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Chassis Coating Agent and Emulsion Paints

Introduction

Chassis coating agents are roughly divided into two types according to their conditions and their main objectives of coatings.

The one is the coating to protect underbodies of automobiles strongly from rust, for which heat-sticking type paints are used in general.The another one is coated at automobile inspections in after the purchase market of automobiles. It is for the protection of underbodies of automobiles from rust additionally every 2 or 3 years by coating already coated surfaces at automobile manufacturing lines.

“The Underbody Coating Standard JASO (Japan Automobile Standard Organization) 7006” (Table 1) is shown in the next page.

Contents

Table 1. The Underbody Coating Standard (JASO 7006)

Item \ Class		First class A	First class B	Second class
Storage stability		No hard mass, no significant isolation of varnish, and no increase in viscosity that hinder the operation.		
Consistency		320 to 380		95 to 115 (KU)
Specific gravity		Determined by the agreement among the parties concerned.		
Remaining ratio after heating (%)		65 or more		60 or more
Workability		Uniform patterns are formed and smooth coating is available without clogging or irregularity in the flow of the spray gun.In addition, without drip or flow of coated layer.		
Drying property (h)	Dry tack	2 or less		
	Baking	24 or less		
Sticking test	Back sticking	—	Without swelling or peeling off.	
	Vertical sticking	—	Without swelling or peeling off.	
Oil resistance		No significant softening, swelling, or peeling is observed after 3 hours of dipping in gear oil at 50°C.		
Boiling water resistance		No significant bleaching, swelling, or peeling is observed after 30 minutes of dipping in water at 98°C.		
Acid resistance		—	—	No swelling, significant softening, and adherence after 24 hours of dipping in 2% sulfuric acid at 20°C.
Alkali resistance		—	—	No swelling, significant softening, and adherence after 6 hours of dipping in 5% sodium carbonate solution at 20°C.
Low temperature adhesion		No cracks or peeling is observed after the prescribed slam test at –20°C.		
Shock Resistance		No cracks or peeling.		
Aging resistance		After 75 hours of accelerated weather resistance test, no significant bleaching, swelling, and peeling is observed. In addition, no peeling after the impact test.		
Combustibility		Can be extinguished within 10 seconds.		
Corrosion resistance		The corroded widths of the both sides of the nick lines are 3mm or less for one side and the rusted area is 25% or less without swelling or softening on the paint film.		
Abrasion resistance		After ten cycles of shots of cast iron is applied with the prescribed method, no lifting or unevenness of paint film is observed in 20% or less area of the paint film.		
Vibration resistance (db)		6.0×10^{-2} or more		

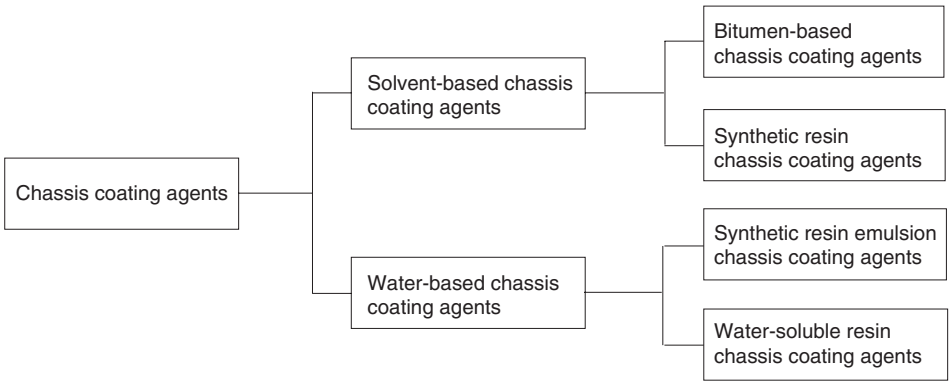
Main component is the first class normal bitumen, coated relatively thick.
A: Baked at room temperature B: Heated and baked

Main component is the second class normal synthetic resin, coated relatively thin.

