Vegetable Oil-Based Highly Branched Polyester Modified Epoxy Based Low VOC High Solid Industrial Paint

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ABSTRACT: *Mesua ferrea* L. seed oil based highly branched polyester modified epoxy resin has been utilized as a binder system for a low volatile organic compound (VOC) containing high solid paint formulation. The highly branched low molecular weight polyester resin acts as the reactive diluent of the epoxy resin which satisfies to reduce VOC emission and produces high solid paint system. The prepared *Mesua ferrea* L. seed oil based highly branched polyester modified epoxy paint (test paint) was assessed against commercially available epoxy paint (standard paint) for protective coating application. Different physical and performance properties of the paint viz. nonvolatile content, specific gravity, viscosity, drying time, scratch hardness, gloss, impact resistance, and

INTRODUCTION

Maintaining of low level of atmospheric pollution, and the stringent environmental rules and regulations restrict the use of volatile organic compounds (VOC) in paint systems.^{1,2} These factors have been driving the coating industry to look for and accelerate research work for developing eco-friendly coating formulations. So, the development of products that can be processed with reduced amounts of organic solvent(s), or even without solvent, is a great challenge.³ Epoxy resins are viscous, tacky materials which are difficult to handle and thereby need to dissolve them in organic solvent(s). These volatile solvents evaporate into the atmosphere as VOC, which is against the regulations for such applications. This is a greatest threat important problem with epoxy resins as binders for paint along with its brittleness character.

Intensive research is being conducted recently on the development of new binder systems for paints, chemical properties such as corrosion resistance, salt spray resistance, UV resistance were measured as per the standard methods. The surface morphology and the thermal stability of the paints were also studied by scanning electron microscope (SEM) and thermogravimetric analysis (TGA). The shear thinning behavior was observed for both the paints as indicated by rheological study. The results indicate that the test paint is comparable with the standard paint. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: *Mesua ferrea* L. seed oil; polyester; epoxy paint; morphology; rheology

which is driven by the need for performance improvements as well as for fulfilling environmental requirements. In this regards different researchers have tried to reduce VOC in the paints through the use of acrylic dispersion, alkyd emulsion or high solid alkyd resin.⁴ The use of reactive diluent is an alternative approach for increasing the solids content of conventional paints in addition to reduce VOC. For number of years high solids commercial paints have been available on the decorative market that meet the requirements for reduced VOC emissions.⁵

Vegetable oil modified polyester resins are the most extensively used binders for different paints including automotive finishing, sewing machines, equipment machines finishes, etc.⁶ Very fans, recently Araujo et al. studied the anticorrosive performance of alkyd paints modified with linseed and soybean oils.⁷ Blustein et al. also studied the improvement of performances of alkyd paints based on medium linseed oil alkyd.8 Although different types of binder systems have been used in paints, but no reports were found in the utilization of vegetable oil based highly branched polyester resin modified epoxy resin as a binder in any paint formulation, even though it has several advantages. In our earlier report we have already shown the satisfactory performance characteristics of vegetable oil based highly branched polyester resin as a surface

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coating material.⁹ Several other reports have also been found on the development of vegetable oil based highly branched polyester resin with desired performance characteristics including flexibility.^{10–12} Coatings made from these highly branched resins have advantages in terms of drying time and their properties fulfill the service demands for decorative paints. In addition to the above, large numbers of functionality, unique structural architecture, low viscosity, and high reactivity of highly branched such polyesters assist in processing and wetting of pigments and other additives and thereby improving performance of the prepared paints. Therefore, the aforementioned polyester resin will be used as reactive diluent for the commercial epoxy resin in the formulations of the epoxy paint.

In the perspective of the above discussion, the authors used Mesua ferrea L. (70% oil content) seed oil as an alternative raw material source in such applications. The potential of this oil based materials in different fields has already been reflected from the reports of the same laboratory.^{13–17} So, the foremost intention of this study is to verify the potentiality of Mesua ferrea L. seed oil based highly branched polyester resin as reactive diluent for a commercial epoxy resin to formulate an easily processable low VOC high solid epoxy paint. Also, the prospective of the Mesua ferrea L. seed oil based highly branched polyester resin in combination with the commercial epoxy resin as binder material needs to be checked. The results of the prepared Mesua ferrea L. seed oil based highly branched polyester modified epoxy paint will be compared with the commercial epoxy paint used for protective coating applications. Therefore, the authors wish to report the suitability of Mesua ferrea L. seed oil modified highly branched polyester resin as a reactive diluent for commercial epoxy resin based easily processable low VOC high solid industrial paint.

