

CHAPTER III
MATERIALS AND TECHNIQUES

Materials and Techniques

3.1. MATERIALS

3.1.1. Epoxidized soybean oil

Epoxidized soybean oil (Ep-So) was delivered from Hobum company, Germany, under the trade name Edenol D 81, and has the following constant:

Table (3.1): Specifications of epoxidized soybean oil

Oxirane content (%)	6.5
Specific gravity	0.98
Viscosity (mPa.s.)	350
Iodine number	125-140

3.1.2. Heterocyclic compounds

Heterocyclic Compounds delivered from Sigma-Aldrich. They were used in preparation of thiol adducts. They were used without further purification. Some physical properties of these compounds are given in Table (3.2).

Table (3.2): Specifications of heterocyclic compounds

Chemical name	Molecular formula	Molecular weight	Melting point (°C)
2-Mercapto- Δ^2 -thiazoline	C ₃ H ₅ NS ₂	119.19	105-107°C
2-Mercaptobenzothiazole	C ₇ H ₅ NS ₂	167.25	179°C
2-Mercapto-5-methyl-1,3,4-thiadiazole	C ₃ H ₄ N ₂ S ₂	132.198	182 -186°C
3-Mercapto-4-methyl-4H-1, 2, 4-triazole.	C ₃ H ₅ N ₃ S	115,16	167 - 173 °C

3.1.3. Surfactants

Non-ionic emulsifier such as Tween 20, Spans 20, and Span 80 were used. They were delivered from Sigma-Aldrich. Their chemical structure can be shown in Figure (3.1).

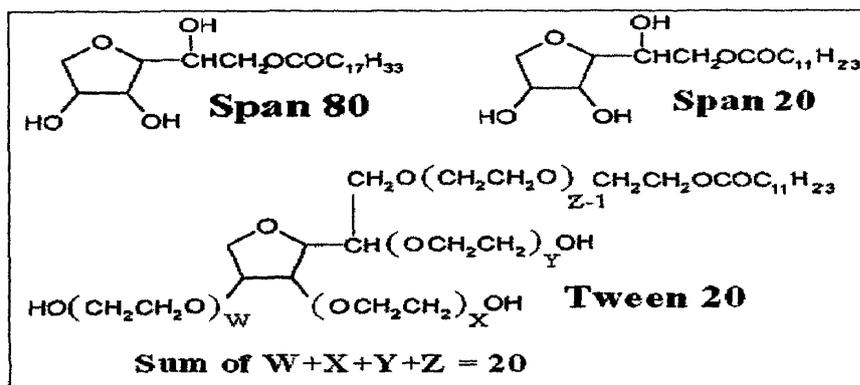


Figure (3.1): Chemical structure of surfactant

The properties of the used emulsifiers (Tween 20, Spans 20 and Span 80) are given in Table (3.3):

Table (3.3): Specifications of the emulsifiers

Trade name	Chemical composition	HLB	M.wt
Tween 20	POE (20) sorbitan monolaurate	16.7	1228
Span 20	Sorbitan monolaurate	8.6	346
Span 80	Sorbitan monooleate	4.3	428

3.1.4. Binders

3.1.4.1. Acrylic emulsion resin

An acrylic emulsion resin under the trade name Joncryl 8211 delivered from Johnson Company was used in water-borne acrylic formulations. It has the following specifications:

Table (3.4): Specifications of the acrylic emulsion resin

Non-volatile content by weight (%)	49.6
Viscosity Brookfield RTV (poise)	100
pH	7-8
Specific gravity	1.064
Particle size (micron)	0.125
High temperature stability	Good
Freeze thaw stability	Excellent
Mechanical stability	Excellent

3.1.4.2. Alkyd emulsion resin

An urethanated short oil alkyd based on linoleic rich oils was delivered from DSM Company, Germany, under the trade name Uradil AZ601 z-44. Its physical properties are given in the following Table (3.5):

Table (3.5): Specifications of the alkyd emulsion resin

Viscosity (mPa.s)	200-450
Solid content by weight (%)	43- 45
pH	6-7.5
Oil length (%)	25
Specific gravity	1.003
Flash point (°C)	> 100
Type of fatty acid	Linoleic rich
Neutralization	Tri ethylamine

