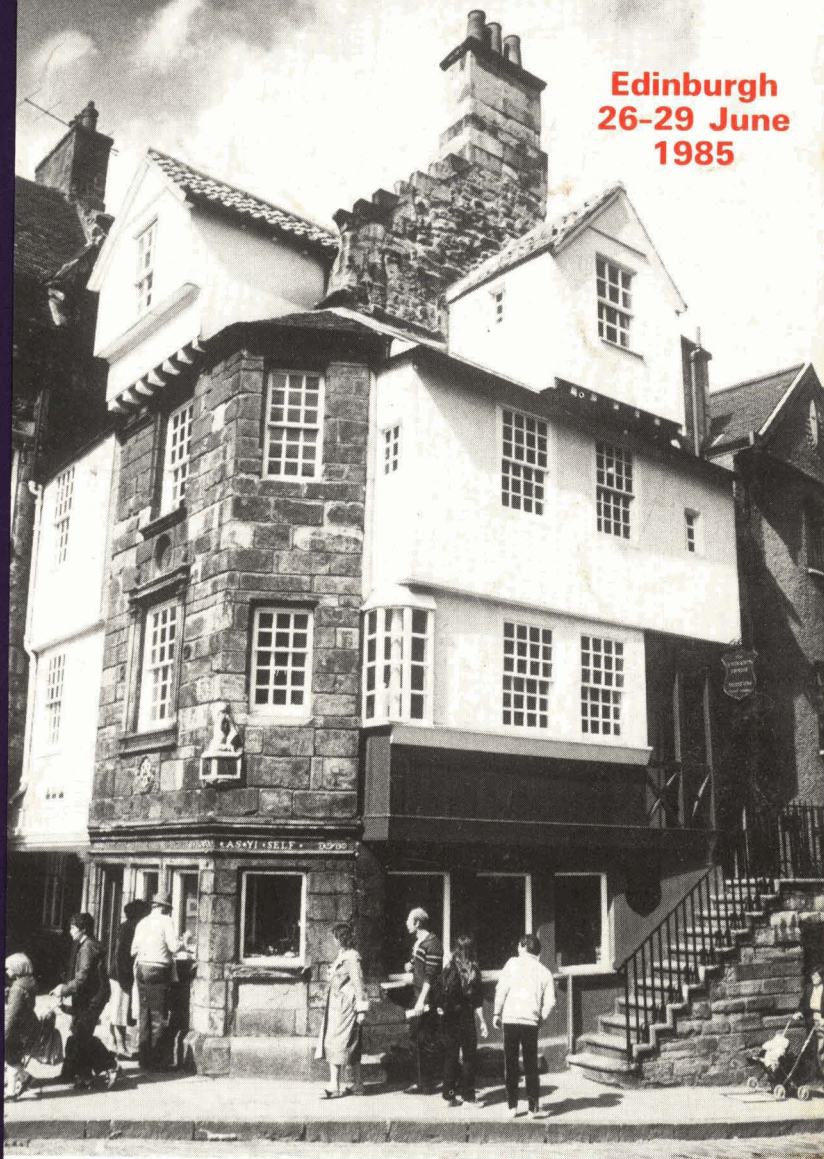


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- Coal tar and bitumen modified protective paints pigmented with aluminium flakes
- Comments on the galvanic action of zinc-rich paints
- Synthesis and study of water-soluble alkyd resins with high long chain fatty acid contents
- Synthesis of glyceryl monooleurate

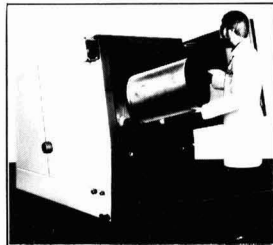
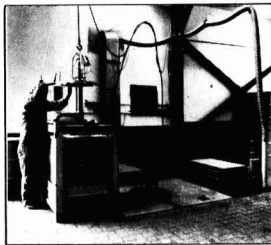
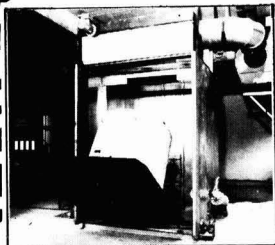
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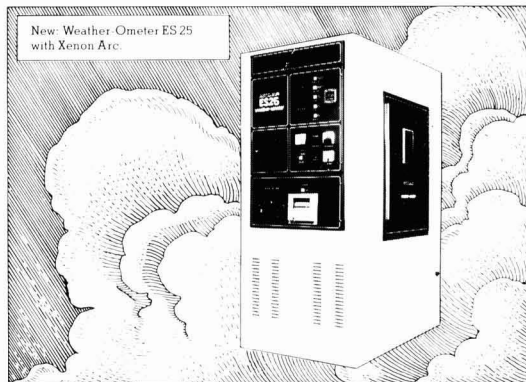
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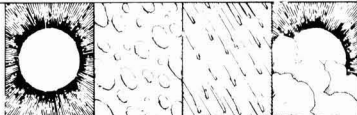
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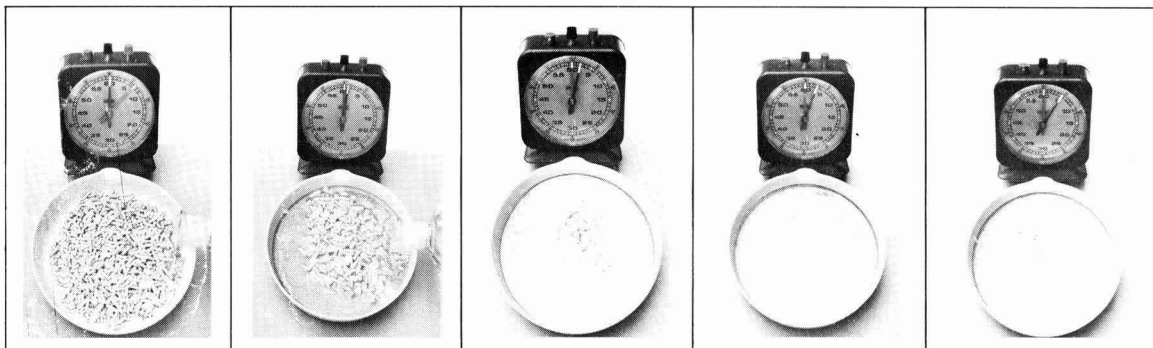
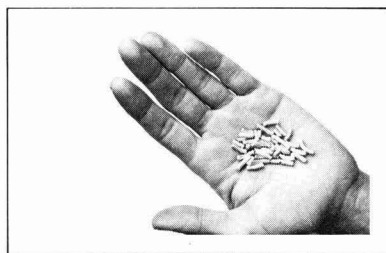
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Metal flake pigments: Laying the dust

I.R. Wheeler - Silberline Ltd.



Historically, metal flake pigments have been available as either dry powder or in solvent damped paste form.

Dry flake, being free of adjuvants which could cause incompatibility in applications systems, has the advantage of versatility. This physical form, however, suffers from several serious disadvantages. The most obvious is dusting, the slightest draught being sufficient to keep the particles airborne, a potent explosive hazard.

Silberline Limited has recognised the need for solvent free metal flakes, but has taken a policy decision not to manufacture or supply dry flake. Instead, the company has introduced a new granular form in which metal flake pigments, principally aluminium, are immobilised within a rigid binder. Sold under the trade name Silvet., the aluminium flake granules have a length of 5 - 15mm and a diameter of approximately 3mm. This size has been found convenient to handle. With a packaging density of at least 0.45, the granules are free flowing and virtually non-dusting. Tests have shown that only 0.1 - 0.2% of the original granular product is below 100 microns. As a typical metal flake may be in the range of 8 - 35 microns in diameter, the proportion of single flakes present is very small indeed.

The rigid binders of the Silvet granules have been selected to provide the widest compatibility in customers' applications systems.

Incorporation into ink

One of the main application areas for Silvet is packaging inks, particularly food packaging, where the barrier properties of the metal flake coupled with the freedom from residual solvent odour are important.

In preparing the ink, rapid dissolution of the Silvet binder releases the metal flake, which already being thoroughly wetted, disperses readily with the minimum of agitation. The use of low energy dispersion methods is critical in achieving the brightest possible finish. One of the main causes of poor results in metallic inks is the use of high energy mixers to disperse the flake.

It cannot be emphasised too strongly that the high shear incorporation of Silvet products is neither necessary nor desirable. The preferred method of incorporation is brief solvent contact, followed by let down with the premixed ink medium under gentle agitation. A slow speed

paddle stirrer is perfectly adequate provided that it is capable of moving the whole contents of the vessel.

Satisfactory results can also be achieved by adding Silvet granules directly to the ink medium under similar agitation, though time to reach complete dispersion may then be slightly longer.

The rapid dispersion of Silvet granules in the absence of stirring is shown. Isopropyl acetate is added to Sparkle Silvet 960-15-G over a 1-minute period. Breakdown on the granules starts at once, as shown in the first frame, taken after 6 seconds. By the fourth frame, taken at 3 minutes, release of the metal flake from the binder is virtually complete. Five minutes from the start, a North gauge demonstrates that a high degree of dispersion has been achieved.