EXPERIMENTAL

Materials

Mesua ferrea L. seed oil was collected from its matured seeds (Sivasagar, Assam). Phthalic anhydride (PA), maleic anhydride (MA), 2,2-bis(hydroxymethyl) propionic acid (bisMPA), glycerol (E-Merck, Germany) and lead monoxide (S.D. Fine Chemical, Mumbai) were used without further purification. *Mesua ferrea* L. seed oil modified highly branched polyester resin was prepared as per the reported method.⁹ *N*,*N*-dimethyl formamide (DMF, E-Merck, India) and xylene (E-Merck, India) were used as solvents after purification by distillation. Commercial epoxy resin (CY250) and poly(amido amine) (HY840) (Ciba Geigy, Mumbai) were used as sup-

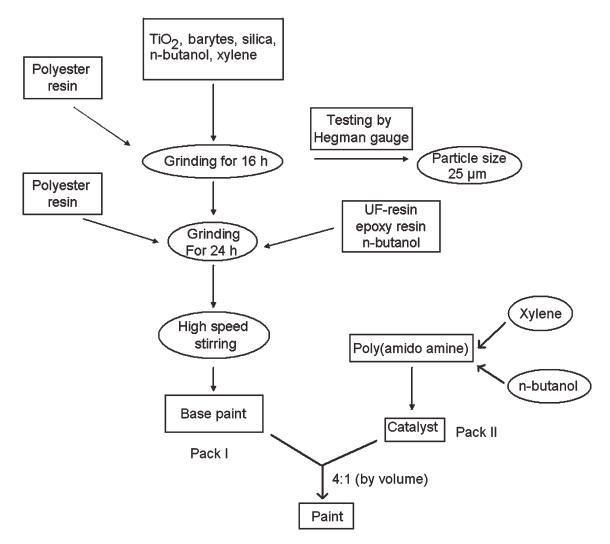
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plied. Commercially available epoxy paint used for protective coating application was used as the standard paint. All other materials related to paint formulations such as titanium dioxide, *n*-butanol, barytes, silica, epoxy resin (70% NVC in xylene), urea-formaldehyde (UF) resin (60% NVC in *n*-butanol) were of commercial grade and obtained from Mumbai, India.

Instruments and methods

Mesua ferrea L. seed oil was extracted from the powdered matured seeds through solvent soaking method using petroleum ether as the solvent. The extracted oil was then purified by alkali refining technique.9 The physical properties such as acid value, viscosity, specific gravity, and nonvolatile matter content of the test and the standard paints were determined by using the standard IS methods.¹⁸⁻²¹ The particle size of the crushed particles was checked by using Hegman Gauge (Sheen Instrument, UK) during the preparation of paint. The morphology of both the test and the standard paints was studied by the JEOL scanning electron microscopy (SEM JSM-6390LV, Germany) after coating the samples on platinum surface. The thermogravimetric analysis (TGA) of both the paints was carried out by using Shimadzu, USA thermal analyzer, TGA 50 under the nitrogen flow rate of 30 mL min⁻¹ and a heating rate of 10°C min⁻¹ from 25 to 700°C. The rheological behavior of both the paints was studied by using CVO100 Rheometer (Malvern, UK). The relationship of viscosity against time was measured at constant stress (200 Pa) under single shear. The variation of shear viscosity with shear rate was carried out with controlled rate, table of shear at 24°C. The coating performance of both the paints was evaluated by determining scratch hardness and gloss by scratch hardness tester, (Sheen Instrument, UK) and digital mini glossmeter at 60°, (Sheen Instrument, UK) respectively. The impact resistance of the paints coated on mild steel panel was measured by using a falling weight impact tester (2 pound weight, SC Dey, Kolkata) as per the standard method.²² The salt spray test, protection against corrosion and UV exposure test for both the paints coated on mild steel panels were performed by using the standard IS methods.²³⁻²⁵