1. Outline of Chassis Coating Agents

Chassis coating agents are roughly divided into four types listed in Table 2.

Table 2. Classification of chassis coating agents



Next outline of each paint is given below.

1-1. Bitumen-based Chassis Coating Agents

Natural gilsonite, asphalt, and coal tar pitch, etc. are cured and fused together with drying oil and solved in organic solvent to be paints. Because of low-cost raw materials, good gloss and rust resistant performance, they have been used from before, however, the consumption is decreasing gradually due to the popularity of synthetic resin paints and water-based emulsion paints recently.

1-2. Synthetic Resin Chassis Coating Agents

They are the paints in which nitro cellulose, alkyd resins, and acrylic resins, etc. are used as bases. Since they become dry relatively fast and excel in various performance, a great amount is used even today. Although depending on the season as well, they are used more than water-based paints especially in cold regions.

1-3. Synthetic Resin Emulsion Chassis Coating Agents

They are chassis coating agents based on acrylic resin emulsion. Since they are aqueous dispersion type with low risk of inflammation, they are becoming popular after the first oil crisis rapidly due to resource saving and the organic solvent ordinance etc.

1-4. Water-soluble Resin Chassis Coating Agents

They are the paints in which water solubilized alkyd resins and acrylic resins are used as bases. Since the molecular weight cannot be larger than the one of emulsion paints, performance of paint film is inferior. In addition, since a great amount of alcohol-based solvents are needed to make paints, they are not used as much as the emulsion ones. However, because of the advantage of fast drying, the consumption is increasing for chassis coating agents for cold regions.

2. Emulsion Paints and Emulsion

Three Bond Co., Ltd. has made efforts to promote emulsion chassis coating agent as a major manufacturer of chassis coating agents in after the purchase market. They have become genuine products of almost all the automobile manufacturers today. In this section, we will explain in detail the outline of emulsion paints, which are our major products, and emulsion.

2-1. Outline of Emulsion Paints

Since the first oil crisis, the trend of solvent free of paint manufacturers and the requests for no pollution or low pollution paints from users altogether have raised requests for emulsion paints. With the following progress in polymerization technology in synthetic resin emulsion, coating performance and decorative functions have been deployed, raising its position in the painting field. Furthermore, since the synthetic resin emulsion paints have less content of organic solvent compared with not only solvent-based paints but also water-based ones, they are in the advantageous position from the viewpoint of pollution and resource saving.

The composition of emulsion paints is complex compared with solution paints because they use dispersion resins. The formulated components include not only synthetic emulsion and pigments but also plasticizing agents, film-formation auxiliary agents, viscosity bodying agents, viscosity adjusters, dispersing agents, wetting agents, antiseptic agents, antifungal agents, anti-foam agents, anti-freezing agents, and stabilizes etc.

2-2. Types of Emulsion for Paints

2-2-1. Classification of Synthetic Emulsions

The form of water dispersion resins are divided into colloidal dispersion, emulsion, and suspension according to the particle size. They are used as vehicles for painting. The characteristics of these dispersion resins and general water-soluble resins are classified in Table 3.

In addition, as resin emulsion for paints, vinyl acetate-based, vinyl acetate-acrylic-based, ethylene-vinyl acetate-based, vinyl acetate-vinyl chloride-ethylene-based, styrene-acrylic-based, acrylic-based, chloroprene-based, styrene-butadiene-based, epoxy-based, and urethane-based, etc. are used.