Binders available

Silvet granules can be prepared from both leafing and non-leafing metal flakes, irrespective of flake size. Table 1 shows the granule binders currently available. Types G and J are recommended for printing inks based on non-leafing flake. Both have wide solvent and resin compat-

ibility, the zero acid value J-type is offered where the high acid value of the rosin derivative, G is unsuitable. Silvet granules for leafing printing inks are normally supplied with the coumarone-indene binder, Type M. Types G, H, J and M are all film formers, making a positive contribution to film integrity.

Where a specialised end use is envisaged, customers' preferred resins can generally be incorporated as a Silvet binder.

A number of less obvious advantages accrue from the use of Silvet in inks. Unlike pastes, Silvet does not dry out in storage. An opened container will therefore not deteriorate between one ink preparation and the next. The dispersibility of Silvet allows tinting corrections to be made easily by direct addition of Silvet to the ink, rather than by predispersion and addition of pastes or dry flake, with consequent wastage. Incorporation of organic pigments with non-leafing aluminium flake in Silvet products has been shown to be feasible, giving particularly attractive gold finishes. The availability and convenience of a standardised gold shade Silvet granule is expected to attract interest in small volume applications.

Binder types for Silvet granules

Type	Composition	Compatible Solvent	Compatible Resins/Polymers
G	Rosin derivative	Aromatic and aliphatic hydrocarbons, esters, ketones, alcohols, but not glycol and glycol ethers	All commonly used printing ink binders except NC and cellulose acetate
H	Low molecular weight polystyrene derivative	Aliphatic/aromatic hydrocarbons, esters, ketones, most plasticisers	PVC, SBR, polystyrene acrylics alkyds, rosins EC
J	Aldehyde resin	All common organic solvents except mineral spirits and chlorinated hydrocarbons	Acrylics, alkyds, cellulose (except EC), coumarone-indene
M	Coumarone-indene	Aliphatic and aromatic hydrocarbons	Cyclised rubbers, coumarone-indene resin mixtures for metallic paints and printing inks

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Contents

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Transactions and Communications

Coal tar and bitumen modified protective paints pigmented with aluminium flakes	29
<i>G. Cinti, A. Papo and G. Torriano</i>	
Comments on the galvanic action of zinc-rich paints	34
<i>S. A. Lindqvist, L. Mészáros and L. Svensson</i>	
Synthesis and study of water-soluble alkyd resins with high long chain fatty acid contents	40
<i>D. Caron, A. Fradet and E. Marechal</i>	
Synthesis of glyceryl monoalcuritate	46
<i>M. Mukherjee, R. N. Majee, S. Kumar and S. N. Mukherjee</i>	
<i>Next month's issue</i>	47
<i>OCCA meetings</i>	47
<i>News</i>	50
OCCA Conference 1985	53
<i>OCCA news</i>	56

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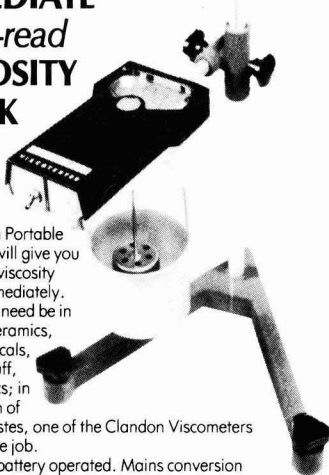
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Coal tar and bitumen modified protective paints pigmented with aluminium flakes

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Summary

Coal tar and bitumen modified protective paints, pigmented with aluminium flakes, are at present widely employed for the protection of steel surfaces in service conditions of high humidity and water immersion. The process of film formation is investigated for a number of paints of this class as far as solvent release from films of different thicknesses is concerned. The aim is to determine the effect of compositions on solvent retention; a gravimetric technique was employed. The resistance of the same paints against sagging is evaluated on the basis of rheological parameters. The equilibrium flow curves, structural build-up and break down kinetics are determined. The resistance to water immersion and salt fog exposure is investigated, and the role of the presence of aluminium pigment in enhancing the resistance is discussed.

Introduction

At present, metallic aluminium flake plays an important role in the paint field, where it is employed as a sole pigment or in conjunction with other pigments and extenders in the formulation of:

1. Protective paints – underwater and outdoor applications, high temperature, wood.
2. Decorative paints – metallic finishes, mainly for the decoration (and protection) of motor cars, hammer finishes, etc.
3. Functional paints – outdoor storage tanks containing volatile liquids, roofs, etc.

The number of scientific and technical papers which have appeared in the literature during recent years indicates the interest of paint manufacturers in metallic aluminium pigments of both the leafing and the non-leafing type.

Objective

The aim of the present paper is to communicate the results recently obtained from a series of investigations carried out on aluminium vinyl coal tar paints intended for the protection of steel surfaces in service in salt water immersion, splash zone and in marine environments generally.

Recent studies on metallic aluminium pigments and paints

A short review is given of the most significant recent papers on metallic aluminium pigments.

Mayborodoff¹ briefly describes the manufacturing processes of aluminium powders and pastes; the main properties of the aluminium flakes (covering area on water, and leafing ability); the differences between leafing and non-leafing types; and the principal types of aluminium pigmented paints. A detailed treatment of metallic paints, with relation to the aluminium pastes and colouring pigments employed in their formulation, is included. Subsequently, the binders used, the technology of preparation and application of metallic and hammer finishes

are dealt with. The “two tone” or “flop” optical property is discussed.

In a review of the various properties of metallic aluminium pigments², the following properties were taken into account: dimensions of the flakes, particle surface area and size distribution, leafing and non-leafing nature, vapour permeability of pigmented films, thermal conductivity, optical properties of films containing leafing and non-leafing types, tinting strength of non-leafing pigments, colour purity, reflectance, and flop.

The problems which arise if aluminium pigments are included in water-borne paints are discussed in an earlier paper³; attention is devoted to the wetting of the flakes and to the stabilization of the dispersed particles in the paint systems and in the pigment pastes.

King⁴ describes the new types of aluminium pigments with reference to particle size, coverage and reflectance. Technical data of powders and paints are reported in earlier works⁵; where the importance of the non-leafing types is emphasized. In another paper⁶ the advantages of the use of the non-leafing types in the formulation of printing inks are shown, and can be summarized as improved mechanical properties and enhanced corrosion resistance. The phenomenon of the orientation of aluminium pigments in automotive finishes is discussed⁷ in relation to the colour of the metallic base coat. The factors which govern the flake orientation are dealt with, with particular regard to paint viscosity which influences the mean inclination angle of the flakes. The inclination angle, however, is also affected by many other parameters; i.e. the main characteristics of pigments and binder and by the application procedure.

Flow in coatings and orientation in metallics are the subject of another work⁸. Types and causes of flow inside of a film are analysed; gravity and surface tension are recognised as first order causes; and differences of properties within the material as second order causes, e.g. surface variations, surface irregularities or viscosity differences caused by concentration differences. The particular feature of metallics of reflecting directionally incident light in directional illumination is discussed. Particle orientation is the main factor which governs the appearance of metallics and it is determined by the conditions in the film. Translation rotation and deformation of the volume elements are movements which take place in a viscous liquid; of these, rotation and deformation involve particle orientation. An examination of the movements connected with spraying follows with regard to the flow out of a drop on liquid film. The effect of the practical spray conditions are discussed with the aim of bridging the gap between the theoretical considerations previously developed and the practical experience. The possibilities of characterizing metallics are taken into account.

As far as the particular aspects of the protective power of aluminium paints are regarded, it is noted that aluminium

Table 1a

Paint composition	Paint No.				
	1	2	3	4	5
Vinyl copolymer*	30	32	34	37	41
Coal tar	70	68	66	63	59
Aluminium	+	+	+	+	+
PVC	11	17	20	24	30

*Vinyl chloride/vinyl acetate/vinyl alcohol copolymer

metallic pigments are often employed in the formulation of protective primers for steel as a sole pigment, as in some shop primers mainly applied in the field of container paints or for ship's bottoms, or together with red iron oxide and other pigments and extenders for all-purpose primers, ship's bottoms, etc. Mention is made⁹, that where an enamel is presented which is based on a conventional varnish and aluminium powder; this product is claimed to give excellent protective films on marine components when applied by spraying. The performance of a primer pigmented with aluminium flakes and zinc oxide is described¹⁰.

Aluminium flakes are employed here because they are able to enhance the protective power of traditional coating systems possibly because they lessen film permeability and so improve the barrier protective mechanism. The anticorrosive properties of the primer formulated with aluminium and zinc oxide are claimed to be equivalent to those of a red lead or of a zinc chromate primer. The application of an aluminium pigmented top coat strongly enhances the anticorrosive properties of the above mentioned primer.

Aluminium powder is employed in the formulation of powder coatings. The relevant production and application problems and the potentiality of these systems have been discussed¹¹.