3.1.4.3. Urabrid AC100 emulsion resin

A hybrid, Alkyd emulsion and polymer dispersion was delivered by DSM Company, Germany, under the trade name Urabrid AC100 Z-43. Its specifications are represented in the following Table (3.6):

Table (3.6): Specifications of the urabrid emulsion resin

Appearance	milky
Solid content (%)	42-44
Viscosity, Brookfield (mPa.s)	50-200
pH	6.0-8.0
Oil length (%)	20
Type of fatty acid	linoleic rich
Specific gravity	Approx. 1.050
Flash point (°C)	> 65

3.1.5. Fillers

Talc

Only talc was used as filler. It was delivered from El- Nasr Phosphate Company, Cairo, Egypt. Its specifications can be seen in the following Table (3.7):

Table (3.7): Specifications of talc

Silica (%)	60- 63
Magnesium oxide (%)	30- 32
Iron oxide (%)	< 1.0
Calcium oxide (%)	< 1.5
Aluminum oxide (%)	< 1.5
Acid soluble materials (%)	2.0
Water soluble materials (%)	0.1
Whiteness (%)	> 90
Particle size, mesh (%)	325

3.1.6. Pigments

3.1.6.1. Titanium dioxide

Rutile titanium dioxide was delivered from Du-pont Company under the trade name Rutile R-902 was used. It has the following specifications:

Table (3.8): Specifications of titanium dioxide

Color	White
Specific gravity	4.0
Oil absorption (g /100 g pigment)	17
Volatile content (%)	0.5
Titanium dioxide content (%)	91
Aluminum oxide content (%)	4.5
Silicon oxide content (%)	2
Others (%)	2
Particle size (μ)m	5

3.1.6.2. Zinc phosphate

Zinc phosphate, $Zn_3(PO_4)_2$, was delivered from Imperial Smelting Corp., Ltd., England, and it has the following specifications:

Table (3.9): Specifications of zinc phosphate

Color	White
Specific gravity	4
Oil absorption (g /100 g pigment)	17
Particle size (µm)	5

3.1.6.3. Zinc chromate

Zinc chromate was delivered from Samoral Trading Office, Cairo, Egypt. Its specifications are shown in the following Table (3.10):

Table (3.10): Specifications of zinc chromate

Molecular formula	ZnCrO ₄
Color	Yellow
Specific gravity	3.4

3.1.7. Paint additives

3.1.7.1. Coalescing solvents

3.1.7.1.a. Butyl glycol

Fast-evaporation glycol ether with an excellent balance of hydrophilic and hydrophobic character was used as a coalescing agent under the trade name of Dowanal EB (DOW Company). It has the following specifications

(3.11): Specifications of butyl glycol

Molecular formula	C ₆ H ₁₄ O ₂
Molecular weight	118.2
Specific gravity	0.9
Boiling point (°C)	171
Flash point (°C)	199
Evaporation rate (n-butyl acetate = 1)	0.079
Solubility in water	∞

3.1.7.1.b. Texanol

It is 2, 2, 4-trimethyl-1, 3-pentanediol monoisobutyrate. Texanol is a slow evaporating solvent, and it is water-insoluble coalescing agent for latex paints. It was delivered from Eastman Chemical Company. Its specifications are represented in the following Table (3.12):

Table (3.12): Specifications of texanol

Molecular formula	$C_{12}H_{24}O_3$
Molecular weight	216.3
Specific gravity (at 20 °C)	0.95
Boiling point (°C)	255
Flash point (°C)	120
Evaporation rate (n-butyl acetate = 1)	0.002
Solubility in water (at 20°C)	860 mg/l.