The thin films of both the paints were prepared by drawing a homogeneous mixture of the components on glass plates using a micro adjustable thickness gauge (Sheen Instrument, UK) under ambient conditions. After removal of sufficient amount of solvent under atmospheric conditions, the coated strips were degassed under vacuum at $(45 \pm 5)^{\circ}$ C for 45 min to remove the last trace of solvent and volatile compounds. Then the coated plates were



Scheme 1 Flow sheet diagram for the manufacturing process of the paint.

cured at room temperature for specified time periods. The cured paint films were kept under ambient conditions for 24 h before further studies. The dried films from the glass plates were peeled off by immersing the plates in warm water followed by drying in a desiccator under vacuum and were stored for seven days before taking SEM micrographs. The thickness of the dried coating films was measured by Pentest, coating thickness gauge (model 1117, Sheen Instrument, U.K.) and was found to be in the range of $30-35 \,\mu\text{m}$.

Preparation of polyester resin

The highly branched polyester resin was prepared by the method reported earlier.⁹ Briefly, 0.12 mol (42.09 g) of monoglyceride, 0.064 mol (6.27 g) of MA and 0.096 mol (14.22 g) of PA were reacted together to form a carboxyl terminated prepolymer. Totally, 20 g of this prepolymer was then reacted with 3.32 g (0.016 mol) of bisMPA to obtain the desired highly branched polyester resin. This resin along with commercial epoxy resin was used as the binder for low VOC high solid epoxy paint formulation.

Preparation of paints

The prepared polyester modified epoxy resin-based paint was two-pack paint system, one pack is epoxy resin containing paint usually called base and the other pack is poly(amido amine) called as catalyst. The flow sheet diagram for the manufacturing process of the paint is shown in Scheme 1. The base paint was prepared in porcelain ball mills using epoxy resin as binder (for standard paint) or highly branched polyester resin (for test paint) with prescribed formulation as given in Table I (Pack I). At first, the ingredients like pigment titanium dioxide, extenders, resin, and around 20% of solvent were charged in the ball mill. The grinding of the pigments and extenders was done for around 16 h to make the particle size about 25 µm, as measured by Hegman gauge. After attaining the required grinding size, the materials were dropped into different

TABLE IFormulations for the Test Paint

Ingredients	Amount (wt %)	Function	
(a) Pack I			
TiO ₂	10	Pigment	
Barytes	12	Filler	
Silica	15	Filler	
Test polyester resin	25	Binder/reactive diluent	
Xylene	14	Solvent	
n-butanol	7	Co-solvent	
Urea-formaldehyde resin (60% solid in n-butanol)	1	Flow promoter	
Epoxy resin (70% NVC in xylene)	16	Binder	
(b) Pack II			
Poly(amido amine)	40	Curing agent	
Xylene	52	Solvent	
n-butanol	8	Co-solvent	

containers and mixed with adequate amount of epoxy resin and finally adjusted with the remaining solvents and other additives to achieve required amount of viscosity of paint (30–40 s in FC4 at 30°C). Finally, the prepared paint was kept for 24 h for wetting.

Preparation of test panels

Before application of paint on mild steel panels, the base paint and the catalyst or curing agent (Pack II of Table I) were mixed in the ratio of 4:1 by volume. The prepared paint mixture was diluted to 46% solid content by adding xylene. The paint solution was then sprayed with the help of specific gravity type spray gun from a distance of 15–20 in. on the mild steel plates ($150 \times 100 \times 1.25 \text{ mm}^3$) placed on spray booth. The prepared panels were then kept in open air for complete drying.

RESULTS AND DISCUSSION

Paint preparation

Mesua ferrea L. seed oil modified highly branched polyester resin⁹ was found to possess the optimal performances as a surface coating material. The low viscosity and large surface functionality of the resin helped in processing and wetting of pigment that results improvement of flow level, gloss and wetting of the substrate. So, this resin was utilized as reactive diluent for an epoxy paint to reduce the VOC emissions and to prepare a high solid paint. Along with the reactive diluent, this resin-modified commercial epoxy resin acts as a binder for the production of the high solid low VOC epoxy paint.

