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Preparation and characterisation of acrylic resin for electrodeposited mono-coat coatings

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ABSTRACT

Acrylic resins are known for their multifarious characteristics. Diversified properties of acrylic resins have been utilised for the development of acrylic cationic electrodeposition multicolour mono-coat corrosion protective coatings by acrylic graft polymer of methyl acrylate, butyl acrylate, styrene, and methyl methacrylate in the presence of hydroxy propyl acrylate at 113-117°C in the presence of AIBN (Azo-bis isobutyronitrile) as the initiator. The concept was to develop acrylic graft polymer wherein chemical bonds are protected against UV radiation 400 h or 18-month outdoor exposure. Developed acrylic resin has special characteristic e.g. excellent metal protection against corrosion 500 h salt spray test as per ASTM B117, excellent durability, economical primer, and finish coat in single application and meeting the International and National legislation. Resulting polymers were characterised by using FTIR spectroscopy, particle size analyser and other Morden instrumental techniques. Thus the obtained polymers have high solid content $(72 \pm 1\%)$ and can be used in electrodeposition emulsion paint as a binder. The coatings are monocoat prepared and their anticorrosion behaviour is discussed in this paper. The experimental results depict that the resin thus synthesised has high anticorrosive property, UV light stability, and excellent weather resistance.

KEYWORDS

Acrylic resins; emulsion paint; corrosion protective coatings; mono-coat

Nomenclature

- AIBN Azo-bis iso-butyronitrile
- DTBP Di tertiary butyl peroxide
- UV Ultra Violet
- VOC Volatile Organic Compound
- FTIR Fourier Transformed Infra-Red Spectroscopy
- TBC Tertiary Butyl Catechol
- MMA Methyl Methacrylate
- BA Butyl Acrylate
- MIBK Methyl Iso Butyl Ketone
- MEK Methyl Ethyl Ketone
- ASTM American Society for Testing and Materials

1. Introduction

Acrylic resin has been used in almost all auto industry in primer paints and topcoat paints [1]. The synthesis and application of electro-depositable acrylic resin is a breakthrough in the paint industry as mono-coat coatings. Acrylic-based electro-depositable mono-coat is newer technology which is

used as a primer and a topcoat in two wheeler industries [2]. The prime techniques for polymer modifications are grafting, crosslinking, blending, and composite formation, which are all multicomponent polymer systems. Such materials have attracted considerable attention in the industrial field as they combine with a variety of functional components in a single material [3]. Micro- and macro-phase separation directly affect the physical and chemical properties in such multi-component polymers. Graft copolymers are branched macromolecules in which the branches are of different types from the polymer backbone. They have a variety of potential applications resulting from the wide range of properties available when different polymer chains are connected to form hybrid branched macromolecules [3,4].

Grafting technique is in use nowadays. This technique provides the advantages of each component, used for grafting, contributing its individual benefits to overall system [5,6]. The present invention relates to graft copolymers and compositions suitable for cathodic electro-deposition of polymeric coatings. Furthermore, it relates to a method of cathodic electro-deposition of such graft copolymers [7]. Grafting materials are well known to combine the balance properties of polymers. In addition, coatings provide a wide range of compatibility with other resins, because the grafted component of the precursors can easily deposit with metal substrates by the surface hydroxylation, the adhesion properties of coatings are critical for the durability of coating and corrosion protection [1-3,8]

In the present scenario, low volatile organic compound (VOC) coatings have become the main concern of the surface coating industries. Thus, from the industrial point of view, water-based sol-gel predecessors have various advantages over the corresponding non-aqueous medium system, including significant VOC reduction and ease of control. Therefore, our prime objective is to introduce cross-linking resins for coating systems to enhance the mechanical and chemical resistance properties of the films and to obtain highly dense coatings on metal surfaces to improve the corrosion resistance capability [7,9,10]. The acrylic group improves other properties of coatings also. We used these Acrylic graft polymers wherein chemical bonds are protected to sustain UV radiation. In this context, our research work describes the preparation of the acrylic graft polymer resins for corrosion-protective coatings using different monomer contents. The anti-corrosion behaviour of the prepared coatings was studied [7–11].