Aluminium cladding coatings for structural and pipe corrosion protection have been discussed¹². Coatings comprise a cladding system based on ground aluminium powder and inorganic cement powder followed by the application of a polyamide resin finish.

In other papers some of the specifications concerning aluminium pigments and paints which were recently issued are mentioned¹³⁻¹⁶.

As far as the problem of the protection of ship's bottoms and boottopping is regarded, excellent results were obtained by the authors in a series of laboratory investigations on aluminium pigmented chlorinated rubber anticorrosive paints¹⁷. On the basis of these studies, a protective cycle could be formulated which is at present largely employed on the Italian market and is being adopted also by a number of European paint manufacturers.

A mechanism is suggested by Anderton *et al*^{18,20} to explain the action of aluminium metallic pigment in protective coatings destined for use in salt water immersion conditions, where it proves to be very useful particularly in presence of a cathodic protection systems.

According to Anderton, alkali ions penetrate under the film via discontinuities – causing the setting up of a water rich layer and due to osmosis at the interface the water content increases and reduces adhesion impairment. Thus, the penetration of oxygen is favoured as well as the cathodic reaction. Because of the strong affinity of aluminium for OH⁻, alkali ions move through the film towards sea water, so lessening the adhesion-impairing alkalinity. As a consequence of the osmotic effect, water concentrates around pigment particles, electrical contact

Table 1b

Paint composition	Paint No.							
	6	7	8	9	10	11	12	
Bitumen	66	58	50	58	58	58	58	
Coal tar	10	10	10	10	10	10	10	
Vinyl copolymer†	24	32	40	32	32	32	32	
Aluminium	+	+	+	+	+	–	–	
Micromica	–	–	–	–	–	+	–	
Micaceous iron oxide	–	–	–	–	–	–	+	
PVC	9	9	9	13	17	9	9	

†Vinyl chloride/vinyl isobutylether copolymer

sets up via the overlapping of hydrated layers, and OH⁻ ion transfer to the external solution is facilitated. Since the formation of OH⁻ ions takes place in the water at the metal-film interface, a dehydration reaction ensues. An alkali-dissolution of aluminium oxides also takes place; thus, bare metallic aluminium surfaces are made available, which act as oxygen scavengers. The rate of oxygen diffusion is reduced, oxygen is impeded from reaching the substrate and OH⁻ production lessens.

Experimental

Materials

Twenty-five aluminium-pigmented paints were formulated with the materials used described below:

Pigments and extenders

- Sixty-five per cent non-leafing (NL) aluminium paste, particle size 99 per cent >45 µm (DIN 53196).
- Micromica, wet ground mica, sp. gr. 2.8, oil absorption Gardner Coleman 60, particle size maximum 0.8 per cent >45 µm.
- Micaceous iron oxide, sp. gr. 4.8, oil absorption Gardner Coleman 20, particle size 1.5-3 per cent >74 µm, 5 per cent >63 µm.

Binders

1. Vinyl copolymer (A): vinyl chloride/vinyl acetate/vinyl alcohol copolymer 91/3/6, sp. gr. 1.39, viscosity 300 mPas at 25°C (20 per cent MIBK/Toluene 1/1 solution).
2. Vinyl copolymer (B): vinyl chloride/vinylisobutylether copolymer, chlorine content 43 ± 1 per cent, sp. gr. 1.24, viscosity 40:70 mPas at 20°C (20 per cent Toluene solution).
3. Coal tar, Special Pitch No. 5, sp. gr. 1.2, exylene content 12 per cent, viscosity 1.5 Pas at 25°C.
4. Bitumen, 75-85 pen.
5. Aerosil, amorphous silica, sp. gr. 2.3, thixotropic agent.

Paint compositions are given in tables 1a, 1b, 1c and 1d.

Testing methods

Film hardening

Film hardness increase was measured in order to obtain information on solvent retention over long periods of time, when gravimetric techniques are applicable. Hardness increase is accounted for by the release of the entrapped residued solvents which act as plasticizers.

Table 1c

Paint composition	Paint No.								
	13	14	15	16	17	18	19	20	
Coal tar (88%)	32.65	27.65	28.40	23.65	37.55	31.40			
Bitumen (60%)			6.25	5.20			46.25	39.85	
NL Aluminium paste (65%)	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	
Aerosil	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	
MIBK	3.85	3.85	1.85	3.65	1.05	3.10		1.65	
Vinyl copolymer* Sol.25%	50.00	55.00	54.00						
Vinyl copolymer† Sol.30%					47.90	52.00	40.25	45.00	
Ethylene glycol	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	
Vinyl copolymer* Sol.30%	30	36	30	36					
Vinyl copolymer† Sol.30%					30	36	30	36	
Coal tar	70	64	61	56	70	64			
Bitumen			9	8			70	64	
PVC	9.7	10.5	9.5	10.5	8.3	9.4	8.6	9.5	
Specific volume	0.96	0.97	0.97	0.98	0.93	0.93	1.00	0.99	
Specific gravity	1.04	1.03	1.03	1.02	1.08	1.07	1.00	1.01	
Yield (volume)	38	35	38	34	40	40	41	38	

*Vinyl chloride/vinyl acetate/vinyl alcohol copolymer
†Vinyl chloride/vinyl isobutylether copolymer

Table 1d

Paint composition	Paint No.				
	21	22	23	24	25
Coal tar (85%)	33.50	27.47	21.44	33.50	27.03
Aerosil	1.00	0.82	0.64	3.00	2.42
NL aluminium paste (65%)		18.00	36.00		17.71
Vinyl copolymer†	45.20	37.06	28.93	45.20	36.47
Xylene	19.80	12.24	12.67	17.80	15.97
Ethylene glycol	0.50	0.41	0.32	0.50	0.40
Vinyl copolymer†	36	36	36	36	36
Coal tar	64	64	64	64	64
Viscosity at 10 ⁴ s ⁻¹ (Pa)	0.19	0.24	0.26	0.23	0.25

†Vinyl chloride/vinyl isobutylether copolymer

Paints were applied on glass plates at 80-100 μm dry film thickness by means of a doctor blade manual applicator and hardness measurements according to DIN 53157 with a Persoz König pendulum were carried out at time intervals.

Hardness is reported in conventional units. In some instances a final hardness value was measured after stoving the samples at 80°C for 120 hours and reduced hardness data $H_t = H_i/H_f$ were given (H_t = reduced hardness, H_f = hardness at time t, after film laying down, H_f = final hardness).

Salt fog exposure

Mild steel panels, 150 mm \times 80 mm \times 3 mm, were employed. They were blasted to SA 3 (Swedish Standard SID 055900) with a G50 iron grit which provides a maximum height of the profile of about 82 μm .

Paints were applied at a 90-100 μm dry film thickness by air-spraying. Film thickness was controlled with an Elektrotest (Elektrophysik Köln, West Germany).

Painted panels were dried for 10 days at 18-20°C, relative humidity 50-60 per cent, then exposed to salt fog according to

ASTM B 117-64. Rusting and blistering were evaluated at time intervals according to ASTM D 610-68 and ASTM D 714-56, respectively. The results were noted as described in the Appendix.

Rheological properties

The rheological tests were performed with a Searle-type rotational viscometer (Rotovisko-Haake 3) using an MVI measuring device (0.956 mm clearance).

Shear rate range explored was 26.44 – 1694 s⁻¹.

The rheograms were registered by means of a X-t Laumann recorder.

The tests were carried out at 25 \pm 0.1°C.

Comments on the results

The results are reported in figures 1-9 and Table 2.

Figure 1

It gives an indication of the fact that the various paints will attain various final hardness values as could be expected on the basis of compositions in which the ratio resin/coal-tar is varied from 30:70 to 41:59, and the PVC from 11 to 30. The most interesting fact is that 51 days after application hardness still increases, which means that the film still contains appreciable amounts of entrapped solvent.

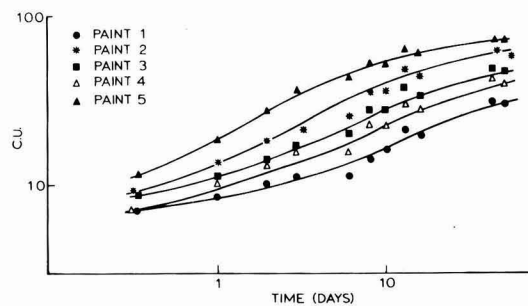


Figure 1. Film hardening (paints 1-5; 400 μm thickness applicator)

Figure 2

The entrapping phenomenon shown in Figure 1, was further investigated for films of higher thickness, which reproduces the usual practical conditions. Three of the paints previously tested were applied at a total thickness of 250-300 μm by 3 coats laid down within 24 hours. Hardening was followed for 460 days. Tests are still taking place. Entrapping phenomena are relevant.