3.1.7.1.c. Iso-propanol

It was produced by TCI Company, and it was used as a water-soluble solvent for alkyd emulsion formulations. Its specification is shown in the following Table (3.13):

Table (3.13): Specifications of iso-propanol

Molecular formula	C_3H_8O
Molecular weight	60.1
Specific gravity (at 20 °C)	0.785
Boiling point (°C)	82.4
Flash point (°C)	12
Evaporation rate (n-butyl acetate = 1)	2.88

3.1.7.2. Dispersing and wetting agent

EDAPLAN 482 produced by Münzing Chemie, Germany, was used as a wetting and dispersing agent for aqueous coatings and pigment concentrates. It is a solution of acrylic polymer. Its physical and chemical specification is shown in the following Table (3.14):

Table (3.14): Specifications of EDAPLAN 482

Appearance	Liquid
Color	Red-brown
Solid content (%)	85.5
Specific gravity (at 20 ^o C)	1.12
Flash point (^o C)	> 100
Solubility in water	easily dispersible
pH (2% in D.I water)	Approx. 9

3.1.7.3. Anti-foaming agent

AGITAN 731 produced by Münzing Chemie, Germany, was used as a highly effective defoamer for aqueous emulsion and water-reducible systems. It is a blend of modified organo polysiloxanes with nonionic alkoxyated compounds. The non-ionic structure provides excellent compatibility with most common binders. Its physical and chemical specification is shown in the following Table (3.15):

Table (3.15): Specifications of AGITAN 731

Appearance	Liquid
Color	Yellowish- turbid
Solid content	100
Specific gravity (at 20 ^o C)	1.01
Flash point (^o C)	> 200
Solubility in water	not emulsifiable
pH (2% in D.I water)	approx. 7

3.1.7.4. Drier

Dapro 7007 produced by Daniel Products Company, USA, was used as a sole drier for urethanated alkyd emulsion formulations. It acts as a chelating catalyst designed to replace cobalt driers. It has a good hydrolysis resistance, which minimizes the initial and post-dry color effects associated with cobalt driers.

3.1.7.5. Biocide

MERGAL K6N was used as a preservative for water-based emulsion paints and has a balanced activity against bacteria, yeast and moulds. It is non-ionic compound and it is very compatible with especially fine particle dispersions. It was delivered from Troy Chemie GmbH, Germany.

3.1.7.6. Flash rust inhibitor

Flash rust inhibitors are usually incorporated in water-borne coatings to prevent flash rusting during the drying of paint over ferrous substrates. In this work, ammonium benzoate ($C_7H_9O_2N$) was used as flash rust inhibitor in water-borne formulations.

3.1.7.7. pH stabilizer

Ethanolamine (C_2H_7NO) was used for adjusting pH of water-borne paints under investigations. It was delivered from Fluka Company, Germany.

3.1.7.8. Thickening agent

TAFIGEL PUR 60 produced by Münzing Chemie, Germany, was used as a highly pseudoplastic associative thickener for water-borne coating formulations. It is non-ionic polyurethane in butyltriglycol/water. It builds up and stabilizes viscosity by forming a network between polyurethane, binder molecules and other paint ingredients. So, it provides strong pseudoplastic rheological profile with excellent shear thinning characteristics. Its physical properties can be shown in the following Table (3.16):

Table (3.16): Specifications of TAFIGEL PUR 60

Appearance	Liquid
Colour	White - turbid
Active ingredients (%)	Approx. 40
Specific gravity (at 20 °C)	1.07
Solubility in water	Dispersible by strong mixing
pH (2% in D.I water)	approx. 7

3.1.7.9. Solvents and chemicals

All solvents and chemical reagents were of pure grade.

3.2. TECHNIQUES

3.2.1. Preparation of corrosion inhibitors⁽²⁰⁵⁾

Heterocyclic compounds and epoxidized soybean oil (Ep-So) were mixed in stoichiometrically amounts. The reactions were carried out in double glass wall sealed ampoules under inert atmosphere at high temperature.

3.2.2. Infra-red spectroscopy measurements⁽²⁰⁶⁾

IR spectroscopy of (Ep-So), thiols and their reaction products with the epoxidized material were measured using FTIR spectrometer, Nexus 670, Nicolet, USA, (Resolution 4cm^{-1}), Microanalytical center, , NRC, Dokki, Cairo, Egypt.

