

**EFFECT OF STRUCTURE AND THICKNESS OF COATINGS AND
CONTAMINANTS
ON THE CORROSION****Manu Gupta^{*}, Deepti Shikha^{**} & P.K. Kamani^{***}**Deptt. of Oil and Paint Technology
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Tel.: +91-0512-2533502, Fax : +91-0512-2533812**ABSTRACT**

The presence of soluble salts (chloride, sulphate & nitrate) and their effect at the coating-metal interface was studied along with the chemistry of coating, water and oxygen permeability, coating thickness and metal surface preparation. Of course, the macroscopic defects, caused due to improper designing, handling, etc. can also not be over looked. What so ever the coating be the surface anomalies have their own role in deciding the coating life. Nevertheless there are still some coating systems which can reduce or postpone corrosion mechanism. Five different popular coating systems (e.g. phenolic, polyurethane, cardanol, vinyl & alkyd) were exposed to humidity for different time, 100 and 400 hours, and the under film corrosion was determined. The study reveals that carefully designed coating systems applied on the substrate with some fundamental knowledge can significantly improve the performance of coating and metal protection as well.

Key words: Soluble Salts, Interfacial Chemistry, Corrosion, metal protection, Film defects.

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INTRODUCTION

Corrosion is a gradual deterioration of a material caused by the chemical or electrochemical reaction with its environment. Since metals compared to non metals e.g. ceramics, plastics, rubber, concrete, etc. have a high electric conductivity, their corrosion is usually of an electrochemical nature. In the case of non metallic materials which are electrically non conducting, the corrosion is their deterioration from chemical causes. In fact, corrosion has been quoted as ‘Vulture of Metallurgy: as it eats away the metals’.

A metal surface which is macroscopically smooth and homogeneous is not so on the atomic scale but has a mosaic or lineage structure, whereas crystal substructure consists of slightly distorted blocks caused by unequal growth of parts of the crystal. Vacancies, dislocation frequently associated with surface steps and long terraced growth spirals — grain —boundaries and sub boundaries introduce a source of disarray of metal lattice, in addition there are some macroscopic defects caused due to poor design, poor processing, poor welding, careless handling or operation etc. The surfaces of materials become strained as a result of sliding, rolling, rubbing and other mechanical activities. A wear surface becomes different electrochemically from its surroundings and thus causes various types of corrosion¹. The presence of stress, particularly tensile stresses are the basis of most important macroscopic defect. All these surface imperfections also influence many of the characteristics of materials, such as mechanical strength, electrical properties and chemical reactions². Corrosion is, therefore, essentially a surface phenomenon, resistance to corrosion is often the result of the formation of some type of film on the metal surface.

The surface active agents, present on metals and non-metals are liable to change the surface activity of these materials and their mechanical properties as well. Tribological properties such as adhesion, friction, deformation wear etc. of solid surfaces are extremely dependent on the absorbed surface – active ions or molecules (i.e. environmental constituents)³

Organic coatings function by protecting substrates from physical and chemical attack. In some of the cases, however, this attack can be promoted rather than hindered by the

presence of the coating, for example when the substrate is contaminated. At the coating-metal interface, due to the presence of soluble foreign ions e.g. chloride or sulphate or leached ions from the coating in presence of even microscopic amounts of water and oxygen, two main phenomena can take place, blistering (local osmotic cells) of the coating and under film corrosion. There are other mechanism too which are responsible for the formation and growth of blister e.g. swelling, phase separation during film formation, temperature cycling or loss of adhesion etc.

Contaminants

The presence of contaminants e.g. oxides, salts, organic compounds and water etc. on steel surfaces prior to the application of coating materials have a deleterious effect on the coating performance. Complete removal of these substances is impossible⁴. Sulphate and chloride ions are most common contaminants in industrial and marine atmospheres, respectively. These contaminants are due to the combustion of coal and other fuels and sea water spray, which under certain wind conditions can penetrate many kilometers inland. De-icing salts on traffic roads are also a source of chloride contamination⁵. Gross characterized saline deposits were noticed on organic coatings applied to bridges. Chloride, sulphate, nitrate and carbonate were the anions and sodium, calcium and ammonium were the cations mainly found, as well as cations leached out from pigments⁶. In areas with high concentration⁵ of industry and dense population the air is strongly polluted with sulphur dioxide, which is also spread over long distances.