2. Experimental

2.1. Materials

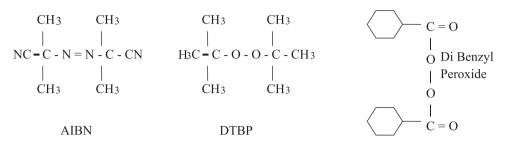
All the monomers, Methyl Methacrylate (MMA), Butyl Acrylate (BA), Hydroxy propyl acrylate, 2 Hydroxy Ethyl Methacrylate, Styrene, and Glacidyl Methacrylate, were procured from Aldrich & Accord Chemical Corporation and freed from inhibitor TBC (Tertiary Butyl Catechol) The polymerisation during storage is prevented by close care to monomer temperature, inhibitor level, polymer content, and oxygen content. Determinations of inhibitor content, oxygen level in the vapour space, polymer content, and monomer temperature are checked on a routine basis. Styrene-containing vessels are protected from the external sources of heat and stored at <25°C to avoid thermal polymerisation. AIBN (Azo-Bis ISO-Butyronitrile) is used as a polymerisation catalyst [2,4].

Acrylic resins are the homo-polymers or copolymers based on acrylic acid and their esters. However, polymers based on styrene are also classified as acrylic resins. Thus acrylic resins are structurally macromolecules of saturated C–C chain obtained by the addition of individual monomer units across the vinyl group. The experimental method, used for synthesis of acrylic resins, is explained here [2,4]

2.2. Raw materials

2.2.1. Initiators

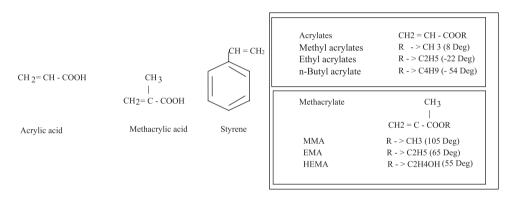
Initiators are used to initiate free radical reaction, e.g. Azo-Bis Iso-Butyronitrile (AIBN), di tertiary butyl peroxide (DTBP), and Di benzyl peroxide.



2.2.2. Monomers

The acrylic monomers and vinyl monomers used are depicted below.

Examples are as follows:



In order to avoid the premature polymerisation of monomers during transport or storage, the monomers are inhibited by adding a suitable inhibitor [12,13].

2.2.3. Solvents

- (1) Acrylic ester polymers with a short side chain are polar and require polar solvents like ester, ketone, ethers, or alcohol.
- (2) As the side chain becomes longer, the polymer becomes less polar and more non-polar, so it can be dissolved in non-polar solvents like aliphatic hydrocarbon.

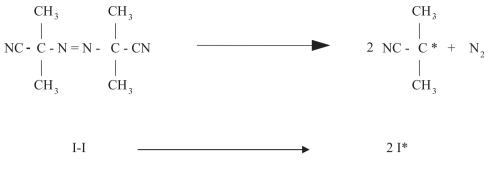
The choice of solvents also depends upon their evaporation rate.

2.3. Mechanism of acrylic resins

Most widely used experimental and commercial process in preparing acrylic copolymers is the free radical polymerisation [5,10,14], which involves three stages.

2.3.1. Initiation

Initiator splits to provide the free radical.



2.3.2. Propagation

Initiator free radical attacks the monomer molecule to propagate the reaction.

 $I + CH_2 = CH-R$ \blacksquare I-CH₂-*CH-R

I-CH₂-*CH-R +CH₂=CH-R \rightarrow I-(CH₂-CH-R) n-(CH₂-*CH-R)

2.3.3. Termination

This takes place when reactive sites on the polymer chain have rendered inactive.

 $2 (I-CH_2-CH-R) n -(CH_2-*CH-R) \longrightarrow I-(CH_2-CH) n-CH_2-CH-(CH_2-CH) n-I$ $| \qquad | \qquad | \qquad | \qquad |$ $R \qquad R \qquad R \qquad R$

Free radical polymerisation of acrylic monomer may be carried out in any of four basic polymerisation systems [2,3,15].