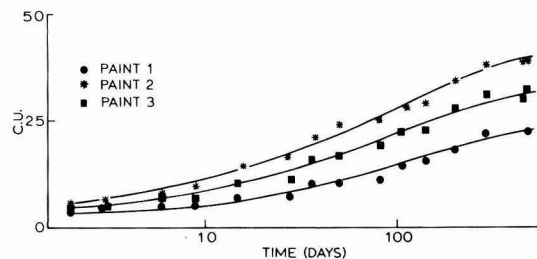


Figure 2. Film hardening (paints 1-3; 1200 μm thickness applicator)

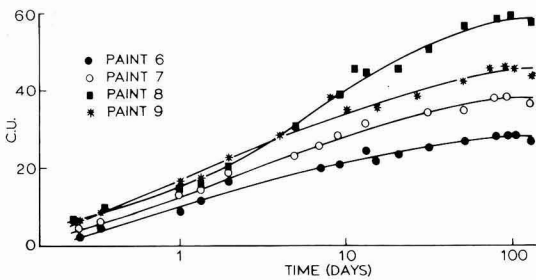


Figure 3(a). Film hardening (paints 6-9; 400 μm thickness applicator)

Figure 3(a)

This shows the hardening of a series of 4 paints formulated with another vinyl resin at various vinyl/coal tar ratios which contain also a fixed quantity of bitumen. For a vinyl/coal tar/bitumen ratio a PVC variation was made. After 135 days hardening is almost complete for 80-100 dry film thicknesses. As expected, there is a strong effect due to the resin/coal tar/bitumen ratio on final hardness (paints 6, 7 and 8). PVC variation (paints 7 and 9), also influences hardness.

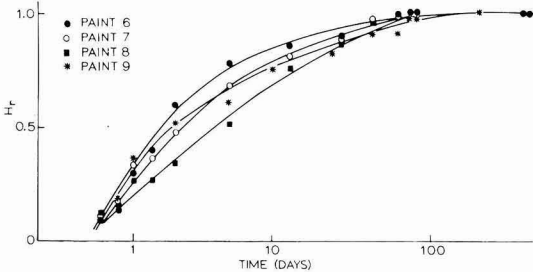


Figure 3(b). Reduced hardness versus time plot (paints 6-9; 400 μm applicator)

Figure 3(b)

In order to obtain a comparison of the kinetics of solvent release Figure 3(b) was drawn. It is shown that a faster solvent release is obtained with a paint of lower hardness, which means that the presence of higher amounts of resin acts unfavourably on solvent retention. No effect of the higher PVC is noticed. Possibly, there are two opposite effects which counterbalance with one another: on one hand the higher amount of pigment which makes the film more open; on the other hand the lamellar shape of the pigment which contributes to increasing insulation.

Figures 4 and 5

These show that the substitution of the aluminium for a lamellar pigment (micaceous iron oxide) or a lamellar extender (micromica) is not satisfactory. The beneficial effect of the aluminium pigment is also shown if paints 9 and 10 are compared with Paint 7. These observations apply to both rusting and blistering. The protective power of paints 9 and 10 is very satisfactory.

Figures 6 and 7

They show that vinyl copolymer*(A), behaves better than vinyl copolymer†(B), in this context. The presence of a small amount of bitumen together with coal tar seems to improve the performance of the vinyl copolymer*(A)/coal tar compositions.

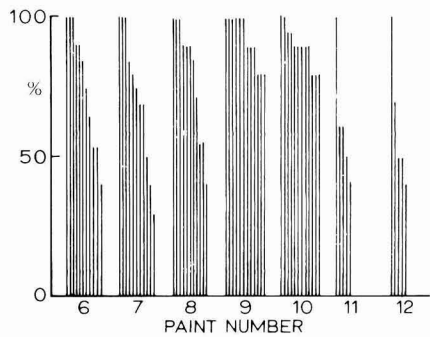


Figure 4. Salt fog exposure-Rusting. Paints 6-12; evaluation after: 500 hours-1st (vertical line), 850 hours-2nd, 1100 hours-3rd, 1400 hours-4th, 1700 hours-5th, 2000 hours-6th, 2300 hours-7th, 2600 hours-8th, 3000 hours-9th, 3600 hours-10th, 4300 hours-11th

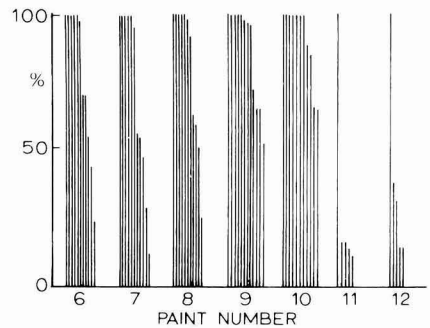


Figure 5. Salt fog exposure-Blistering. Paints 6-12; evaluation after: 500 hours-1st (vertical line), 850 hours-2nd, 1100 hours-3rd, 1400 hours-4th, 1700 hours-5th, 2000 hours-6th, 2300 hours-7th, 2600 hours-8th, 3000 hours-9th, 3600 hours-10th, 4300 hours-11th

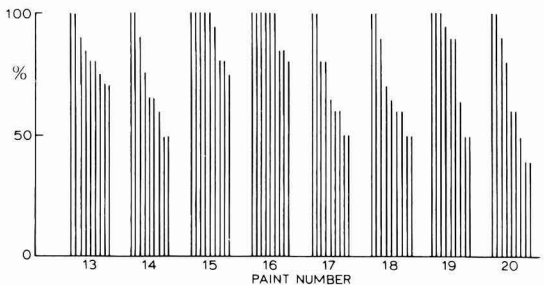


Figure 6. Salt fog exposure-Rusting. Paints 13-20; evaluation after: 200 hours-1st (vertical line), 500 hours-2nd, 900 hours-3rd, 1350 hours-4th, 1400 hours-5th, 2000 hours-6th, 2300 hours-7th, 2700 hours-8th

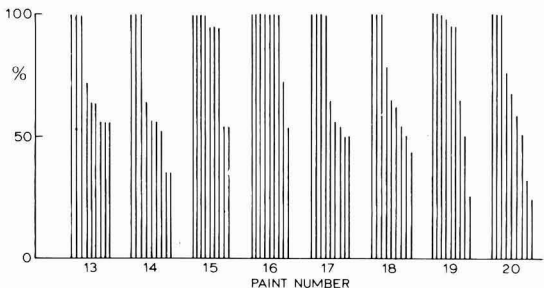


Figure 7. Salt fog exposure-Blistering. Paints 13-20; evaluation after: 200 hours-1st (vertical line), 500 hours-2nd, 900 hours-3rd, 1350 hours-4th, 1500 hours-5th, 2000 hours-6th, 2300 hours-7th, 2700 hours-8th

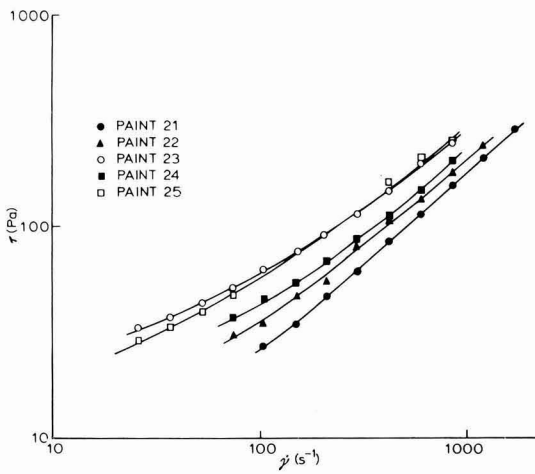


Figure 8. Shear stress versus shear rate plot (paints 21-25)

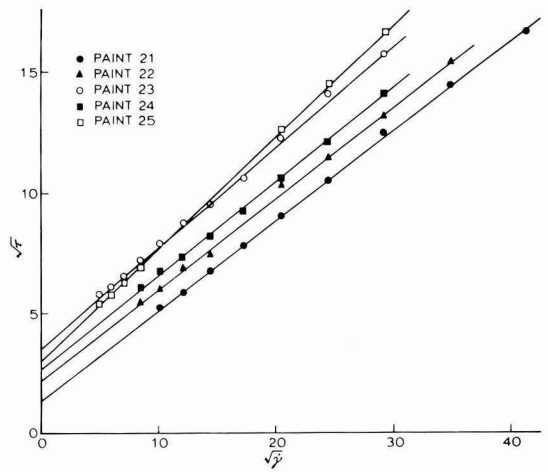


Figure 9. Square root of shear stress versus square root of shear rate plot (paints 21-25)

Figures 8 and 9, and Table 2

All the paints rheologically tested (paints 21-25) exhibited a thixotropic flow behaviour more marked for paints 24 and 25 which contain a higher amount of Aerosil. Equilibrium shear stress and shear rate data were correlated with the Casson equation

$$\tau^{1/2} = \tau_0^{1/2} + \eta_1^{1/2} \dot{\gamma}^{1/2}$$

where τ_0 is the yield stress and η_1 is the apparent viscosity at infinite shear rate. It can be noticed that τ_0 increases with increasing aluminium content; η_1 also increases with increasing aluminium content, and a marked increase is obtained with a combined higher content of aluminium and thixotropic agent.