Blistering and Adhesion

Of the various mechanism, osmosis is considered to be the most responsible cause for blister formation in the organic coatings on metal surfaces, particularly on steel. Osmotic pressure here may be between 2500-3500 k Pa⁷⁻⁹ while mechanical resistance of the coatings to deformational forces are considerably lower, 6 to 40 kPa. The development of blister is due to the loss of adhesion over the respective area and in rest of the areas the coating is intact. The adhesive tape test depicted the loss of adhesion even before the blistering was visible. Obviously, although the interfacial forces keep the film on the substrate at the area surrounding the blister but are weak

enough to resist the force of tape. This weakened coating/metal interfaces allow a direct electrolytic connection of anodes and cathodes.

In electrochemical corrosion, oxygen depolarizes cathodic areas with production of hydroxylanions. In the presence of salts say NaCl, as electrolytes, cations may migrate to cathodic areas and form NaOH, which is responsible for the strong alkaline reaction of the aqueous solution present in these blisters. Migration of cations to cathodic areas may take place through the coating¹⁰ or along the coating-metal interface. The diffusion rates of Na cations is very slow [10^{-9} — 10^{-13} , cm^2/sec^2] even with the films relatively permeable^{11, 12}. The initial concentration of osmotically active substances in the film-substrate interface is generally lower than that of the external aqueous solution of NaCl used in salt-spray or immersion tests. This difference increases the cation diffusion through the coating to cathodic areas of the metal surface. Thus migration of sodium ions from paint film causes blister formation at cathodic areas¹³.

Protective Coatings

Rust-protective paints can be made today with a very high quality. With a good surface preparation and a sufficient film thickness, a life of 15-20 years can be expected for an organic coating. Corrosion in a shorter time is generally limited to pores, mechanical damages, and areas where the film thickness is low, e.g. at edges (Fig. 1).

Such defects can usually not be totally avoided. It is therefore, of great importance that the paint should have the ability to protect the surface from the spread of rust around a defect. Metal substrate profile is also a very important factor as regard to the life of coating. The flow out of a highly viscous coating will only occur on a surface profile that provides even peak to valley configuration. A profile of bent- over peaks, cracked surfaces, etc. will not allow displacement and wet out by a fast curing highly viscous coating. Any voids left in the coating-metal interface at the point of permeation will of course be immediately filled with water, promoting the chemical reactions necessary to form blister. We are also aware the coatings do not fail by the square inch; they fail one molecule at a time. The effect of blast cleaning was

observed¹⁴. The physical configuration of metal surfaces, prepared by aluminum oxide or silica sand blasting, provided excellent flow out on a protective coating. The craterlike profile provided by steel shot was found to have excellent flow out characteristics while the steel grits provided the worst surface for flow out characteristics.

Bayliss and Bray have observed that in tests of polyurethane film very small voids or bubbles can be found in the film, and they report that protective coatings applied by the airless spray method tend to have more voids or bubbles trapped in the film than to those applied with conventional sprayer applicator. That means the life of coatings also depends upon the application mode. A new plasma coating system with significantly improved corrosion resistance of automotive steel has been reported¹⁵.

Most of the work have been done on some particular water soluble contaminants e.g. sodium chloride and iron sulphate. This paper deals with other water soluble contaminants e.g. sulphate, chloride and nitrate salts of cation (Na^+), at the coating — metal interface, various types of organic binders are taken as coating materials for the study.

EXPERIMENTAL

The varnishes were prepared by dissolving the chosen resins (Table 1) in suitable solvents of suitable consistency for brush application and named in the same order as resins, shown in the following table.