Bulk	- Absence of any solvent.
Solution	- Polymerisation carried out in the presence of a solvent.
Suspension	- Polymerisation carried out in the presence of a non-solvent.
Emulsion	- Polymerisation carried out in the presence of water [2,9].

2.4. Controlling of molecular weight during process

2.4.1. Rate of addition

The degree of polymerisation depends upon the rate of addition. A faster addition rate will tend to increase the monomer concentration resulting in high molecular weight resin. On the other hand, a slower addition rate will give low molecular weight resin.

2.4.2. Initiator concentration

As initiator concentration increases, molecular weight decreases.

2.4.3. Type of initiator

AIBN gives a lower molecular weight than DTBP.

2.4.4. Processing solids

High processing solid polymerisation reduces chain transfer activity resulting in higher molecular weight system. Viscosity or molecular weight is proportional to monomer concentration and inversely proportional to initiator concentration [9,10].

2.5. Precautions to be taken during processing of acrylic resins

2.5.1. Temperature

Since the molecular weight is highly sensitive to temperature, proper temperature maintenance is necessary due to an increase in molecular weight.

2.5.2. Proper weighing of monomers and initiator

Since monomer concentration and initiator concentration play an important role in determining the molecular weight of resin, the weighing should be perfect.

2.5.3. Use of metering pump

Molecular weight also depends upon monomer concentration or processing solids. A faster addition rate will tend to increase the monomer concentration resulting in high molecular weight resin. On the other hand, a slower addition rate will give low molecular weight resin. Hence, controlled rate of addition using a metering pump is necessary to have a desired molecular weight and viscosity.

2.5.4. Storage

Storage of monomers, initiators, intermediates need to be adhered strictly to the storage condition [10].

2.6. Preparation method of acrylic resin

Processing of the acrylic resins was carried out in a four-necked flask equipped with a mechanical stirrer, a water condenser, a thermometer pocket, and a dropping funnel. The butyl cellosolve and MIBK were taken in the flask and the purged contents were heated to $115 \pm 2^{\circ}$ C.

The selected amounts of monomers (methyl methacrylate, butyl acrylate, 2 hydroxy ethyl methacrylate) were charged to the flask. The initiator AIBN was added to the charge under stirring and stirred further for 15 min to dissolve it. The monomer mixture was added dropwise to the flask over the period of 3 h at temp $115 \pm 2^{\circ}$ C and the exothermic reaction at temp $115 \pm 2^{\circ}$ C for 1 h was controlled.

The AIBN was added in 4 equal parts at 15 min interval and held at a temperature of $115 \pm 2^{\circ}$ C for 1 h. The flask was cooled to 65°C and the diethyl amine was added. The content was heated to 100°C in about 90 min and the contents were held at 100°C for 1 h. The temperature was then raised to 120°C in about 1 h till the desired viscosity and epoxy value are obtained (about 120 min).

The optimum formulation and processing details after various batches are given in Table 1. The processing temperature was $115 \pm 2^{\circ}$ C and the monomer/initiator addition time was 180 min [9,10,14].

Average number molecular weight of prepared acrylic resin is 4500 g/mol (Table 2).

2.7. Preparation of emulsion

2.7.1. Neutralisation process

The developed acrylic resin, epoxy resin, poly urethane oxime resins, and catalysts in 5 L vessel were charged and stirred for 15 min then PU curing agent, dispersing agent, Emulsifier, Glycolic Acid, and Acetic Acid (Glacial) were added under agitation for 30 min for complete neutralisation. Finally, deionised water was added. This is known as pre-emulsion.

2.7.2. Emulsification

23% deionised water and 77% pre-emulsion were taken in a stainless steel vessel. This mass was stirred with a high speed stirrer for 30 min. This forms the emulsion. During emulsion, temperature is maintained at $20 \pm 2^{\circ}$ C [6,11,16].

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Table 1. Formulation of acrylic resin.