The pigment particles exist largely in the form of clusters or aggregates²⁶ in dry condition. So, these

clusters should be broken for proper dispersion of individual pigment particles in the paint medium. The pigment was pulverized and dispersed in the vehicle (binder) along with all other additives to obtain a fine dispersion of the pigment particles in the medium.²⁷ The pulverization process was continued until the degree of dispersion became acceptable for the paint as determined by the Hegman gauge (particle sizes of 25 μ m). Stable dispersion of the dispersed particles was obtained through wetting of the dispersed phase in the binder medium. At last, high-speed stirring was executed to descent the dispersion with additional vehicle to get the final paint composition with optimum level of performance characteristics.²⁸

Physical properties

The different physical properties like viscosity, specific gravity, nonvolatile content, acid value and hydroxyl value of the test resin as well as the paints are given in Table II. The viscosity of the test resin was low which is due to the highly branched globular structure.⁹ The viscosity of the test paint was found to lower than the analogous standard paint. This low value of viscosity helps in the processing, storage, and film formation of the paint. The acid value of the test resin was also low. The specific gravities of both the paints were comparable. The test paint has slightly higher nonvolatile content as compared to the standard paint. From these results it can be anticipated that the test paint can exhibit the requisite physical properties for an industrial paint.

Morphological study

Uniform distribution of the dispersed phases was observed in the SEM micrograph of *Mesua ferrea* L. seed oil-based polyester modified epoxy paint which is comparable to the standard paint (Fig. 1). The nature and composition of the binder system,

TABLE II				
Physical Parameters of the Polyester Resin and Paints				
(Standard and Test)				

Parameters	Polyester	Paint standard	Test
NVC (wt %)	61.20	68.70	72.76
Specific gravity ^a	1.01	1.41	1.42
Viscosity (30°Č, min) ^b	2.5	40	30
Acid value (mg KOH/g, base paint)	10.66	3.23	11.31
Hydroxyl value (mg KOH/g, base paint)	102.3	172.6	186.6

^a Unit less, relative to standard medium.

^b Measured by Ford cup method.

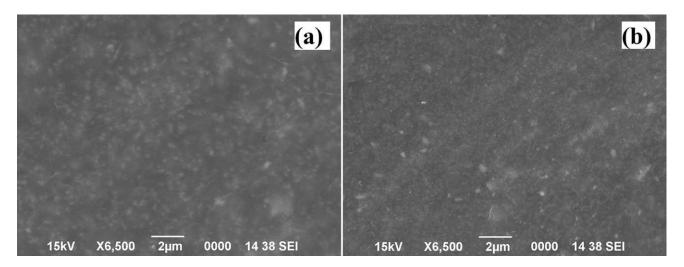


Figure 1 SEM micrographs for (a) test and (b) standard paints.

evaporation rate of solvent and viscosity influences the morphology of the paint.¹² The domain sizes of dispersed phases in the test paint were smaller than the standard paint which is due to the better compatibility of the components with the highly branched polyester resin. This is due to the confined geometry and low viscosity of the highly branched polyester resin.⁹ Furthermore, the high mechanical stress during ball milling had broken the dispersed phase into smaller particles in the matrix. So, all the ingredients were homogeneously disintegrated in both the paint systems that were observed from the SEM micrographs (Fig. 1).

Performance characteristics

The different performance characteristics of both the paints are given in Table III. Mesua ferrea L. seed oilbased polyester modified epoxy paint showed comparable gloss value with the industrially used epoxy paint for protective coating application. This gloss of the paints is due to smooth surface of the paint films which comes from good dimensional stability by the formation of dense network structure²⁹ as supported by swelling value (Table III). Also the resin itself possesses high gloss.⁹ Although the touch dry and hard dry times of the test paint were found to higher compared with the relevant standard epoxy paint, still they meet the requirements for protective coating applications and hence could be a focus for the customers. These comparable drying times are mainly due to the high surface functionality of the highly branched resin that takes part in the crosslinking reaction with the epoxy resin. The possible reaction occurs between the hydroxyl/ester group of polyester resin and hydroxyl/epoxide group of epoxy resin via H-bonding or other polar-polar interactions (Scheme 2). Self-life is the competence of the paint to remain stable without phase separation