Table 3. Characteristic table of water-based paints by form

Characteristics	Water-solubility	Colloidal dispersion	Emulsion	Suspension
Particle size (μ)	0.005>	0.01 to 0.05	0.05 to 0.5	0.5 to 10
Molecular weight	Low	Medium	High	Low to Medium
Viscosity	High	Medium	Low	Low
Resin solid content	Low	Medium	High	High
Amount of organic solvent used	Much	Medium	Non	Medium
Coating workability (bubble and drip)	Bad	A bit bad	Good	Good
Gloss	Good	A bit bad	Bad	A bit bad
Film formation	Good	A bit bad	Bad	A bit bad
Physical performance	Bad	A bit bad	Good	A bit bad
Water resistance	Bad	A bit bad	A bit bad	A bit bad
Chemical resistance	Bad	A bit bad	A bit bad	A bit bad

2-2-2. Functional Emulsion Resins

Recently, the methods such as soap free or soapless, the combination of heterogeneous polymers, and crosslinked emulsion for the improvement in performance and the diversification of functionalities of emulsion paints are introduced at the synthetic stage of emulsion, leading to the significant expansion of applications and the application range.

Next, we will explain the outline of functional emulsions.

(1) Soap Free Emulsion

The surfactants, which are used for stabilization of emulsion particles and for the formation of the places for polymerization by micelles in the normal emulsion polymerization system, on the other hand, adversely affects coating performance such as water resistance and durability. Therefore, in place of the normal emulsifying agents, for the stabilization of polymer particles, besides the polymerization type and the self-emulsifying emulsifiers, the utilization of fragments (SO4– or –OH groups, etc.) of water-soluble resins or initiators, the introduction of hydrophilic groups and the like are put to practical use.

(2) Complex Emulsion

Using the polymer emulsion synthesized by emulsion polymerization as seeds, by polymerizing heterogeneous monomers inside these particles, complex emulsion with heterogeneous phase structure, in which two or more kinds of polymer molecules exist non-uniformly, have become available (see Figure 1).

As complex emulsion, there are the Core-Shell type, in which each heterogeneous polymer is layered in particles, the POO (Polymer Oil in Oil) type of sea and island structure, the PL (Partial Localized) type, and the IPN's (Interpenetrating Polymer Networks) type, in which heterogeneous molecular chains are intertwined.

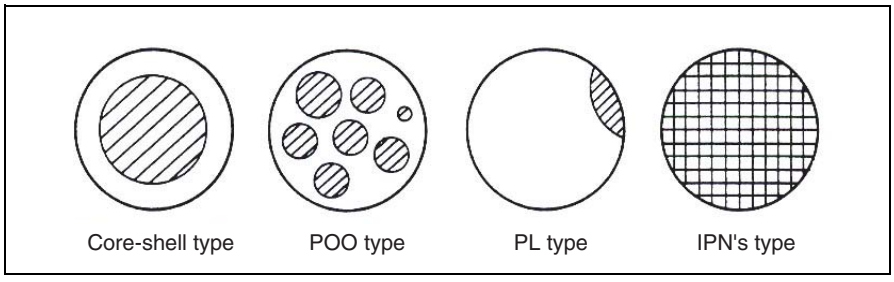


Figure 1. Types of complex emulsion

By selecting polymerization methods, chemical structures, and layer structures, diversification of performance such as corresponding physical properties have become available. These complex emulsions have become positioned as functional materials with wide range of applications.

With these complex emulsion resins, as coating materials as well, simultaneous realization of physical properties, film forming property, blocking resistance, pigment miscibility, and stain resistance, etc., which cannot be obtained with uniform emulsion resins, have become available. By using the Core-Shell type emulsion, in which rigid polymers are used in the core part and polymers with good compatibility miscibility and film forming property are used in the shell part, wide range of applications such as single-layer elastic covering materials, to which contradictory characteristics such as physical properties and stain resistance are required, and gloss emulsion paints have become available.

(3) Ultrafine Particle Polymer Dispersion

Ultrafine particle polymers such as acrylic hydrosol introduced by DuPont Corp. with the particle diameter of less than one tenth or around of that of normal emulsion are provided with compactness and gloss of coating film comparable to solution paints. In addition, because of the characteristics of high osmosis due to ultrafine particles, they are used widely for gloss emulsion paints for metals and building materials, building sealers, plastic paints, and leather paints, etc.