Preparation of acrylic resin for electrodeposition					
Compound	Functional	Quantity by weight (%)	Quantity by weight in (g)		
Butyl cellusolve	Solvent	6	180		
Methyl iso butyl ketone (MIBK)	Solvent	16	480		
Methyl methacrylate Monomer	Monomer	26	780		
Butyl Acrylate Monomer	Monomer	17.5	525		
2 Hydroxy Ethyl Methacrylate	Monomer	22	660		
AIBN (Azo-bis iso- butyronitrile)	Catalyst	5	150		
Methyl iso butyl ketone MIBK	Solvent	5.0	150		
Diethyl amine	Hardener	2.5	75		
Total		100	3000		

Table 2. Physical properties of acrylic resin.

Parameter	Calculated value	Experimental value
Non-volatile (%) by weight	72	72.8
Appearance (visual)	Clear	Clear
Amine value (mg KOH/g)	26	25.1
Viscosity by Gardner bubble tube	J	К

2.8. Preparation of pigment paste

A 2 L stainless steel vessel was taken. The deionised water, acetic acid (glacial), epoxy resin, thickening agent, wetting agent were added to the vessel and mixed homogeneously then Bismuth Hydroxide pigment powder was added under stirring and the mass was mixed for 30 min and the condition of the paste was checked (no agglomerates visually).

The above slurry material was ground and the grinding fineness was checked (up to 5 micron). Then the material with deionised water was flushed and the viscosity/solid contents of pigment paste were adjusted [5,6].

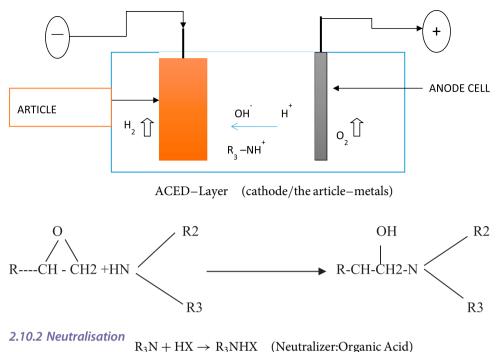
2.9. Cathodic electrodeposition coating formulation and film preparation

A homogenous suspension bath for electrodeposition emulsion 35%, pigment paste 2.5%, and deionised water 61.5% was prepared as per coating film formulation.

Acrylic cathodic electrodeposition paint is applied in the pre-treated steel panel through the electrodeposition process in which panels are kept in cathode position and the bare panel in anode position. After applying the voltage from the range of 125 V to 250 V for 1 min to 3 min. 15–20 min flushed panel is baked in 160°C oven for 20 min. Film thickness can be controlled by voltage and/or application time for maintaining a uniform film thickness of 25–28 micron for the characterisation of general chemical resistance and mechanical properties. The cured films were stored for three days under ambient atmospheric conditions before testing [17–19].

2.10. Electrodeposition chemistry

Graft copolymer is useful in electrodeposition. For example, A polyamine/epoxy ester graft copolymer, having an acrylic polyamine backbone with secondary amine and hydroxy functionality (part of which is terminal primary hydroxy groups), onto which an epoxy ester is grafted, has been found suitable in eclectrodeposition. The ester is an epoxy resin reacted with a fatty acid resulting in a mono-epoxide ester with one or less epoxy group per acid group. The ester is reacted with part of the secondary amine on the acrylic backbone. This system can be formulated to a nearly neutral pH cathodic dispersion with a good cure response at 160°C using conventional aminoplast cross-linkers.



 $R_3N + HX \rightarrow R_3NHX$ (Neutralizer:Organic A

2.10.3. Ionisation At cathode (Substrate)

 $2H2O + 2\overline{e} \rightarrow 2OH^- + H_2 \uparrow electrolysis of water$

 $R_3NH^+ + OH^- \rightarrow R_3N + H2O$

 R_3NH^+ (water-soluble) positively charged resin gets deposited on the substrate as R_3N (water insoluble deposited) after electrical discharging at cathode.

At Anode (electrode)

 $2H2O \rightarrow 4H^+ + 4\overline{e} + O2$ electrolysis of water

At anode, neutralisation of counter ions by H⁺ ions to generate the acid

 $X^- + H^+ \rightarrow HX$

drain the electrolyte to maintain pH [20].