Cold rolled mild steel and glass panels were prepared for different tests. The viscosity of the prepared binder solution were measured by ford cup No. 4. The films were tested against acid resistance, alkali resistance, corrosion scratch test, adhesion and hardness as per standard test methods. For corrosion scratch test, artificial sea water (a representative sample) was prepared by dissolving the following quantities of chemicals in one litre of water.

Chemicals	Water (gm)
Sodium chloride	28.05
Magnesium chloride	2.95
Magnesium sulphate	1.75
Calcium sulphate	1.30
Potassium chloride	0.65
Potassium bicarbonate	0.15
Potassium bromide	0.10

Corrosion Scratch Test

The 5 cm x 10 cm x 1 mm thick mild steel panels were degreased, sanded and coated. The coated panels were left for a week (7 days) in the laboratory at room temperature for complete curing. They were edged with wax and one face of each panel was scratched to the substrate with a sharp blade. The panels were exposed to artificial (synthetic) sea water for 500 hours; then washed with distilled water and dried panels were observed for rusting. The specimens were periodically inspected in order to evaluate rusting and blistering. The adhesion along with groove was determined in conventional manner.

Humidity Test

Cold rolled mild steel panels without visible rust was used. One side of the specimen was contaminated using 200 and 700 mg/m² of Cl⁻¹, SO₄⁻² and NO₃⁻¹. Uncontaminated steel panels were used as controls. Sodium chloride, sodium sulphate and sodium nitrate solutions were prepared by using reagent grades and distilled water. The clear coatings were applied and the coated specimens were left for a week in the laboratory at room temperature for complete curing. Thereafter the uncontaminated reverse side of the specimens was protected by a strippable coating. The edges were sealed with wax. The coating was applied in two thickness 20 and 60 µm. The exposure times were 100 and 400 h.

The specimens were carefully observed during the test for rusting and blistering according to ASTM D610 and D714 specifications, respectively. The under film corrosion rate was determined gravimetrically by weighing the specimens before the application of the contaminants and after the test and removal of the coating and corrosion products.

RESULTS

The laboratory tests (acid resistance, alkali resistance, corrosion scratch test, adhesion and hardness) were performed on all the varnishes. The results obtained are summarized in Table 2.

* Rust in groove only and no spreading under the film and good adhesion along the groove sides and no film defects.

** slight rust spot under the coating in addition to rust in the groove and no loss of adhesion

Table 3 depicts the humidity test results after 100 and 400 h of exposure. The rating of rusting was done visually and compared with the ASTM D-610 specification and also with the results⁴.

Table 4 shows the blister performance in 100 and 400 hours. Blistering was rated by visual examination and compared with the ASTM D714 specification. Under film corrosion rate was determined by gravimetric method after 100 and 400 hrs and has been shown in Table 5.

DISCUSSIONS

The laboratory test results of all the varnishes have been shown in Table 2. They all have good film properties. The corrosion scratch test possessed good corrosion protection except one based on alkyd which possess slight rust spot under the coating.

Most of the corrosion activities are playing their role at the coating-substrate interface. The availability of oxygen at the interface depends on the permeability of the coating. The thickness of the coating and chemical structure are the common

deciding factor of permeability. A highly polar binder has excellent gas barrier properties, and is very sensitive to water permeation, whereas for a non-polar binder the reverse is true¹⁶⁻¹⁸. The oxygen permeability of an organic coating may be high, but not sufficient for the corrosion to take place as on bare surface⁵ but on the other hand water permeability is generally higher than what is required for the corrosion process¹⁹.

It is imperative to have water and oxygen both for the cathodic reaction of the metallic substrate corrosion and their influence on the corrosion process is discussed here. Table 3 shows that panels coated with alkyd generally depict stronger rusting than those coated with cardanol, vinyl polyurethane and phenolic. Table 4 shows that polyurethane and phenolics have higher water permeation than other resins and vinyl has lowest among them, however it is also shown in the Table 6 that the alkyd and vinyls are having higher oxygen permeability compared to rest. It has also been said by many researchers that oxygen permeability is the controlling factor, determines the corrosion process specially in low film thickness¹⁹.