Furthermore, utilizing reactivity of carboxyl groups and hydroxyl groups introduced on the surface of particles, besides interparticle crosslink with the resins with amino groups and methylol groups, they are used as already crosslinked colloidal dispersion, in which crosslinked structure exists inside the particles as well.

(4) Reaction Type Emulsion

By introducing crosslinked system, not only water resistance, alkali resistance, solvent resistance, stain resistance, and blocking resistance but also improvement of hardness, adjustment of physical property behavior and the like become available. Besides thermal curing crosslinking using addition reaction or condensation reaction, a lot of room temperature crosslink emulsions using air oxidation, ionic bonds, various addition reactions, and UV curing, etc. have been developed.

For crosslinking system, besides uniform crosslinked type, in which crosslinking bonds are dispersed to the whole system and surface-crosslinked type, in which crosslinking is conducted mainly on the surface of the particles, mixed emulsion of crosslinked polymers and thermoplastic polymers and the like have been put to practical use.

Since the emulsion paint with the crosslinking system demonstrates functionalities specific to dispersion systems, from the viewpoint of low pollution, resource saving, and safety and health, it is considered as a predominant material in the movement from the current solvent-based paints to the water-based ones. It is believed that as the progress in technological development mainly in room temperature closslinking system, it will occupy important roles much more in the coating field in the future.

2-3. Formulated Components and Film Formation Mechanism of Emulsion Paints

We explained formation of emulsion paints briefly with the example of our chassis black paint in Figure 2.(see page 6)

It explains the model of simplified film formation, and we think that it will be helpful for the average person to understand.

However, the more complex model is actually needed.

We have explained composition of emulsion paints in 2-1. These roles are explained in (A) to (D) of Figure 2.

At first, (A) shows the state with the paints inside the container. The quality characteristics requested at this state are listed below.

(1) No color separation.

(2) The viscosity of the paint is the same to the prescribed viscosity and always constant.

(3) No mold.

(4) Even in case of freezing in winter, emulsion is not destroyed.

To maintain these qualities, the followings are needed.

(a) dispersing agents, (b) viscosity bodying agents, (c) stabilizing agents, (d) antifungal agents, (e) antifreezing agents etc.

(B) shows the state with the paint coated. The quality characteristics requested at this state are listed below.

(5) Easy spraying by the coating gun at painting.

(6) Constant coating film thickness is retained without collapsing.

(7) Good wettability of paints with the adherend.

To demonstrate these characteristics, the followings are needed.

(f) viscosity adjusters, (g) wetting agents, (b) viscosity bodying agents

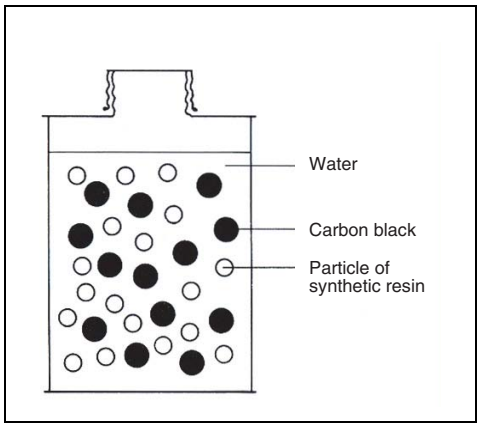
(C) and (D) show the model to form a film through water vaporization, the following characteristics are needed for smooth film formation.

(8) To keep prescribed time until water evaporates gradually to form a film.

(9) To form a film even at low temperature without freezing.
To demonstrate these characteristics, the followings are needed.

(h) plasticizing agents, (i) film-formation auxiliary agents, (e) antifreezing agents etc.