Conclusion

Vinyl coal tar and bitumen aluminium paints showed excellent resistance to salt fog exposure and satisfactory characteristics as far as application and film formation are concerned.

Investigations on these paints are still in progress, in particular the resistance to salt water immersion in the presence of a cathodic protection system is being taken into account, as well as the determination of solvent release during the wet stage.

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Appendix

Assessment of film degradation

Blistering quantification

A quality criterion was employed. Accordingly, the following numbers were assigned to blister density:

No blister	= 10
Very few	= 9
Few	= 8
Few-medium	= 7
Medium	= 6
Medium-medium dense	= 5
Medium-dense	= 4
Dense	= 2
Destroyed	= 0

Table 2

Parameters of the Casson equation

Paint No.	τ_0 (Pa)	η_1 (Pas)
21	1.626	0.143
22	4.215	0.148
23	12.08	0.179
24	6.477	0.154
25	8.988	0.205

Blistering degradation was evaluated according to the expression:

$$\text{Protective power} = \text{density number} \times \text{size number}$$

where the size number is that given in ASTM D 714.

Rusting assessment

This was obtained by multiplying by ten the rust value determined according to ASTM D 610-43.

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Comments on the galvanic action of zinc-rich paints

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Summary

Cathodic protection of steel at voids in zinc-rich-paint coats and in hot-dipped-zinc coats, both with and without conventional paints, has been studied. Two commercial zinc-rich paints, one epoxy-based and one ethyl-silicate-based, were used. The coated steel panels (1.2, 3.4, 6.8, and 10 mm thick), all had one unprotected edge which acted as void. They were each exposed to a solution of 0.1 M sodium chloride for a maximum of 12 months.

The hot-dipped, galvanized steel gave cathodic protection that was a minimum of three times longer than that of the best zinc-rich paint tested.

The cathodic protection of the ethyl-silicate-based paint was superior to that of the epoxy-based paint.

Top coating both zinc-rich paints and galvanized steel with conventional paint drastically reduced the duration of the cathodic protection at the void.

SEM and WDS analysis was performed on cross sections made perpendicular to the exposed paint surface in order to get concentration profiles of Zn, Fe, Cl, Si, and O in the paints. A buildup of chloride was found both at the outer paint surface and at the steel-paint interface.

There is a positive correlation between the duration of cathodic protection from immersion tests such as the one described in this paper and that from previous electrochemical investigations of the same paints.

The duration of cathodic protection afforded by the zinc-rich paints was much shorter than that of the corrosion protection afforded by an intact, zinc-rich paint coat. In other words, cathodic protection is more than just the single, corrosion-inhibiting mechanism provided by the zinc in zinc-rich paints.

Introduction

The cathodic protection afforded to steel by metallic zinc, and the importance of this protection in corrosion protection, were pointed out by H. Davy at the beginning of the last century. Metallic zinc particles have been used as a corrosion-inhibiting pigment in paints for more than 80 years¹. When used in paints with a high pigment-weight concentration (PWC), ($\approx > 80$ per cent), the zinc particles were thought to give long-lasting cathodic protection to a steel substrate in a manner similar to that found in hot-dipped galvanized steel to take but one example.

The basis of the cathodic protection by a zinc-rich paint (ZRP) of a steel substrate is the electrical conductivity between the zinc particles in the paint film, and between the zinc particles and the steel substrate.

A decrease in the cathodic protective property of ZRP by

exposure to salt solutions has been studied using the techniques of AC resistance, impedance, potential, and polarization^{2,4}. The reason for this decrease is simply that the zinc particles in a ZRP used for steel protection will, in a corrosive environment:

- Oxidize as a result of the cathodic protection afforded to the steel substrate by the zinc;
- Corrode spontaneously not only on the paint surface but also within the paint film, as a result of the porous structure of the highly pigmented ZRP film.

As the zinc particles become covered with corrosion products, the particles will be electrically isolated both from each other and from the substrate, leading to the elimination of the corrosion protection by cathodic protection. But in spite of this, ZRP's are successfully used today for the corrosion protection of steel under different conditions of exposure.

The present investigation aims to:

1. Determine if the corrosion protection afforded by ZRP's lasts longer than the cathodic protection afforded by the zinc, by studying immersed ZRP-coated steel panels with one unprotected edge.
2. Compare the results of objective (1) with results obtained from the same ZRP by polarization, potential, impedance, and AC-resistance measurements.
3. Identify the zinc-corrosion products by X-ray analysis.
4. Determine the distribution of elements in ZRP films as found in cross sections made perpendicular to the exposed paint surface, using SEM and WDS analysis.

Experimental

The specimens were steel panels coated with either zinc-rich paints or hot-dipped zinc, either with or without a conventional topcoat. Steel panels having only a conventional topcoat served as experimental controls. One edge of each panel was left unprotected.

The experiment consisted of a simple exposure of (a) painted steel panels and (b) hot-dipped, galvanized steel panels in a bath of 0.1 M sodium-chloride solution at room temperature. The painted panels were exposed for 6 months and the hot-dipped, galvanized panels for 12 months. The electrolyte was replaced twice a week during the first six weeks and then at 14-day intervals.

The panels were placed along the circumference of a poly-ethylene vessel with the unprotected edge upward. The distance

between the air-electrolyte surface and the unprotected edge of the panels was 5 cm.

During exposure the first visible sign of growing steel corrosion on the unprotected edges was noted, and the spread of iron-corrosion products over the unprotected edges was visually noted during the 45 first days.

The panels were lifted out of the baths after exposure periods of seven days and one and a half, three and six months. The zinc-corrosion products on the ZRP surfaces were scraped off and analysed by X-ray diffraction. The condition of the paint surface and of cross sections made perpendicular to the exposed surface were registered by SEM. Quantitative WDS analysis for Zn, Fe, Cl, Na, Si and O, was performed in the surface of the cross sections taken perpendicular to the exposed surface to obtain concentration profiles for the ZRP lacking a conventional top coat after periods of exposure to a 0.1 M sodium-chloride solution of 0, 1½, 3, and 6 months. The area analyzed was 20 µm high and 1.5 µm wide, and the ZRP film was analyzed at points 1.5 µm apart. In other words, in a 100 µm thick sample of paint film, 30 discrete areas were analyzed with respect to the elements mentioned above. The quantitative WDS-analysis results were normalized for the elements investigated.

Panel preparation

Both sides of the panels were painted and all the samples were prepared in duplicate.

Panel material

Cold-rolled (SIS 1142) and hot-rolled (SIS 1312) steel plate of the following thicknesses: 1, 2, and 3 mm (cold-rolled); 4, 6, 8, and 10 mm (hot-rolled).

Panel dimensions

60 × 120 mm, with thicknesses of 1, 2, 3, 4, 6, 8, and 10 mm;

Pre-treatment

Panels for hot-dip galvanizing were alkali-cleaned on a production line in a factory. The remaining panels were blasted to Sa 2½ with Al₂O₃ after cleaning in xylene.

Hot-dip galvanizing

The panels were hot-dip galvanized on a production line in a factory, giving a zinc-coating thickness of 65-220 µm.

Painting with ZRP

Two commercial ZRPs were used, one based on epoxy (91 per cent PWC) and one on ethyl-silicate (93 per cent PWC) binder. They were air-sprayed onto the blasted panels giving a dry-film thickness of 70 to 150 µm and 80 to 100 µm, respectively. The ethyl-silicate-based ZRP was cured for two days before top coating.

Top coating with conventional paint

Zinc-rich-painted, hot-dip-galvanized and blasted panels were top coated with a conventional titanium-dioxide-pigmented, epoxy primer by air-spraying to a dry-film thickness of 90 to 120 µm of the epoxy primer. The hot-dip galvanized steel panels were cleaned in xylene before painting.

Edge preparation

All corners and edges were covered with 100 per cent epoxy resin, except one, a 60 mm long edge, which was abraded with carborundum emery paper, 340-mesh, to produce clean steel.

The painted panels were allowed to dry for two weeks before they were used in the immersion tests.

Results and discussion

Figure 1, shows the time required for first visible signs of growing steel corrosion to appear at the unprotected edges of the plates of thicknesses of 1, 2, 3, 4, 6, 8, and 10 mm. These signs are interpreted as proof of the termination of cathodic protection. The following coating systems were investigated:

1. Hot-dipped, galvanized steel with and without conventional paint.
2. Epoxy-based ZRP with and without conventional paint.
3. Ethyl-silicate ZRP with and without conventional paint.
4. Conventional paint only.