3.2.3. Molecular weight measurements⁽¹⁶⁰⁾

Molecular weights of the epoxidized soybean oil and the prepared adducts were determined by gel permeation chromatography (GPC) using Agilent-1100 GPC-Agilent technologies-Germany. The refractive index detector was G-1362A with 100-104-105 Ao Altrastyrigel columns connected in series. Tetrahydrofuran (THF) was used as the eluent with flow rate 1ml min^{-1} . Commercially available linear poly methyl methacrylate and polystyrene standards were used to calibrate the columns. The GPC apparatus was run under the following conditions: Flow rate = 2.000 ml/min , Injection volume = $100.000\ \mu\text{l}$, sample concentration = 1.000 g/l . The values of molecular weights were computed by means of a computer program.

3.2.4. Emulsification of the prepared adducts⁽²⁰⁷⁾

Emulsification of the prepared adducts was carried out using suitable emulsifiers by mean of high shear mixer (homogenizer), which manufactured by Greaves Company, England.

3.2.5. Surface tension measurements⁽²⁰⁸⁾

A Krüss tensiometer K6, employing the ring method, was used in measuring interfacial surface tension of emulsified oil adduct samples. The apparatus consists of a ring connected to a balance beam immersed into a liquid and afterwards it slowly withdrawn. The interfacial tension is expressed by

$$\sigma = k / 4\pi r$$

Where k is the force necessary to withdraw the ring against interfacial tension, and (r) corresponds to the average value of outer and inner ring diameter.

3.2.6. Emulsion stability⁽²⁰⁹⁾

Constant volume of the prepared emulsion was transferred to test tube. The increasing of 10 °C in the temperature of the prepared emulsion is considered to double the rate of most reactions. Therefore, three months at 45 - 50 °C is equivalent to one year at 20 - 25 °C, (also one week at 45 - 50 °C is equivalent to one month at 20 - 25 °C) for many systems. It was recognized that heating changes the solubility balance of the surfactant, facilitates chemical reactions and accelerating the coagulation processes

3.2.7. Preparation of emulsion paint formulations

Generally, emulsion paint formulations are prepared using two common operations:

3.2.7.1. Grinding (mill base) operation⁽²¹⁰⁾

During this stage the pigments and extenders are transformed into dispersion of particles which can reach dimensions as small as the primary particle size.

A ceramic ball mill was used for grinding pigments and extenders mixture. Wetting and dispersing agents were added to the mill solution to improve pigment particles wetting and dispersion characteristics with minimum required work. Other additives were added to the mill solution; include pH stabilizers, anti-foaming agent and co-solvents.

Water is added periodically to maintain adequate consistency of the mill base for good milling operation. In order to obtain the best efficiency, milling was carried out under the following operating conditions:

- Speed of rotation = 55 – 65% of the critical speed of the mill.
- Volume of ceramic balls = 45 – 50 % of the ball mill.
- Minimum Charge of paste = 25 % of mill volume.

Dispersion time varied from batch to another according to the type and size of pigment mixture and the required fineness of grind.

3.2.7.2. Letdown operation ⁽²¹¹⁾

This stage includes the following three steps:

1. Mixing the emulsion resin with formula additives in a correct order.
2. Gradual addition of mill base to resin/additives mixture with continuous stirring.
3. Adjustment of paint viscosity by thickener addition.

3.2.8. Measurement of fineness of grind ⁽²¹²⁾

Fineness of grind of pigments was measured using Hegman-type-gauge, as shown in Fig. (3.2).



Fig. (3.2): Hegman type gauge

The degree of fineness is determined by calibrations of the material with Hegman units and micrometers as shown in Table (3.17).

Table (3.17): Hegman scale

Hegman scale	0	1	2	3	4	5	6	7	8
Depth (µm)	100	90	75	65	50	40	25	15	0

3.2.9. Determination of solid content and volatile content ⁽²¹³⁾

The solid content of dry paint can be determined by weighing an adequate quantity of paint specimen in a crucible, and heated in an aerated oven at $100 \pm 5^\circ\text{C}$ till constant weight.

$$\text{Solid content by weight (\%)} = \frac{W_1}{W_0} \times 100$$

Where W_1 = the final weight.