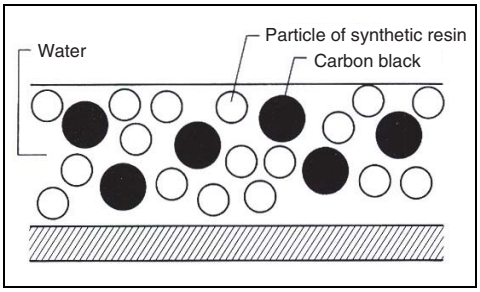
(A)



Synthetic resins and carbon blacks are dispersed in water. This is not the state that they are solved in water, thus it is different from resolution with the solvent type ones.

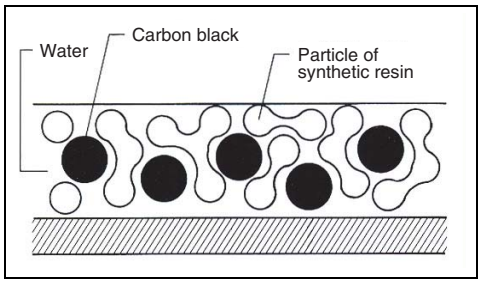
If the synthetic resins contact each other in water, two particles stick together. If this amount becomes large, the paint becomes gelled before the coating, making the paint useless. The paints provided with stability before usage are needed.

(B)



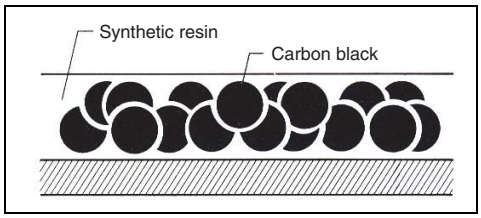
After painting, as water evaporates, the concentration of particles of synthetic resins and carbon blacks increases, which means the particles are close together. Then the particles contact each other to begin crosslinking.

(C)



As water evaporation proceeds, the distances between particles become much closer. With the proceeds of crosslinking among particles, the particles collapse to adhere.

(D)



As water evaporates, the particles adhere each other one after another, making a uniform film of synthetic resin.

This type of the film are filled with carbon blacks inside. This is how the black paint film is formed.

Figure 2. Paint film formation process of emulsion paints

3. Quality Characteristics Required for Chassis Paints and Advantages and Disadvantages of Water-based Chassis Paints

3-1. Quality Characteristics Required for Chassis Paints

Some quality characteristics required for chassis paints are listed below.

(1) Anyone can use the paint easily as it is. (cartridge type and aerosol type)

(2) Sufficient performance is obtained within 24 hours after coating.

(3) Room temperature drying type.

(4) Good base coat compatibility with all undercoatings.

(5) No hazardous materials of the fire protection law.

(6) Out of the organic solvent ordinance.(not contain 5 wt% or more of organic solvents of the Ordinance on the Prevention of Organic Solvent Poisoning in Japan)

3-2. Advantages of Water-based Chassis Paints

With water-based chassis paints, especially in case of emulsion paints, there are advantages listed below.

(a) Because of the high molecular weight, the high concentration, and low viscosity, emulsions can be high solid content paints. Furthermore, because of the high molecular weight, coating films provided with good water resistance, rust resistance, and weather resistance can be obtained.

(b) Since the Tg of the resin can be controlled by the combination of resins and selection of plasticizing agents, glossy paint films can be obtained with relatively small amount of solvent.

(c) By increasing the amount of solid content or selecting film-formation auxiliary agents, drying time can be shortened in spite of water-based paints.

(d) The organic solvent ordinance of the safety statutes of Japan can be cleared in general.

3-3. Disadvantages of Water-based Chassis Paints

(a) Slow drying speed compared with the solvent-based paints in general.

(b) Extremely slow drying speed at high temperature and high humidity, which may cause dripping or bleaching.

(c) Slow drying at less than 5°C in winter.

In all cases listed above, slower drying speed compared with the solvent-based ones are disadvantages, which are our research objectives in the future.

The dripping or bleaching at high temperature and high humidity of above-mentioned (b) is relatively often reported in water-based chassis coating agents. We will explain some causes for reference. (Figure 3.)