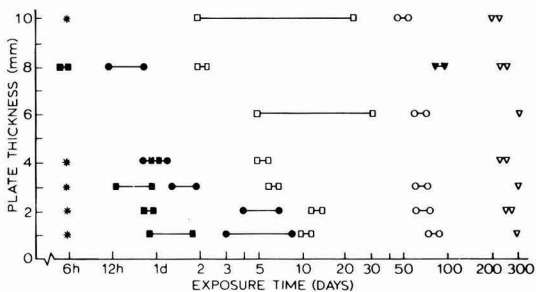


Figure 1. First visible signs of growing steel corrosion at the unprotected edges of plates of different thicknesses as a function of time of exposure to a 0.1 M sodium-chloride solution. Symbols: ★ Conventional paint only; ■ Epoxy-based ZRP with conventional paint; □ Ethyl-silicate ZRP only; ● Ethyl-silicate ZRP with conventional paint; ○ Ethyl-silicate ZRP only; ▼ Hot-dipped galvanizing with conventional paint; ▽ Hot-dipped galvanizing only

The following figures show the areas covered by steel-corrosion products on the unprotected edges of plates treated with top-coated, epoxy-based ZRP (Figure 2), top-coated, ethyl-silicate ZRP (Figure 3), and only conventional paint (Figure 4), during 45 days of exposure to a 0.1 M sodium-chloride solution. The bottom row shows the truly corroded area after the removal of the corrosion products after 45 days of exposure.

SEM micrographs and element analyses (concentration profiles) of cross sections made perpendicular to the exposed paint surfaces after exposures of 0, 1½, 3, and 6 months of the panels to a solution of 0.1 M sodium chloride are shown in figures 5 and 6 respectively for the epoxy-based and ethyl-silicate-based ZRPs.

Figure 1 shows that the unprotected edge of the plates having only a conventional paint coating corrode within six hours, irrespective of plate thickness. For the ZRP panels, the unprotected edges of the top coated panels corroded more rapidly than those lacking a top coat. The cathodic protection afforded by the ethyl-silicate-based ZRP was superior to that of the epoxy-based ZRP. From figures 1-4 it is evident that, by top coating a ZRP, cathodic protection is reduced drastically at a void in the paint film down to the steel, as the exposed area ratio ZRP/steel becomes very small. The exposed ZRP area is the one corresponding to the film thickness (x) in Figure 7.

Figure 2. Areas covered by steel-corrosion products on the unprotected edges of top-coated, epoxy, zinc-rich painted steel panels of different thicknesses as a function of exposure time in a 0.1 M, sodium-chloride solution. The bottom row shows the truly corroded area after the removal of corrosion products after 45 days of exposure. The 4 mm panels were not examined, as these were used for both SEM and WDS analyses

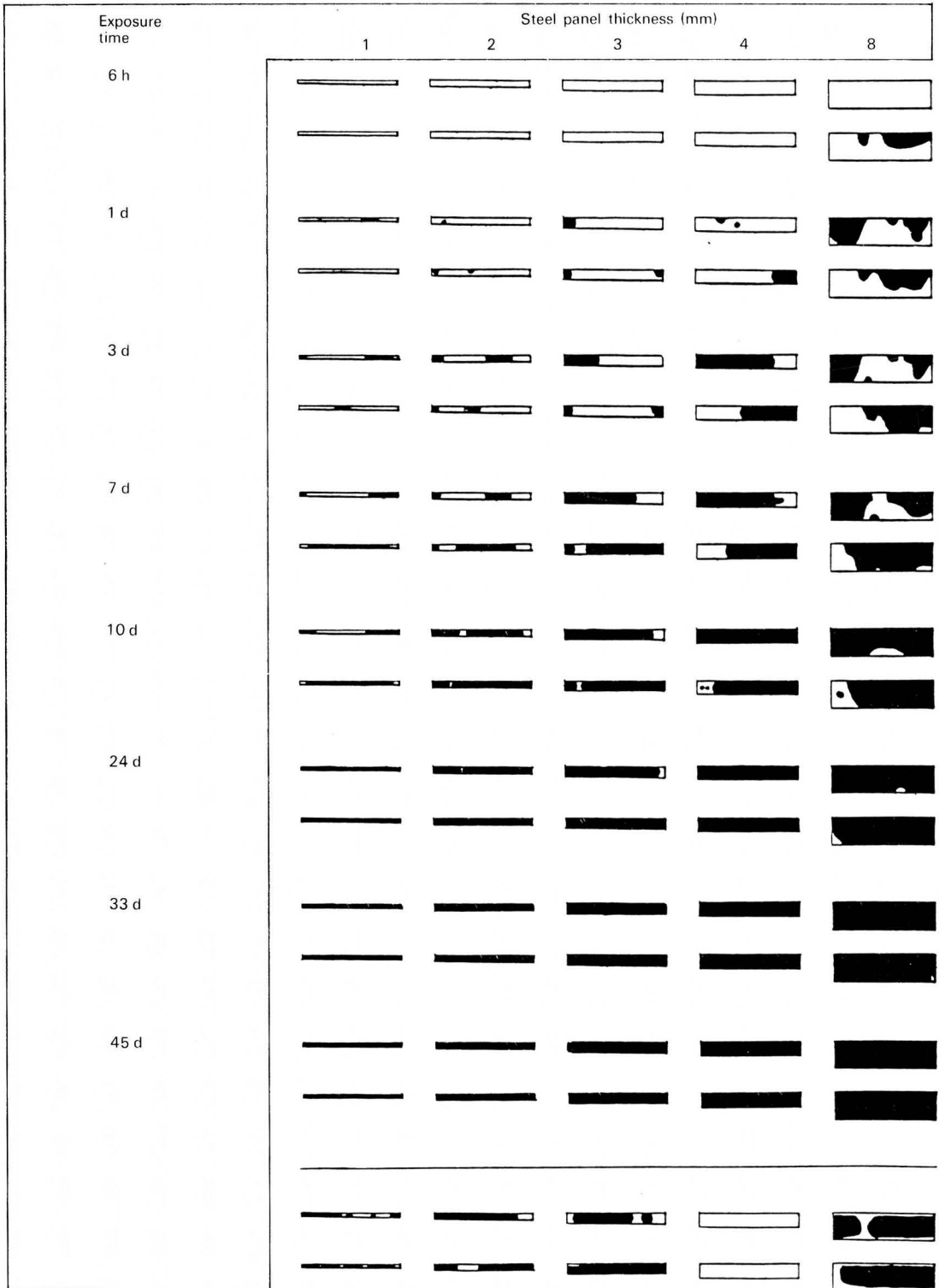


Figure 3. Areas covered by steel-corrosion products on the unprotected edges of topcoated, ethyl-silicate, zinc-rich-painted steel panels of different thicknesses as a function of exposure time in a 0.1 M, sodium-chloride solution. The bottom row shows the truly corroded area after the removal of corrosion products after 45 days of exposure. The 4 mm panels were not examined, as these were used for both SEM and WDS analyses