3. Characterisation of acrylic resins

The acrylic resins, prepared as above, were characterised for corrosion resistance in salt spray test (Table 3) and humidity test (Table 4). The coated panels were also evaluated for their chemical resistance properties like water resistance (Table 5), acid resistance (Table 6), alkali resistance (Table 7), and mechanical properties like adhesion and gloss (Table 8).

3.1. Corrosion resistance properties

The parameters for salt spray test {5% (w/w) solution of NaCl at a temperature range of 33.3–36.1°C as per ASTM B 117} and for the humidity test (temperature cycle of 42–48–42°C of 1 h each at a relative humidity of 100%) are given in Table 3 and Table 4, respectively.

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Table 3. Salt spray test – ASTM-B117.

Parameter	Specification	Polymer	Results
Salt spray resistance ASTM -B117	Cross cut and exposed for 500 h $-$ creep width 2.0 mm max on either side of scribe line and blister size maximum 8F	Acrylic Grafting Acrylic	2.5 mm/6D 1.5 mm/6D

Table 4. Humidity test.

Parameter	Specification	Polymer	Results
Humidity	Humidity cabinet – Temp.: 50 \pm 2°C, RH \geq 95% for 500 h	Acrylic Grafting Acrylic	Inferior Passed

Table 5. Water resistance of paint films.

Sample	Polymer	Deionised water	Sea water
Acrylic cathodic electrodeposited panel	Acrylic	α	β
	Grafting Acrylic	α	а

When dipped for 120 h, α = unaffected, β = slightly affected, γ = affected.

Table 6. Acid resistance of paint films.

		Acetic acid		Hydrochloric acid	
Sample	Polymer	5% (v/v)	15% (v/v)	5% (v/v)	10% (v/v)
Acrylic cathodic electrodeposited panel	Acrylic Grafting Acrylic	E E	G E	E E	G E

When dipped for 24 h, E = Excellent, G = Good, P = Poor.

Table 7. Alkali resistance of paint films.

		Sodium hydroxide		Ammonium hydroxide	
Sample	Polymer	5%(w/v)	10%(w/v)	10% (v/v)	20% (v/v)
Acrylic cathodic electrodeposited panel	Acrylic	E	G	E	G
· · ·	Grafting Acrylic	E	E	E	E

When dipped for 24 h, E = Excellent, G = Good, P = Poor.

Table 8. Mechanical properties of paint films.

SI. No.	Parameter	Specification	Polymer	Results
1	Hardness (Scratch)	1800 g	Acrylic	Inferior
			Grafting Acrylic	Passes
2	Adhesion (no. of squares retained)- Mild steel	0/100 No lifting of film	Acrylic	Passes 0/100
			Grafting Acrylic	Passes 0/100
3	Flexibility		Acrylic	1/8" passes
			Grafting Acrylic	1/8" passes
4	Impact resistance Direct (DuPont Impact	min 50 cm	Acrylic	passes 50 cm
	Tester 500 g 1/2" dia)		Grafting Acrylic	Passes 50 cm
5	Gloss at 60°	85 unit	Acrylic	89
			Grafting Acrylic	87

3.2. Chemical resistance properties

The chemical resistance tests for water, acid, and alkali were performed on coating systems prepared as mentioned earlier. All panels were cured at 160°C temperature for 20 min before subjected to test. The coated panels were sealed from three sides and then immersed in water, acids, and alkalis separately (Tables 5–7) [2,4].

3.3. Mechanical properties

3.3.1. Hardness

This property deals with the resistance of a material to indentation of scratching. The hardness of a coating material is generally tested by scratch hardness test which is done by scratch hardness tester (ASTM D 5178, Sheen Instruments Limited England). The panels were loaded with different weights until a clear scratch, showing the bare metal surface, was seen.

3.3.2. Adhesion

The percentage adhesion was determined by using a crosshatch adhesion tester (ASTM D 3359, Sheen Instruments Limited, England).

3.3.3. Flexibility

The flexibility of the coated film was determined by bending the panels to 180° using a ¼ inch mandrel (ASTM D522, Sheen Instruments Limited England).

3.3.4. Gloss

The gloss of the films coated on the panels was determined by a Multi-angle Specular Gloss meter (ASTM D523, Sheen Instruments Limited England).