W_0 = the initial weight.

The volatile percent can be calculated from the loss in weight as follow:

$$\text{Volatile content (\%)} = 100 - \text{solid content}$$

3.2.10. Preparation of steel panel surface ⁽²¹⁴⁾

Steel panels must be treated before use. The following procedure was followed in order to achieve a good surface preparation of steel panels:

1. Removal of all detrimental foreign matter such as oil, grease, dirt, and other contaminants from steel by the use of suitable solvent(s).
2. Removing of all rust scale, mill scale and loose rust by sand blasting with different grades of emery paper in both sides and edges of steel panels.
3. Rubbing the panels with a clean cloth to remove any residual impurities.
4. After rubbing, the panels are allowed to dry at temperature between 50 and 100°C , and coated immediately or stored in a desiccator for future use.

3.2.11. Film application ⁽²¹⁵⁾

The wet paint film was applied on metal or glass panels using film applicator as shown in Fig. (3.3). Recoating was made after at least 48 hours interval to ensure reasonable drying of the first coat.



Fig. (3.3): Different types of film applicators

3.2.12. Measurements of dry paint film thickness ⁽²¹⁶⁾

Ecotest plus was used to measure the dry paint films, as shown in Figure (3.4). The instrument digitally measures the thickness of the dry coat film on steel (except electroplated nickel), and all non-conductive coatings on non-ferrous metals such as aluminum, stainless steel, brass etc. This instrument operates by measuring the parallel magnetic flux. The effect of an applied paint film on the metal is act as an air gap, thus the magnetic-flux is proportion to the thickness of the paint film. In the present work the film thickness was in the range of 100-120 μm .



Fig.(3.4): Ecotest plus instrument

3.2.13. Measurements of dry paint film hardness ⁽²¹⁷⁾

A measurement of paint film hardness was carried out by using pencil hardness tester according to ASTM D3363, using the following procedure:

A dry coated film panel is placed on a firm horizontal surface. The pencil is held firmly against the paint film at 45° angle and pushed away from the operator in a 6.5 mm stroke. The process is started with the hardest pencil and continued down the scale of hardness to either of two end points as follow:

1. Gouge hardness: the hardest pencil that will leave the film uncut for a stroke length of at least 3mm.
2. Scratch hardness: the hardest pencil that will not rupture or scratch the paint film.

A set of calibrated wood pencils meeting the following scale of hardness was used:

6B- 5B- 4B- 3B- 2B- B- HB- F- H- 2H- 3H- 4H- 5H- 6H

Softer

Harder

3.2.14. Cupping test ⁽²¹⁸⁾

Cupping or ductility test is performed to measure the elasticity and adhesive quality of lacquers, paints and other protective coatings applied to a metal substrate. In this test, Eriechsen cupping instrument was used. The cupping test determines the depth (in-mm) of a hemispherical indenter, which applied under uniform pressure to the uncoated side of the test panel until the coating film tears or flakes. The procedure was performed according to DIN 50101.

3.2.15. Bending test ⁽²¹⁹⁾

Direct desensitization tests of the films on metals require very rugged apparatus. In order to obtain a range of elongation, an elongation test equipment (cylindrical mandrel bending tester) consisting of series of mandrels of different diameters of 25 mm, 19 mm, 12.7 mm, 9.5 mm, 6.4 mm, and 3.2 mm, was used, as shown in Fig.(3.5).

After drying or curing of paint film, the coated panels are bent over cylindrical mandrels of various diameters and the resistance to cracking of the coating is determined.

The resistance to cracking value for a coating is taken as the mandrel diameter at which cracking occurs. In the present work, a mandrel of 9.5 mm diameter was used.

Table (3.18): Elongation (%)

Mandrel diameter, mm	25	19	12.7	9.5	6.4	3.2
Elongation (%)	3.3	4.4	6.75	9	14	28
Correction factor	0.21	0.26	0.38	0.50	0.71	1.40

The total elongation of the coating can be calculated as follows:

$$E = e_1 + tc_1$$

Where: E total elongation,
 e_1 elongation (%) from the Table,
 t thickness of the panel (mm),
 c_1 correction factor

Actually, the observed elongation is greater than that of the theoretical values and it varies from metal to metal.