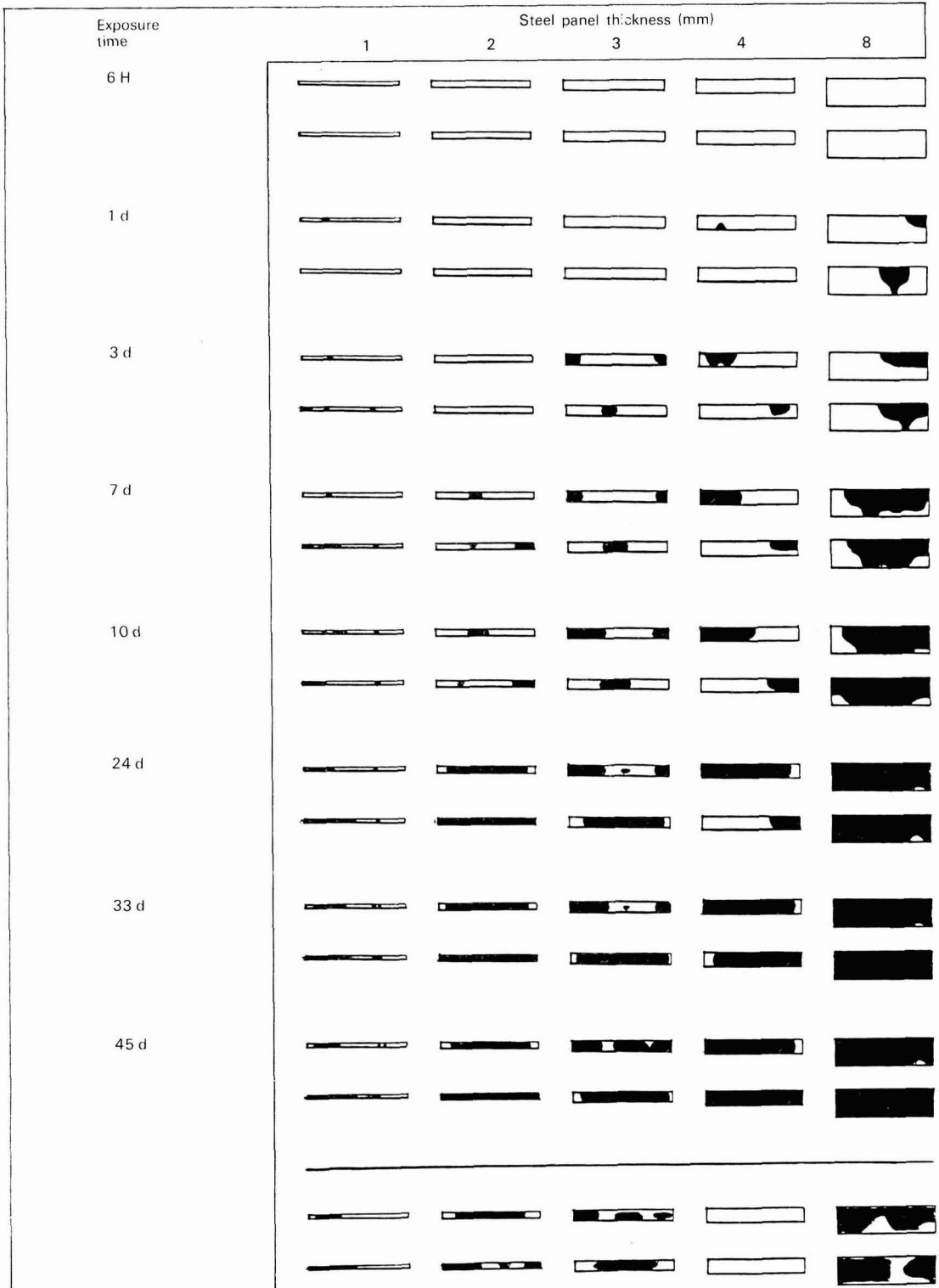
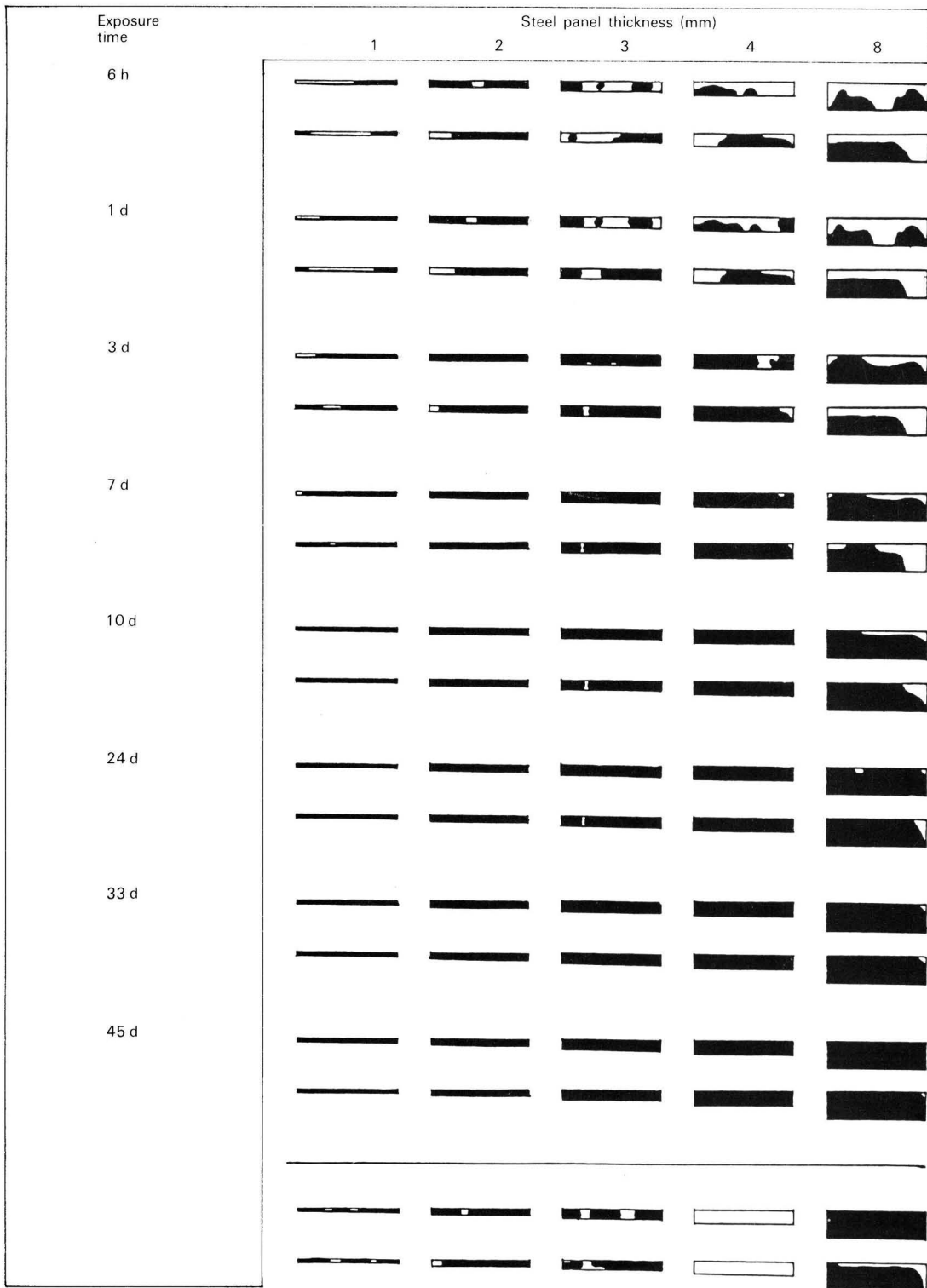
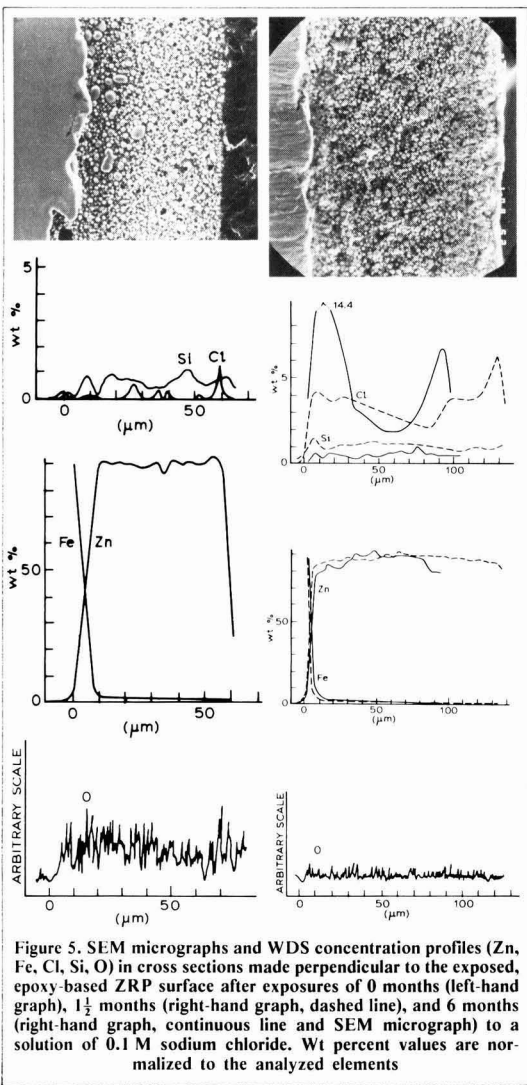


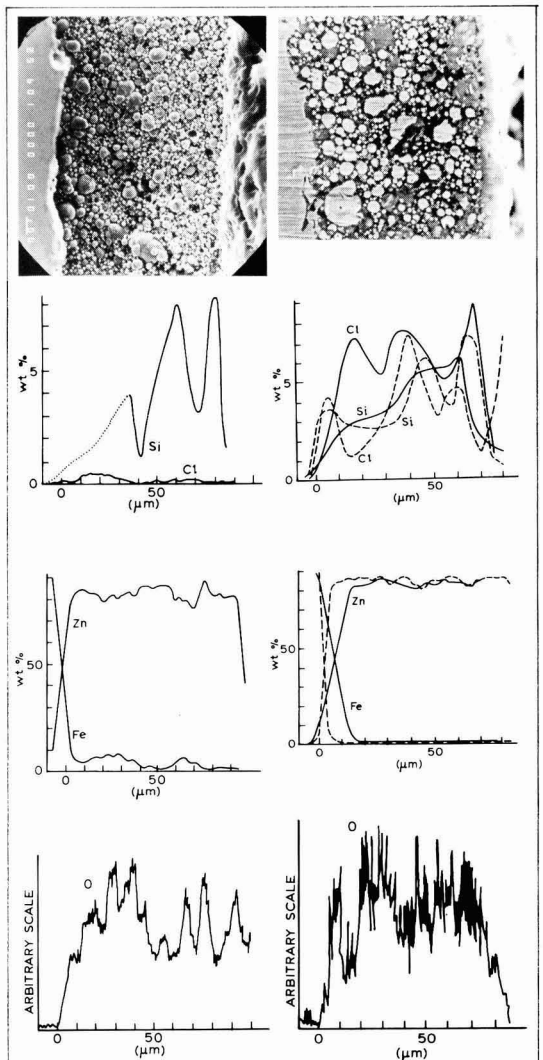
Figure 4. Areas covered by steel-corrosion products on the unprotected edges of topcoated, non-ZRP-primed, steel panels of different thicknesses as a function of exposure time in a 0.1 M, sodium-chloride solution. The bottom row shows the truly corroded area after the removal of corrosion products after 45 days of exposure. The 4 mm panels were not examined, as these were used for both SEM and WDS analyses





In earlier investigations^{2,4}, the cathodic protection obtained in ten commercial ZRP's has been investigated by techniques of polarization, potential, impedance, and AC resistance. The duration of the cathodic protection of the ZRP as found by these methods was shorter than the general corrosion protection given by these paints in practical applications. Therefore a practical bath test was performed as described in this paper to compare the duration of the cathodic protection of the uncoated edge with the duration of the corrosion protection of the painted surfaces. Results pertaining to the duration of cathodic protection from this bath exposure correlate positively with those obtained in tests in which electrochemical methods were used. The polarization technique in particular gave unambiguous results when cathodic protection had completely ceased.