3.3.5. Gloss retention

Gloss retention properties of the coating prepared were examined by a QUV wetherometer. The paint films were exposed to alternate cycles of UVA-340 nm radiations for 4 h at 50°C and condensation for 4 h at 45°C with an irradiance of 0.67 W/m² using a UVB 313 lamp, followed by condensation at 50 deg cycle for 700 h test. The cycles were repeated and the coating was observed for gloss retention. The results of gloss retention tests are shown in Figure 1 [2,4].

4. Results and discussion

4.1. Spectral analysis

The polymer characterisation was done by the IR analysis. IR spectrum was used to confirm the reactive groups in monomers and to confirm that all groups have reacted. Theoretically, all monomers should react and final polymer should not have any unreacted monomer group present. Acrylic monomers with functional groups that can be used to functionalise acrylic polymers to provide properties, such as crosslinking, self-crosslinking, improved adhesion or pigment wetting. The peaks at 3560, 2959, 1734, 1440, 1146, and 720 cm⁻¹ resulted from the O–H, C–H, C=O, C–C, C–O, and C=C stretching vibrations, respectively. The acrylic resins are functionalised with hydroxyl function which was represented as peaks at 3560 cm⁻¹ to provide grafting properties.

The IR spectra of acrylic polymer are shown in Figure 2.

4.2. Particle size analysis

From particle size graphs, Figure 3, we can see the particle size distribution of acrylic polymer emulsion. This acrylic emulsion particle size distribution has a narrow range (less than 0.30 micron). The finer particles of monomers provide better film integrity and packing, resulting in better chemical and physical properties [2,21,22].

4.3. Corrosion resistance properties of grafting films

From the results, given in Table 3 and Table 4, it can be seen that incorporation of grafting monomer in the backbone of acrylic resins has helped to develop a coating with improved salt spray resistance and humidity. The resistance properties of the prepared coating increase with the increase of the grafting agent content in resin [19,23–25].

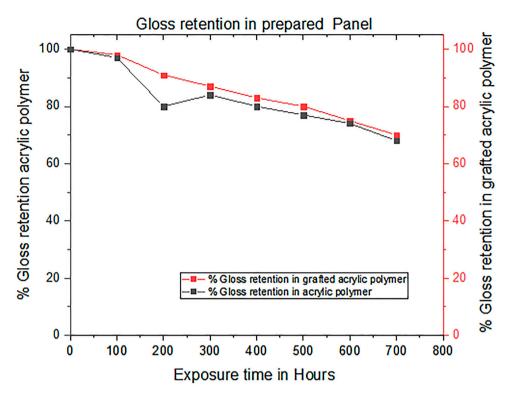


Figure 1. Gloss retention performance of prepared samples.

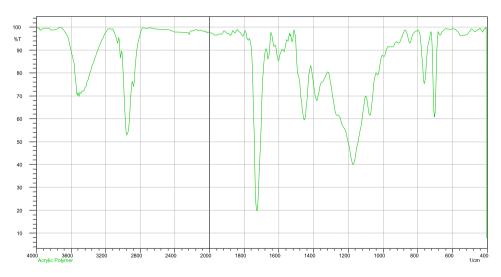


Figure 2. Infra-red spectrum of acrylic polymer.

4.3.1. Salt spray test

Test at $35\pm1^{\circ}$ C with 5% NaCl in distilled water, panels should be placed at 15–30 deg. angle to vertical and fog collection 1-2 ml/h. Tested sample has to be cleaned, dried, and checked within 1/2 h. Use paint film remover like adhesive tape to evaluate as per ASTM B117 and ASTM D1654 after