The results in Table 4 show that the water at coating-metal interface is the basic culprit in the adhesion failure, agree with the literature⁵.

The coating acts as a semi permeable membrane and the contaminant form the blister as the water permeates through the film and lowers the concentration of contaminants⁵. The coating fails due to blister (Table 6).

The polar nature of resins shows more blister as in polyurethane and phenolic while the non polarity presents stronger rusting as in alkyd and vinyl (shown in Table 4 and 5). Hence, diffusion of water controls the loss of adhesion of the coating. With reference to the Table 3, 4 and 5 and Morcillo. It can be said that 100 hours are sufficient for water to permeate through the coating and dissolve the contaminants present at the coating-metal interface, but not enough to produce perforation of the coating as a consequence of water accumulation or the growth of rust. The concentration of contaminants at the interface is prone to underfilm corrosion and does not much depend upon the type of contaminants. The underfilm corrosion is more in low film thickness coatings and as the coating thickness increases (above 35-

40 μm to 60 and to 80 μm) the corrosion is not much affected but below 20 μm corrosion is very much dependent on film thickness. As the film thickness increases, the oxygen permeability in the beginning decreases and after a certain film thickness it becomes almost constant.

The sulphur dioxide is not only a danger from biological point of view but has also a strong corrosive action (Table 7). Sulphur dioxide is absorbed to nearly 100% in a humid rust layer and readily oxidized to sulfate which is a dangerous component active in the corrosion process²⁰.

Heavily polluted

When iron sulfate is oxidized to iron oxide (Fe_2O_3) the released sulfate ion, reacts with more iron. Figure 2 shows the relation between integral corrosion and sulfur dioxide deposition rate²¹.

Sodium chloride is in the natural environment found mostly in coastal areas and little in road salting. Out door deposition rates are given in Table 8. The stimulating action of sodium chloride on corrosion is due to the fact that the iron chlorides are soluble and hygroscopic, that they increase the surface conductivity and that the chlorides actively prohibit passivation. In out door exposure there is a close connection between the integral corrosion and the deposition rate of sodium chloride in the absence of air pollution²² Fig.3.

CONCLUSION

It has been concluded that there is no universally applicable mechanism of cathodic disbondment, however, the more durable the coating (more resistant to alkaline hydrolysis) the more likely is the interfacial separation rather than a cohesive failure as a result of coatings degradation. In some cases (where the oxide is thick) it may be a precursor to disbondment as hydroxyl ions are more readily available at the metal-oxide interface than of the oxide-polymer junction.

The corrosion can be minimized to certain extent by arresting the oxygen permeation. It can also be seen that chloride contaminations are more corrosive than nitrate contaminations and sodium sulphate does not show remarkable corrosion even with low film thickness. Corrosion can also be subsidized to a greater extent by

1. Controlling osmotic pressure which also depends upon the type of contaminants at the coating metal interface²³.
2. The conductivity of the saline solution at the interface is also an important factor which increases the corrosion rate with increase in the conductivity.
3. The solubility of contaminants at the interface shows (Table 9) that sulphates have low solubility and Nitrates have high osmotic pressure and consequently high solubility and low dilution, which causes low corrosion rate.
4. Thickness and structure of coatings are also the important parameters in the corrosion. As the thickness increases, the corrosion first decreases and after certain thickness corrosion becomes almost constant. It has also been reported²⁵ that single thick coating is not as good in corrosion control as double layer coating providing the parallel film thickness.
5. The presence of oxygen stimulate the corrosion and the concentration of the corrosion stimulant define the under film corrosion process.