Water-based emulsion paints are uniform dispersion system of synthetic resins in water. They are originally polymerized with the addition of emulsifying agents and the like to improve wettability. As film-formation auxiliary agent, small amount of solvent, which promotes evaporation simultaneously with water, is added to the system to make paint. In case of under high temperature and high humidity, although a large amount of film-formation auxiliary agent and a small amount of water in the coated layer will evaporate, a large amount of water in the air will be absorbed into the coated layer. As a result, the coating film swells with water and the viscosity decreases, which causes dripping.

Furthermore, although this coating film will be dried at last, in the state of reduced amount of film-formation auxiliary agent, the film will not be formed uniformly. Therefore, delustering and bleaching occur.

There is a data showing that this phenomenon tends to occur under the condition with the humidity of 80% or more in general, therefore it can be prevented to some degree by flowing air compulsory using fans and the like as a countermeasure.This method is effective in winter as

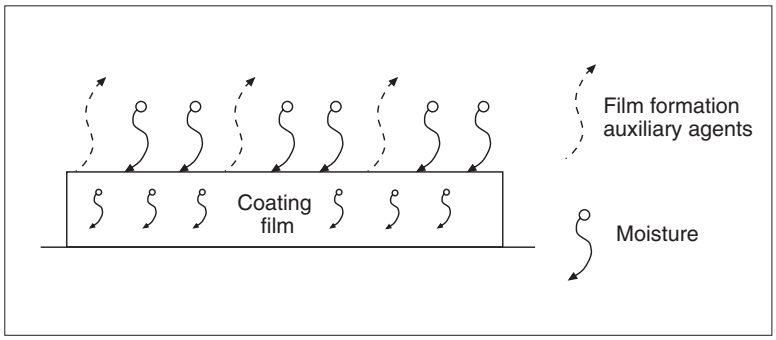


Figure 3. Causes of dripping and bleaching at high temperature and high humidity

4. Reference Performance and Characteristic Values of Water-based Chassis Coating Agents and Performance and Characteristic Values of our Products on the Market

Table 4. Reference performance and characteristic values of water-based chassis coating agents and performance and characteristic values of our products on the market

Item		Characteristics	TB6161* on the market	Test conditions and remarks
1 Tests regarding physical properties of paints				
Appearance		Black liquid	Black liquid	25°C visually
Viscosity		100 to 250cP	200cP	25°C BL type viscometer
Kinematic viscosity		20 to 25sec	25sec	25°C Ford cup No.4 JIS (Japanese Industrial Standard) K 5402
Specific gravity		0.95 to 1.1	1.0	25°C Cartesian diver technique
PH		8 to 10	9.0	25°C Glass electrode type
Remaining ratio after heating		30% or more	35%	JIS K 5400 8.2
2 Tests regarding utility properties of paints				
Dry tack time		Within 20min	15min	25°C 60%RH
Baking time		Within 60min	36min	25°C 60%RH
Minimum film-growing humidity		0°C or less	Passed	
Freezing temperature		−5°C or less	Passed	
Thermal cycle		Pass with 10 cycles or more	Passed with 10 cycles	−20°C×8h+40°C×16h
Sag test		Wet 100μm without dripping	Wet 150μm No dripping	25°C 60%RH Sag tester
3 Tests regarding physical properties of paint film				
Pencil scratch test		B to H	F to HB	JIS K 5400 6.14
Print resistance		Degree of adherence is not significant	Passed	JIS K 5400 6.8
60 degrees of specular glossiness		85 or more	90	JIS K 5400 6.7
Ball impact test		Once or more	Passed 3 times	JIS K 5400 6.13 B method 1/2 inch 500g 50cm
Ericksen test		5mm or more	Passed with 8mm	Ericksen tester
Flex resistance		10ϕ or less	Passed with 3ϕ	JIS K 5400 6.15
Adhesion	Cross cut test	100/100	100/100	1mm 100 squares Sellotape peeling
	Spiral scoring test	Without peeling	Passed	10mm eccentric circle Sellotape peeling
Overglazing compatibility	Appearance	Without repelling, cracks, holes, or swelling, etc.	Passed	
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	JIS K 5400 6.10
	Ericksen test	5mm or more	Passed with 8mm	
	Ball impact test	Once or more	Passed 3 times	
	Flex resistance	10ϕ or less	Passed with 3ϕ	
Under coat compatibility	Appearance	Without repelling, cracks, holes, or swelling, etc.	Passed	
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	Under coat is selected optionally
	Ericksen test	5mm or more	Passed with 8mm	
	Ball impact test	Once or more	Passed 3 times	
	Flex resistance	10ϕ or less	Passed with 3ϕ	
Top coat compatibility	Appearance	Without repelling, cracks, holes, or swelling, etc.	Passed	
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	JIS K 5400 6.11 Top coat is selected as optionally
	Ericksen test	5mm or more	Passed with 8mm	
	Ball impact test	Once or more	Passed 3 times	
	Flex resistance	10ϕ or less	Passed with 3ϕ	
Heat resistance test	Appearance	Without bubbles, cracks, swelling, or peeling, etc.	Passed	
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	130°C×3h
	Ericksen test	5mm or more	Passed with 8mm	
	Ball impact test	Once or more	Passed 3 times	
	Flex resistance	10ϕ or less	Passed with 10ϕ	

