D2794-93]

The impact resistance of the coating on the steel panels shall be tested by means of dropped steel ball of two pounds (890 gm) from a 1,00m height. After impact, no cracks shall be visible by a magnification of ten times. When the area of the impact and the area of 5mm around the impressed area shows flakes after scratching with a sharp needle the coating failed.

2.3.10 CHEMICAL RESISTANCE TESTING

❑   *Hot Water Resistance*
     [ASTM Method, D870-92]

The varnish was poured on a glass panel (3 x 5 in) and allowed to drain in nearly vertical position and allowed to dry for 48 hours at 25°C and 10 % relative humidity. The edges of the panels were sealed by paraffin wax. The coated panels were placed in a beaker containing about 7cm (2.5in) of distilled water at room temperature and allowed to remain in the water for 24 hours or other suitable period. The panel was then removed from the water, wiped carefully and allowed to dry at room

temperature. The panel was tested for any change. For longer testing period, the test liquid was changed at least every 72 hours to avoid contamination. To accelerate the test, the same procedure was applied with the exception that water was gently boiled throughout the immersion period, which may be from 15 minutes to several hours. The panels were removed and examined. The films were considered not affected when no film defects were observed after 24 hours immersing or 15 minutes in boiling water.

The procedures used to evaluate the hot water resistance of the coatings were as follows:

- The test panels were immersed in hot water (85°C) for 48h then; removed from the water, rinsed and lightly brushed in warm tap water.

- The test panels were cooled to room temperature (25°C).

- The coat was cut to bare steel with a razor blade as follow:

i) Two parallel cuts have 1.3 cm long, were made 0.6cm a part.

ii) One cut has 0.6cm long, was made between two adjacent ends of the parallel cuts.

- Attempts were made to lift the coating, between the parallel cuts, from the test panel with a dull knife blade. The coating failed the hot water resistance test if the coating could be lifted from the test panel and passed the test if the coating could not be lifted from the test panel [ASTM D1647-89].

❑   *Solvent Resistance Test*

[ASTM Method, D2792-69]

● *Immersion Testing*

The coated panels were immersed in benzene / mineral turpentine solvent mixture (1:3 by volume) for 15 minutes at room temperature. The panels were removed from the test solvent followed by drying in vertical position for one hour. The panels were then tested for any defects.

The coated panels were immersed in diesel fuel and the changes were observed periodically. Visual inspection is made to determine softening of the film or separation from the substrate, which may take the form of wrinkling or blistering. Results are rated by number, with 10 indicating virtually no effect, and 0 indicating complete failure. In-between scores are subjective **[Tess 1988]**.

● *Acetone Rub Test*

A cotton tissue was drained with acetone and then rubbed 100 times forwards and backwards over one spot of cured coating. A complete cross-linked polymer should show no changes on the film surfaces after this treatment **[Howard et al. 1977]**.

❑ *Acid and Alkali Resistance Tests*

The acid and alkali resistance of coated panels with epoxy were determined after immersion in 10% HCl and 5% NaOH aqueous solutions using distilled water. The period of test was 90 days at 38°C. The tasted panels were examined to determine the degree of adhesion and visual inspection of blister and cracks **[ASTM D1647-89]**.

❑ *The Resistance To The Corrosive Environments Was Assessed Using The Procedure Outlined In [ASTM C581-94].*

The large panels were cut into plaques (125 mm x 100 mm) and the edges sealed with resin. Plaques were immersed in the corrosive media (hydrochloric acid 32 wt%, sulphuric acid 25 wt%, sodium hydroxide 5 wt% solutions, and uncut Kerosene), in large glass bottles and placed in an oven at 66°C (150°F). The concentrated hydrochloric acid medium was replaced every 6 weeks. The sulphuric acid and sodium hydroxide were replaced at each withdrawal. Plaques were withdrawn for testing at 3 monthly intervals.

2. 3.11 CORROSION RESISTANCES

The coated steel panels to be tested were exposed to salt spray (Fog) corrosion cabinet, manufactured by Sheen Instruments LTD. England, for a period of 700 hours. This cabinet is used to measure the corrosion resistance of materials and components. This cabinet is designed to expose test pieces to aggressive environments that simulate prolonged exposure in natural environment. It gives better correlation between accelerated laboratory tests and "real-world" exposure. The salt solution is pumped to the atomizer via a variable speed peristaltic pump. This ensures accurate control and monitoring of the salt fog out rates [ASTM Method, B117-94].

✓ **Salt Solution**

The salt solution prepared by dissolving 5±1 parts by weight of sodium chloride in 95 parts of water.

✓ **Position of Specimens During Test**

The position of the specimens in the salt spray chamber during the test shall be supported or suspended between 15 and 30° from the vertical and preferable to the principal direction of horizontal flow of fog through the chamber.

✓ **Air Supply**

The air supply to the nozzle for atomizing the salt solution shall be free of oil and dirt and maintained between 69 and 172 KN/m^2 (10 and 25 psi).

✓ **Period of Test**

The test method does not prescribe the type of the test specimen or exposure periods to be used for a specific product. The period of test shall be as designated by the specifications covering the material or product being tested, but exposure periods of multiples of 24 h are suggested.

✓ The films were examined for any defects such as blistering and rusting.

2.3.12 Evaluation of Painted Specimens Subjected to Environments (SCRIBE FAILURE) [ASTM Method, D1654-92]

This test method covers the treatment of previously painted or coated specimens for accelerated and atmospheric exposure tests and their subsequent evaluation in respect to corrosion and blistering associated with corrosion, loss of adhesion at scribe mark and other film failure.

Painted or coated specimens subjected to a corrosive environment are evaluated by record the average (mean) maximum and minimum creep age from the scribe mark. Record creep values in millimeters, inches, or rating numbers as represented in *table (2.1)*.

Table (2.1): Rating of Failure at Scribe

Millimeters	Inches (Approximate)	Rating Number
Zero	0	10
Over 0 to 0.5	0 to 1/64	9
Over 0.5 to 1	1/64 to 1/32	8
Over 1 to 2	1/32 to 1/16	7
Over 2 to 3	1/16 to 1/8	6
Over 3 to 5	1/ 8 to 3/16	5
Over 5 to 7	3/16 to ¼	4
Over 7 to 10	¼ to 3/8	3
Over 10 to 13	3/8 to ½	2
Over 13 to 16	½ to 5/8	1
Over16 to more	5/8 to more	0