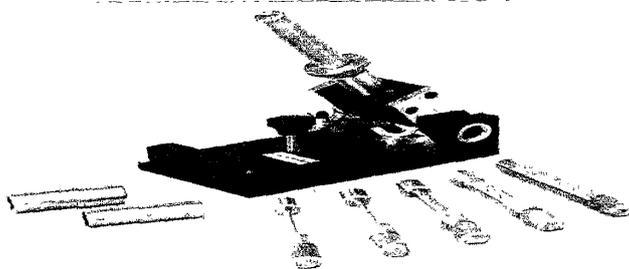


Fig.(3.5): Cylindrical mandrel bending tester

3.2.16. Adhesion test ⁽²²⁰⁾

The used apparatus consisting of: cutting tool with 11 blades in cutting distance of 1 mm tap, magnifying glass, and a brush; it was manufactured by Sheen Company. The cross-cut test procedure according to DIN53151 includes the following steps:

1. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate.
2. A sectional strip with 11 cuts to which a further sectional strip is added perpendicularly resulting in a grid of 100 squares (cross-cut).
3. Brush the area and inspect the incisions for reflection of light from the substrate. If the metal has not been reached, make another grid in a different location.
4. Place the center of the tape over the grid and in the area of the smooth into place by a finger. To ensure good contact with the film, rub the tape firmly with the eraser at the end of a pencil.
5. Within 90 ± 30 s of application, remove the tape by seizing the free end and rapidly (not jerked) back upon it self at as close to an angle of 180° as possible.
6. Inspect the grid area for removal of coating from the substrate.
7. Rate the adhesion in accordance with the following Table:

Table (3.19): The evaluation of adhesion test

Nominal value	Cross-cut	Description
Gt0		The edges of cuts are completely smooth; none of squares of the lattice is detached.
Gt1		Small flakes of the coating are detached at intersections; less than 5% of the area is affected.
Gt2		Small flakes of the coating are detached at intersections; less than 15% of the area is affected.
Gt3		The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
Gt4		The coating has flaked along the edges of cuts in large ribbons and the whole squares have detached. The area affected is 35 to 65 % of the lattice.

3.2.17. Preparation of artificial sea water ⁽²²¹⁾

A representative sample was prepared by dissolving the following quantities of chemicals in one liter of distilled water, as shown in Table (3.20):

Table (3.20): Chemical composition of artificial sea water

Component	Weight, (gm)
Sodium chloride	27.26
Magnesium chloride	3.51
Magnesium sulfate	1.84
Potassium chloride	0.69
Sodium bicarbonate	0.11
Potassium bromide	0.09
Calcium sulfate	1.29

3.2.18. Corrosion tests

3.2.18.1. Corrosion scratch test ⁽²²²⁾

Steel panels were used in the corrosion scratch test. Each panel was covered with coat on its both sides. The panels were left to dry for 7 days. They were edged with wax to prevent the attack of saline solution from the edges, and one face of each panel was scratched with a sharp blade to obtain X-cut through

the coating under test. The scratch should be of a sufficient length to cover the significant test area until expose the bare metal, but it should not contact the edges of the specimen. The panels were then exposed to artificial sea water up to 28 days. At the end of this test, they were washed with distilled water and dried. The corrosion creepage or coating delamination at the scribe can be assessed, according to the following rating:

Table (3.21): Rating of failure at the scribe

Creepage of corrosion from scribe (Millimeters)	Rating number
Zero	10
Over 0 to 0.5	9
Over 0.5 to 1.0	8
Over 1.0 to 2.0	7
Over 2.0 to 3.0	6
Over 3.0 to 5.0	5
Over 5.0 to 7.0	4
Over 7.0 to 10.0	3
Over 10.0 to 13.0	2
Over 13.0 to 16.0	1
Over 16.0 to more	0

3.2.18.2. Blistering resistance test ⁽²²³⁾

It is the evaluation degree of blistering of paint or the ability of coating to resist the formation of dome-shaped, liquid or gas-filled projection resulting from local loss of adhesion and lifting of the film from the previously applied coating of the substrate.