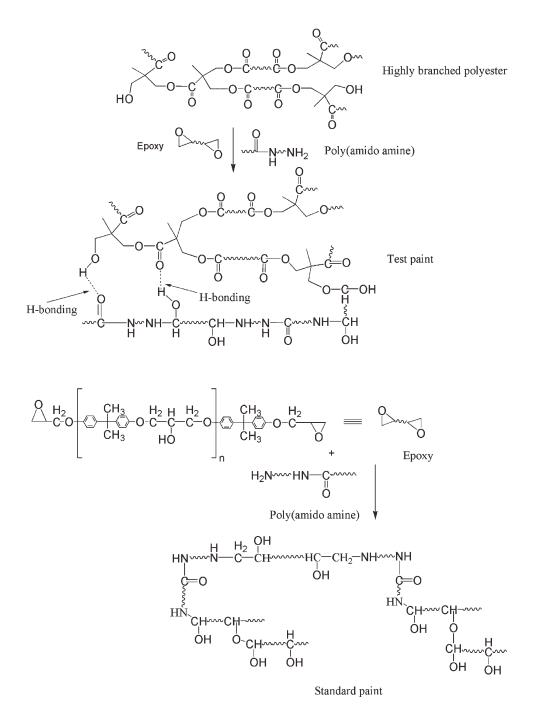
that is occurring in the stored container for a particular duration and can be used without any detrimental affect to its application properties or ultimate performance. The test paint as well as the standard paint possessed a pot life of 8 h without loss of homogeneity, which indicates their excellent storage availability. This is due to their low tendency towards skinning and appropriate viscosity of the binder systems. The scratch hardness value of the test paint was also comparable to the standard paint which fulfill service requirement for paint applications. The impact resistance of the test paint was quite satisfactory which may be due to good mechanical strength obtained from optimum crosslinking density and good flexibility³⁰ of the hydrocarbon chains of the fatty acids of the oils.⁹

The protection against corrosion for both the paints was assessed by exposing them to various chemical environments for a specified period of

 TABLE III

 Performance Characteristics of Test and Standard Paints

	Standard	Test	
Properties	paint	paint	
Protection against	Pass	Pass	
corrosion (after 25 days)			
UV exposure (after 544 h)	Pass	Pass	
Salt spray (after 15 days)	Pass	Pass	
Initial gloss (60° Byk)	55.6	60.4	
Percent gloss reduction	84	86	
exposure (544 h)			
Pot life (8 h)	Pass	Pass	
Scratch hardness (kg)	1.2	1.3	
Impact resistance (100 cm)	Pass	Pass	
Touch dry (h, at room	3	4	
temperature)			
Handleable dry (h, at room	8	10	
temperature)			
Swelling (% in xylene,	29	23	
after seven days)			



Scheme 2 Possible curing reaction mechanism of the paints.

exposure. Both the test and the standard paints were resistant to corrosion upto 25 days. The corrosion resistance test is important as it reflects the actual field performance of the paints. Initial damage of the paint was indicated by color fading. It is mainly due to the attack of moisture through oxidation or rust formation on the metal surface.³¹ To evaluate the resistance of the paint samples to corrosive chemicals environment the salt spray test was performed. In this test, the samples were kept in the environment of corrosive chemicals such as moisture, NaCl etc. Both the test and the standard paints were found to

possess excellent salt spray resistance of greater than 15 days. Such good chemical resistance of the paints is due to the low penetration rate of the corrosive chemicals through the paint films.⁶ The strong threedimensional networks and the presence of rigid aromatic moieties in the networks prevent the chemicals to pass through the paint films. The stability of both the paints towards harmful UV rays was checked by exposing them under UV rays for 544 h. According to IS 8662:2004, the durability of any painted panel under UV exposure of 250 h can withstand one year under normal environmental condition. So UV

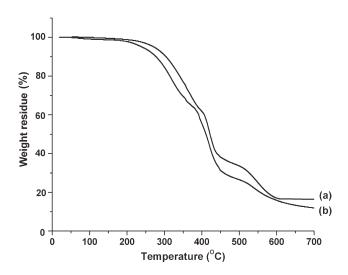


Figure 2 TGA thermograms for (a) test and (b) standard paints.

exposure of 544 h is sufficient time for durability study. Both the paints showed good resistance towards the UV rays for this specified period of time. However it was found that after the exposure to UV rays the gloss of the paint films was reduced and percent gloss reduction of the test paint was comparable to the standard paint. Generally, gloss retention of epoxy based paint under UV exposure is not very good and that is why epoxy based paint is not used for top coat application. This reduction of gloss after exposure may be due to the degradation of epoxy resin by the exposure of UV light.