Item		Characteristics	TB6161* on the market	Test conditions and remarks
Cold resistance test	Appearance	Without bubbles, cracks, swelling, or peeling, etc.	Passed	
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	−20°C×3h
	Ericksen test	5mm or more	Passed with 8mm	
	Ball impact test	Once or more	Passed once	
	Flex resistance	10ϕ or less	Passed with 10ϕ	
4 Tests regarding chemical properties of paint film				
Water resistance test	Appearance	Without wrinkles, cracks, swelling, peeling, or delustering, etc.	Passed	25°C×24h
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
Hot water resistance test	Appearance	Without wrinkles, cracks, swelling, or peeling, etc.	Passed	50°C×24h
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
Boiling water resistance test	Appearance	Without wrinkles, cracks, swelling, or peeling, etc.	Passed	95°C or higher × 15min
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
Salt water resistance test	Appearance	Without cracks, peeling, and rust, etc.	Passed	NaCl 3% solution 25°C×96h
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
Salt water spraying resistance test	Appearance	Without rust, swelling or delustering, etc.	Passed	JIS K 5400 7.8 35°C×48H
	Rusted width of cross cut part	Within 3mm for one side	Within 1mm for one side	
	Cellotape peeling of cut part	Without peeling	Passed	
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
Accelerated storage stability	Deterioration of paints	Without deterioration	No deterioration	40°C×1 month
	Precipitation of pigments	Without significant precipitation	Almost non	
	Change in viscosity	Within ±20%	155cP	
	Change in kinematic viscosity	Within ±20%	24sec	
	Tests for paint films in section 2 to section 4	Without difference	No difference	
Storage stability	Deterioration of paints	Without deterioration	No deterioration	25°C×6 months
	Precipitation of pigments	Without significant precipitation	Almost non	
	Change in viscosity	Within ±20%	160cP	
	Change in kinematic viscosity	Within ±20%	25sec	
	Tests for paint films in page 2 to page 4	Without difference	No difference	
Accelerated weathering test	Appearance	Without bubbles, cracks, swelling, or peeling, etc.	Passed	Accelerated weathering tester 200h
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
	Ericksen test	5mm or more	Passed with 8mm	
	Ball impact test	Once or more	Passed 3 times	
	Flex resistance	10ϕ or less	Passed with 10ϕ	
Weathering test	Appearance	Without bubbles, cracks, swelling, or peeling, etc.	Passed	6 months of outdoor exposure
	Cross cut test	100/100	100/100	
	Spiral scoring test	Without peeling	Passed	
	Ericksen test	5mm or more	Passed with 5mm	
	Ball impact test	Once or more	Passed once	
	Flex resistance	10ϕ or less	Passed with 10ϕ	