6. The kinetic of the steel corrosion is governed by the osmotic pressure, ionic conductivity and oxygen solubility of the aqueous electrolyte solution, and by the water solubility of the corrosion products⁴.
7. Even the best protective coating can fail premature by disbonding if the metal surface is not properly prepared for coating¹⁴.

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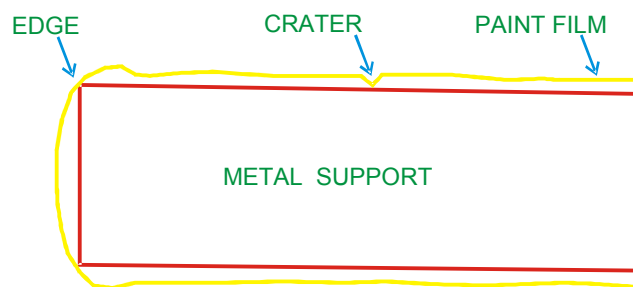


Fig. 1 Week points of a Coating

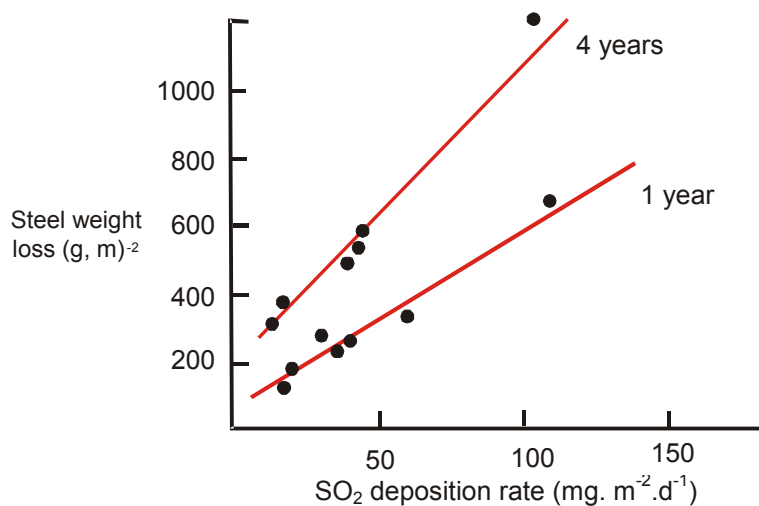


Fig. 2 Integral Corrosion vs. Sulphur Dioxide Deposition Rate

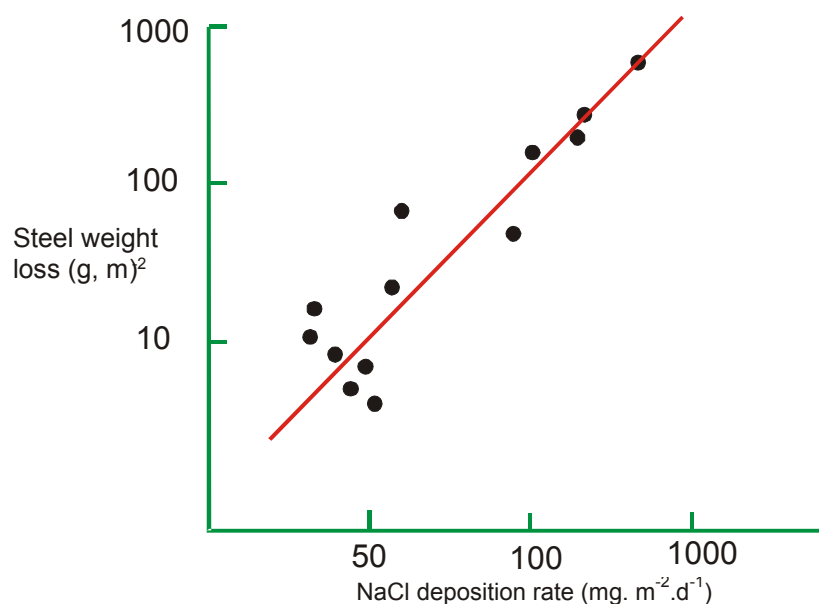


Fig. 3 Integral Corrosion vs. sodium chloride deposition rate

TABLE 1: Resins/Binders with Their Composition

1.	Phenolic	Oil — soluble, alkyl- phenol — formaldehyde
2.	Polyurethane	Polyester resin- aliphatic isocyanate (44:36)
3.	Cardanol	Cardanol — epoxy (75:25)
4.	Vinyl	Vinyl chloride- vinyl acetate (85:15). Chlorinated paraffin
5.	Alkyd	64% long linseed oil