Although most of the properties of the test paint were comparable to the standard industrial paint, yet there are added advantages of the *Mesua ferrea* L seed oil based highly branched polyester due to its low viscosity, high solubility and highly branch globular structure with freely expose groups, which helps in the processing, storage, application, and film formation of paints. Also it acts as reactive diluent for the epoxy resin, reduces the VOC emissions, and forms high solid epoxy paint.

Thermal study

TGA curves for the test and the standard paints are shown in Figure 2. From the figure, two-step degradation patterns were observed and found that the test as well as standard paints were thermally stable upto 240°C. However, the overall thermal decomposition temperature of the test paint was found to be higher than the standard paint. This higher thermostability of the test paint may be due to the presence of various thermostable units present in the renewable natural oil based binder system.⁹ Also, from the figure it can be seen that 50 wt % decompositions of both the paints was occurred at 410–421°C. Again the weight residue for the test paint was found to be

Rheological study

Rheological experiments were conducted on a controlled strain rheometer equipped with parallel plate geometry. Oscillatory measurements were performed to compare the rheological behavior of the highly branched polyester resin modified epoxy paint with the standard commercial epoxy paint. The viscosity is the most important rheological property for paint. To achieve good application characteristics and improve the manufacturing process, paints have to be non-Newtonian liquids, which are highly shear dependent.³² The variation of viscosity with time at controlled stress and single shear value is shown in Figure 3, where viscosity remained almost constant with time for both the paints. However an overall viscosity of the test paint was found to be lower compared with the standard paint. This is due to the highly branched globular structure of the binder of the test paint.⁹

Figure 4 shows the shear viscosity as a function of shear rate for both the paints. Viscosity at high shear rates must be controlled in a narrow range to give sufficient film buildup and coverage without extreme brush or roller drag, or too high pressure in spray painting.³³ The low shear rate viscosity must be high enough to prevent in-can settling and sagging after painting. As a result, shear-thinning behavior is needed. The shear viscosity of both the paint systems decreased with the increase of shear rate showing shear thinning behavior. At low shear rates, the shear viscosity data exhibited a strong pseudoplastic behavior of both the paints.

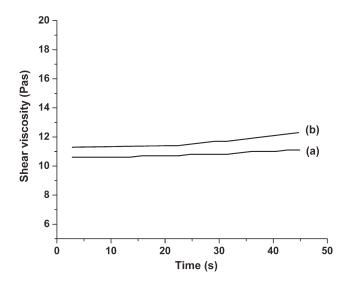


Figure 3 Variation of shear viscosity against time at constant stress and temperature for (a) test and (b) standard paints.

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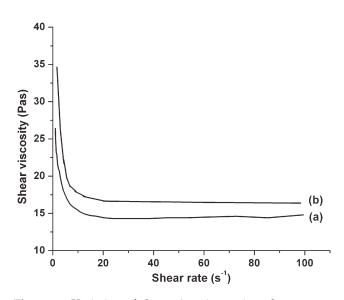


Figure 4 Variation of shear viscosity against shear rate at constant temperature for (a) test and (b) standard paints.

CONCLUSIONS

Mesua ferrea L. seed oil based highly branched polyester resin can be successfully utilized as a reactive diluent of epoxy resin system for the preparation of epoxy paint. The various physical properties, performance characteristics of the tested paint are found to be comparable to that of the standard industrial epoxy paint. Mesua ferrea L. seed oil may be used as an alternative raw material since it is renewable, oil content of the seeds is exceptionally high and available in many parts of the world. Moreover, the low viscosity of Mesua ferrea L. seed oil based highly branched polyester resin acts as reactive diluent of epoxy resin results to high solid content of the paint and low VOC emission which can serve as a remedial step to maintain clean environment, an important aspects of eco-friendly coating. Because of excellent resistance towards salt spray, corrosion, UV-ray of the test paint it would gain many perspectives as high performance industrial paint in the automotive and wood industries.

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