* ThreeBond 6161

5. Future Issues of Water-based Chassis Coating Agents

Although the market of water-based chassis coating agents will grow in proportion to the numbers of automobiles in Japan, drastic growth is not expected in future. Furthermore, there are some severe factors such as the influence of the shortening of the interval of the automobile inspection to 3 years after the purchase. Under these circumstances, how to extend its market share will be a key issue to extend volume of sales for each company as well. It means that how to plan marketing strategy and the like will be important. From the viewpoint of the products side, how to differentiate

from the competitors' products will be an important factor. The following factors are conceivable.

(1) Gloss

To provide gloss like oil-based lacquer by water-based one.

(2) Drying Property

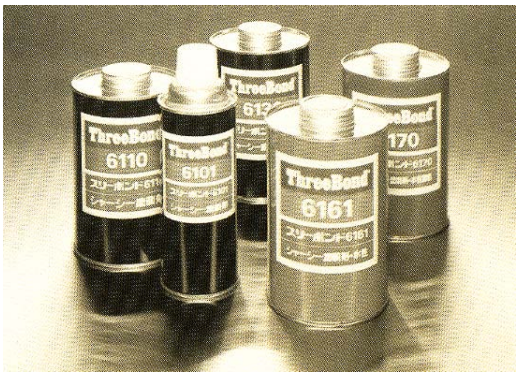
How to shorten the time from the dry tack to baking.

(3) Durability of Coating Film (especially to maintain gloss)

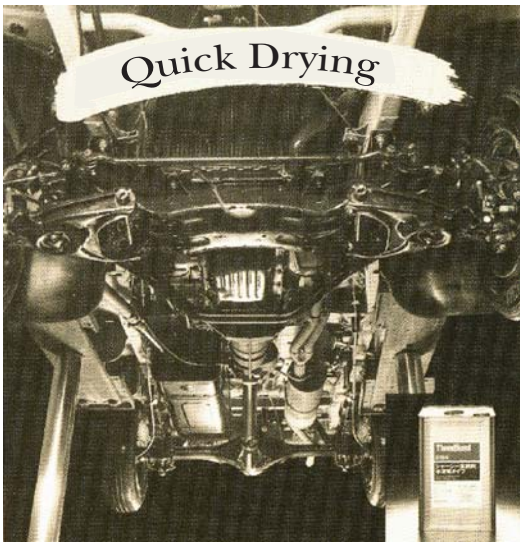
It is the performance especially requested from the truck market of large vehicles.

(4) High-security Products

One of the characteristics of water-based paints is that they can clear the organic solvent ordinance. Since the fire protection law is under review for amendment at present, there is no information about what the final regulation would be like. Not only in the organic solvent ordinance but also how loose regulation would be applied in the fire protection law would be a key issue as well.



Our Chassis Coating Agents



Conclusion

We have explained various subjects regarding emulsion paints, however, we don't feel fully satisfied because many of them were about general subjects.

In the field of paints, the compounding technique is the main issue. Since there is a variety of know-how, we could not explain it quite in detail.

For example, we could not explain how to provide gloss with small amount of film-formation auxiliary agent and how to prevent dripping and bleaching at high temperature and high humidity because they were kinds of deceptively easy know-how. Instead, we will produce new products using compounding technique know-how for you in future. We hope that you will use them to be satisfied with them. Please excuse our insufficient explanation in this technical news.

≪ References ≫

(1) K. Hanamura, “Coating Technology” Sep. 1979

(2) H. Kiryuu, H. Kasamatsu: “Development of High-functional Coating Materials” (CMC Publishing CO., LTD.)

(3) Br.Pat.1,114,133 (1965),US.Pat.37,740,367 (1973)

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