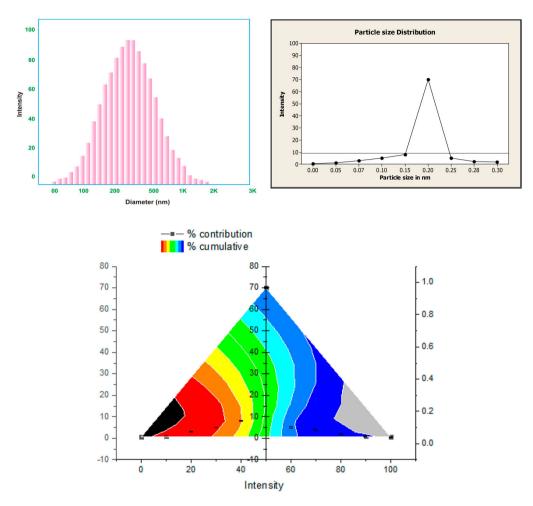


Figure 3. Particle size distribution graph.

stipulated 500 h of test creepage along X mark to be max. 2 mm on either side. No rust, no blister and no adhesion failure beyond 2 mm from X mark.



Grafted acylic system Cross cut (X) panel before



Grafted acrylic system Salt spray test



Non grafted acrylic system 500 h exposed panels after salt spray test

The salt spray test of the paint films is shown in Table 3 and panels. It was found that corrosion resistance of the film in grafted polymer is comparatively better and within specification than non-grafted polymer.

4.3.2. Humidity test

The humidity test was carried out in humidity cabinet maintained at 98–100% relative humidity at a temperature cycle of 42–48–42°C in 1 h. The distilled water was taken to maintain humidity and samples were examined as per IS 101 (Part 6 /Sec.1)1988.

No rust, no blister, and no adhesion failure was found after 500 h in grafted polymer film, but inferior adhesion results were observed in non-grafted polymer.

4.3.3. Water resistance

The resistance of the paint films against deionised water and sea water is shown in Table 5. It was found that the water (deionised and sea) resistance of the film in grafted polymer is higher than that in non-grafted polymer.

By dipping half portion of the panel in a water bath of temperature 40–50°C for 120 h the panels were observed per given table and good results were found in grafted polymer film against non-grafted polymer film.

4.3.4. Acid resistance

The acid resistance of films is shown in Table 6. The acid resistance of the film in grafted polymer is comparably better than that in non-grafted polymer.

4.3.5. Alkali resistance

Resistance to alkali of films is shown in Table 7. Alkali resistance of all the films, pure acrylic to grafted acrylic were found satisfactory.

4.4. Mechanical properties of paint films

The mechanical properties are given in Table 8. It can be seen by the data given in table that the hardness of the film has passed in grafting polymer compared to acrylic polymer, which shows the impact of grafting polymer on the hardness property. Data from the adhesion test show that both polymers pass the adhesion test. The flexibility test data show a comparable result. Impact resistance is showing considerable improvement in performance in grafting polymer. Gloss value is slightly inferior in grafting polymer. Thus, it is seen that grafting performs better in terms of Hardness and Impact resistance. But, adhesion and flexibility on the system remains unaltered [2,4,26]

Gloss retention: Gloss retention results analyse 80% gloss retention observed after 500 h exposure of grafted acrylic polymer coating against 400 h exposure of non-grafted acrylic polymer coating. Hence the UV resistance of grafted acrylic polymer is better than that of non-grafted acrylic polymer.

5. Conclusion

Acrylic polymer and acrylic grafting polymer are synthesised in aqueous media by emulsion polymerisation, leading to lower VOC than conventional systems [7]. It is a challenge to develop the multicolour shade (Red, Blue, etc.) in electrodeposition mono-coat coatings. Currently electrodepositable coatings are available in grey and black shades. Acrylic cationic electrodeposition coating has a good efficiency in application with different colour compared to brush coating or a spray coating, excellent adhesion and a high corrosion-resistance and can minimise an environmental contamination because of employing water as a solvent. Its application is as primer coat as well as top coat for two wheelers, household electric appliances, and industrial machines. From the performance data, it is seen that an increase in the extent of grafting improves corrosion resistance and chemical resistance properties of the coating [24,27] and other mechanical properties were improved by acrylic polymer. It proves that in a grafting system we can improve the properties of the system by adding grafting agent compared to acrylic polymer. So these resins can be used in coatings where good corrosion resistance, adhesion, flexibility, gloss, and resistance to various chemicals required. The performance can further be improved by increasing the extent of grafting in acrylic resin.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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