TABLE 2: Test Results

Varnish No.	Acid Resistance	Alkali Resistance	Corrosion Scratch	Adhesion & Hardness
1.	Passed	Passed	A*	Good
2.	„	„	A*	„
3.	„	„	A*	„
4.	„	„	A*	„
5.	„	„	B**	„

Table 3 : Humidity Test Results

Resin /binder (thickness μm) Concentration (mg/m^2)	Rusting grade ***					
	NaCl Time (h)		Na ₂ SO ₄ Time (h)		NaNO ₃ Time (h)	
	100	400	100	400	100	400
PHENOLIC						
(20)* 200**	10	10	9	9	9	10
(20) 700	10	9	9	8	8	8
(60) 200	10	10	9	9	10	10
(60) 700	10	10	9	8	9	8
POLYURETHANE						
(20) 200	10	10	9	8	10	9
(20) 700	10	8	8	7	8	7
(60) 200	10	10	9	9	10	9
(60) 700	10	10	8	7	7	9
CARDANOL						
(20) 200	10	10	9	8	9	9
(20) 700	9	9	8	7	9	8
(60) 200	10	10	10	9	10	9
(60) 700	10	9	9	8	9	8
VINYL						
(20) 200	9	8	9	9	10	9
(20) 700	8	8	9	8	9	7
(60) 200	10	10	10	9	10	10
(60) 700	9	8	7	7	10	9
ALKYD						
(20) 200	10	9	10	8	9	8
(20) 700	9	8	8	7	8	8
(60) 200	10	10	9	9	10	9
(60) 700	9	9	8	7	7	8

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* film thickness μm

** concentration of contaminants (mg/m^2)

*** ASTM D610 Specification : numerical rusting scale of rusted surface, expressed as area % : 10, <0.03; 8, <0.1%; 7, <0.3; 6, <1%; 5, 3%; 4, 10%; 3, 16%; 2, 33%; 1, 50%; 0.

Table 4 : Blistering Results

Resin /binder (thickness μm) Concentration (mg/m^2)	Rusting grade ***					
	NaCl Time (h)		Na ₂ SO ₄ Time (h)		NaNO ₃ Time (h)	
	100	400	100	400	100	400
PHENOLIC						
(20)* 200**	10	8 MD	8 MD	6 MD	8 MD	6 M
(20) 700	8 MD	6 MD	6 D	4 F	6 F	4 F
(60) 200	8 MD	6 M	6 M	4 M	8 F	6 MD
(60) 700	6 MD	2 MD	4 MD	4 F	4 M	4 F
POLYURETHANE						
(20) 200	10	8 MD	8 M	6 MD	8 D	6 M
(20) 700	10	6 MD	6 M	2 F	4 F	4 F
(60) 200	8 MD	6 D	6 M	4 M	8 F	6 D
(60) 700	6 D	2 MD	4 M	4 F	4 M	4 M
CARDANOL						
(20) 200	8 M	6 MD	8 M	6 M	8 M	6 M
(20) 700	8 MD	6 MD	6 MD	4 M	4 F	2 F
(60) 200	10	8 MD	6 M	4 M	8 F	6 F
(60) 700	6 MD	4 D	4 M	4 F	4 M	4 F
VINYL						
(20) 200	6 MD	6 M	6 M	6 MD	8 MD	8 D
(20) 700	4 MD	4 D	4 M	2 MD	2 MD	2 M
(60) 200	8 MD	6 F	6 F	6 M	8 M	8 F
(60) 700	8 D	4 F	4 M	4 F	6 M	6 D
ALKYD						
(20) 200	6 MD	6 M	8 M	6 M	6 F	6 M
(20) 700	6 M	4 F	6 M	4 F	4 F	4 MD
(60) 200	8 M	6 MD	6 MD	6 M	8 F	6 MD