Photographic reference standards were used to evaluate the degree of blistering after immersion in artificial sea water that may develop when paint systems are subjected to conditions which cause blistering. Actually, blisters evaluation can be done according to two standards, size reference standards and frequency reference standards.

Size reference standards have been selected for four steps as to size on a numerical scale from 10 to 0. Number 10 represents no blistering. Blistering standard number 8 represents the smallest size blister easily seen by the unaided eye, while blistering standard numbers 6, 4, and 2 represent progressively larger sizes. Frequency reference standards have been selected in steps of four blister frequencies rated as follows: Dense (D), Medium dense (MD), Medium (M), and Few (F).

3.2.18.3. Barrier resistance test ⁽²²⁴⁾

The surfaces of paint samples were visually compared with the photographic reference standards to determine the percentage of the rusted area. The numerical rust grade scale is an exponential function of the area of rust. So, the slight amount of first rusting has the greatest effect on lowering the rust grade. The apparent corrosion or degree of rusting can be assessed according to the following scale:

Table (3.22): Scale and description of rust grade

Rust grades	Description
10	Less than or equal to 0.01 percent
9	Greater than 0.01 percent and up to 0.03 percent
8	Greater than 0.03 percent and up to 0.1 percent
7	Greater than 0.1 percent and up to 0.3 percent
6	Greater than 0.3 percent and up to 1.0 percent
5	Greater than 1.0 percent and up to 3.0 percent
4	Greater than 3.0 percent and up to 10.0 percent
3	Greater than 10.0 percent and up to 16.0 percent
2	Greater than 16.0 percent and up to 33.0 percent
1	Greater than 33.0 percent and up to 50.0 percent
0	Greater than 50 percent

3.2.19. Alkali resistance test ⁽²²⁵⁾

The coated glass panels of the dimensions 15 x 15 cm were immersed to half-length in an aqueous solution containing 5 gm of anhydrous sodium carbonate or sodium hydroxide per 100 ml of distilled water for 24 hours. The panels were removed, washed thoroughly and the immersed portions of the films were examined after drying for 2 hours. The immersed parts of the film were examined for any defects and any changes in hardness or color. In the present work, the test was extended to one week.

3.2.20. Acid resistance test

The coated glass panels of the dimensions 15 x 15 cm were immersed to half-length in an aqueous solution containing 20 gm of sulfuric acid per 100 ml of distilled water, and were allowed to stand for 24 hours, at room temperature. The panels were then removed, washed thoroughly with cold distilled water and then allowed to dry in a vertical position for two hours, at room temperature. The immersed parts of the films were examined for any defects and any change in hardness or color. In the present work, the test was extended to one week.

3.2.21. Determination of water up-take ⁽²²⁶⁾

3 x 3 cm painted, unwaxed thin plastic panels are used for paint application. The panels are removed from the water at intervals of 2, 4, 9, 14, 21 and 29 days. At the end of each immersion period, the panels are shaken to remove excess water, quickly placed in the center of a pack of filter paper and gently pressed; this is repeated with a fresh pack. The panels is then quickly transferred to a balance and weighed. It is then suspended in a dessicator to dry up completely and reweigh till constant weight. The percentage of water up-take is then plotted against time.

In the present work, the panels were removed from distilled water at intervals of 5, 10, 15, 20, 25 and 30 days.

3.2.22. Weight loss measurements ⁽²²⁷⁾

Steel panels of area 3 x 3 cm were used. Each panel has a hole at its upper part for hanging in the reaction chamber. The panels were well prepared, then weighed. The area of the steel panel was also calculated before coating. The coated panels were immersed in a chamber containing artificial sea water. The specimens were successively removed after intervals of 10 days. The panels were reweighed after removing the paint film and corrosion products on the surface of the metal. The weight loss values were calculated in mg/cm². In the present work, the panels were removed from artificial sea water at intervals of 10, 20, and 30 up to 60 days.