(60) 700	6 M	4 F	4 M	4 MD	6 M	4 F
<p>* film thickness μm ** concentration of contaminants (mg/m^2)</p> <p>- ASTM D714 specification</p> <p>- numerical scale 10-no blister, 8-smallest size blister can be seen by naked eye and 6,4,2 in increasing order of blister size, D-dense, MD-medium dense, M-medium, F-few</p>						

Table 5 : Under Film Corrosion

Resin /binder (thickness μm) Concentration (mg/m^2)	Under film corrosion rate $10^{-6} \text{ g}/\text{cm}^2/\text{day}$							
	NaCl Time (h)		Na ₂ SO ₄ Time (h)		NaNO ₃ Time (h)			
PHENOLIC	100	400	100	400	100	400		
(20)* 200**	ND	ND	ND	ND	ND	ND		
(20) 700	8	ND	ND	ND	ND	ND		
(60) 200	ND	ND	ND	ND	ND	ND		
(60) 700	ND	10	ND	15	28	20		
POLYURETHANE								
(20) 200	ND	ND	ND	ND	ND	ND		
(20) 700	ND	32	ND	ND	ND	ND		
(60) 200	ND	ND	ND	ND	ND	ND		
(60) 700	ND	ND	ND	ND	ND	ND		
CARDANOL								
(20) 200	20	10	ND	ND	33	63		
(20) 700	180	96	20	8	87	72		
(60) 200	ND	ND	ND	ND	ND	ND		
(60) 700	ND	14	ND	ND	ND	21		
VINYL								
(20) 200	ND	15	30	ND	ND	ND		
(20) 700	70	52	45	10	20	ND		
(60) 200	ND	ND	ND	ND	ND	ND		
(60) 700	ND	ND	ND	6	8	ND		
ALKYD								
(20) 200	40	33	21	8	14	24		
(20) 700	244	170	55	43	112	97		
(60) 200	20	9	ND	ND	12	6		

(60) 700	48	29	44	ND	27	19
* film thickness μm						
** salt concentration (mg/m^2)						
ND – Not detectable						

TABLE 6: Water & Oxygen Permeability of Resins

Types of resins	Permeability	
	Water	Oxygen
	mg/cm ² /day	
Phenolic	17.41	8×10^{-3}
Polyurethane	16.12	13×10^{-3}
Cardanol	15.2	21.6×10^{-3}
Vinyl	3.34	113×10^{-3}
Alkyd	8.20	82×10^{-3}

TABLE 7 Occurance of Atmospheric Sulfur Compounds according to ISO N43E

Deposition rate SO ₂ , mg.m ⁻² .day ⁻¹	Concentration in air µg.m ⁻³	Type of atmosphere
0 – 20	0 – 30	Clean, rural
20 – 60	30 – 75	Urban
60 – 110	75 – 130	Industrial
110 - 250	130 - 290	Heavily polluted

TABLE 8 Occurrence of Airborne Salinity according to ISO N53E

Deposition rate NaCl, mg.m ⁻² .day ⁻¹	Type of atmosphere
0 – 50	Clean, rural
	> 100 – 200 m from sea
50 – 100	Maritime
	> 200 – 300 m from sea
100 – 500	Marine, outside splash zone
500 – 1500	Splash zone

TABLE 9 Solubility of Reaction Product

Reaction product	g dissolved / 100 g.H ₂ O
FeCl ₂	64.4 (10°C)
FeSO ₄	Slightly soluble
Fe (NO ₃) ₂ .6H ₂ O	83.5(20°C)