It is quite clear, however, that even the uncoated, intact ZRP provides corrosion protection to a steel substrate for a much longer time than that corresponding to the time of active cathodic protection afforded by the zinc. Panels in which the cathodic protection of the unprotected edge had disappeared within two months (which is also shown in electrochemical measurements^{2,4}), had painted areas that were in good con-



dition even after six months of exposure. Corrosion-inhibiting mechanisms of ZRP other than cathodic protection have been summarized in an earlier paper⁴.

The duration of the cathodic protection of the unprotected edges of the hot-dipped, galvanized steel panels without paint was a minimum of three times longer than that of the best ZRP tested. The difference between panels with, and without topcoats, would have been more pronounced due to lower water permeability if a conventional topcoat with low pigment-volume concentration instead of an epoxy primer had been used.

From figures 2-4 is evident that the true corrosion of the unprotected edge is unevenly distributed; some areas of the steel are not corroded at all. The cause of this is simply the presence of separated anodic and cathodic areas. This agrees with

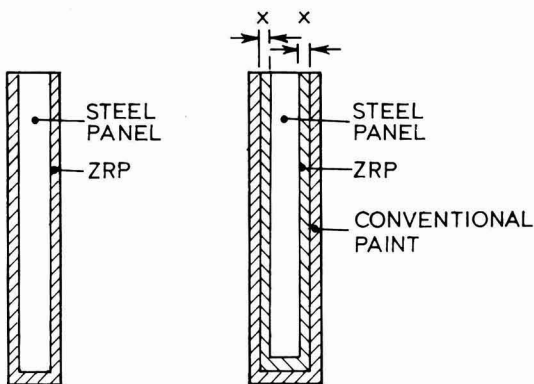


Figure 7. Schematic view of cross sections of a ZRP-painted panel (left) and a ZRP-painted and top-coated panel (right) with one unprotected edge. x = ZRP-film thickness

previous findings of pitting in scribed lines in conventional paint systems on steel in immersed conditions⁵.

X-ray diffraction analysis of the zinc-corrosion products found on ZRP and on hot-dipped, galvanized areas after 45 days and 6 months of exposure to a solution of 0.1 M sodium chloride showed the corrosion products to be mixtures of several zinc compounds, $Zn_4CO_3(OH)_3 \cdot H_2O$ and $Zn_4(CO_3)_2(OH)_5$, but although present, no chloride-containing compounds were identified.

The sample amounts in the cross sections are too small for phase analysis, but the concentration profiles for Zn, Fe, Cl, Si, and O are shown in figures 5 and 6 for the epoxy-based and the ethyl-silicate-based ZRP's respectively, after 0, 1½, and 6 months of exposure. The quantitative results were normalized to the elements analyzed. The accuracy of the WDS analysis is limited by difficulties in sample preparation and by the fact that the chemical formulas of the compounds formed are unknown and thus precluded the performance of correcting calculations. Uncertain results arising from difficulties in sample preparation account for the dashed curve for Si in the unexposed specimen in the region close to the steel surface in Figure 6. The chloride profiles show two or more maxima in the ZRP film; these are present at the profiles for 1½, 3, and 6 months. The maximum at the outer paint surface is due to the

corrosion of the ZRP surface, producing chloride-containing corrosion products. The maximum at the steel/ZRP interface seems to increase with exposure time, but there is no corresponding increase of iron in the ZRP film; i.e. the chloride is bound as zinc-corrosion products at the steel surface.

Conclusions

The conclusions from this investigation (which are valid for immersed subjects), can be summarized as follows:

1. At voids in the coat, the cathodic protection afforded by zinc-rich paints lasts much less time than does that afforded by hot-dip zinc.
2. Ethyl-silicate-based, zinc-rich paints give superior cathodic protection compared to epoxy-based ones.
3. Top coating zinc-rich paints or galvanized steel drastically reduces the cathodic protection at voids which extend to the level of the steel.
4. There is a positive correlation between the duration of cathodic protection from immersion tests such as the one described in this paper and that from previous electrochemical investigations of the same paints.
5. Using WDS, chloride accumulation was found both at the outer surface of the zinc-rich paint and at the steel/zinc-rich paint interface.

Acknowledgement

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Synthesis and study of water-soluble alkyd resins with high long chain fatty acid contents

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Summary

In view of their use as high gloss architectural coatings, water-soluble long oil (>60 per cent) alkyd resins were obtained by the introduction of ionic groups on the chains. Trialkylammonium carboxylates gave better compounds than sulfonates. Several means of synthesis were investigated: acrylic acid grafting or maleic anhydride reaction with unsaturated groups of a preformed resin were not successful. The introduction of 2,2-bishydroxy-methylpropanoic acid (DMPA), trimellitic anhydride (TMA) or pyromellitic dianhydride (PMDA) in

the alkyd resin reaction mixture led to compounds giving clear aqueous solutions of low viscosity after neutralization of residual carboxyl groups by tertiary amines. The influence of average viscosity, carboxyl group content, nature and amount of cosolvent and of neutralizing amine is discussed.

Introduction

Water soluble polymers have been the object of a steadily

increasing interest as binders for the coating industry over the years and they are destined to play a leading role in the near future^{1,5}.

However, most of these polymers including polyurethanes or polyacrylates, are only water dispersible^{6,7} and therefore cannot be used when a high gloss is required. This also holds true for the water dilutable alkyd resins described in a number of patents^{8,15}. When solutions to the problems are claimed, some points remain unsatisfactory, leading to limitations in their possible uses, mainly:

- Low air drying rates¹⁶
- The necessity for high-temperature curing^{17,19}
- The necessity for high cosolvent content^{16,18,20}

The aim of the present work is to study the solubilisation of alkyd resins containing more than 60 per cent wt of oil (long-oil alkyds) in aqueous media with low organic cosolvent content. The resulting solutions which must have a low viscosity, will be used for architectural gloss finishes.

Bibliographic study and preliminary experiments

Aqueous solubilisation of organic polymers is possible when their chains bear a large number of hydrophilic groups. Some patents concern the incorporation of hydrophilic polymers, such as polyoxyethylenes, in the alkyd resin backbone^{11,13}; however the hydrophilic nature of these groups is not sufficient to solubilize resins with a medium to a high oil content in water; and lead to the formation of emulsions or dispersions rather than to the formation of solutions. The most powerful solubilizing agents are inorganic or organic salts: metal sulfonates or trialkylammonium carboxylates.

Alkyd resin solubilisation by sulfonate groups

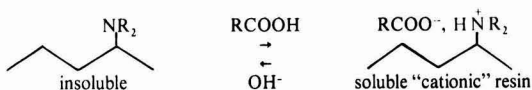
Some water-soluble polyesters possessing sulfonate groups are described in the literature.

Some authors reacted the usual precursors of polyesters with sulfonated monomers such as 2-sodio-sulfonatobutanedioic acid (A) dimethylester²¹ or 5-sodio-sulfonato 1,3-benzenedicarboxylic acid (B) dimethyl ester^{22,24}. Moiseenko *et al.*²⁵ described a two step process, a HSO₃ group of hydrogensulfite is added to the maleate double bonds of a preformed polyester. However, the resulting mixtures are probably very complex, and we did not use this method.

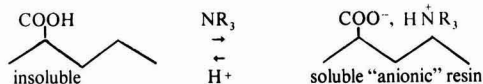
On the other hand an attempt was made to carry out the synthesis of water-soluble alkyd resins by the reaction of tall oil fatty acids, isophthalic or phthalic acids, with A or B dimethylesters or bishydroxyethylesters, in all cases the synthesis failed badly, two phases of products being obtained. From sulfur titration and IR, ¹³C and ¹H NMR determinations it appeared that one phase contained sulfonate groups and no alkyd chains, the other alkyd chains, but no sulfonates. This was considered as being due to the fact that owing to their incompatibility, monomers react with pentaerythritol in two separate phases, resulting in two different products. In order to solve this problem of incompatibility, we used sulfonated fatty acids (C) as the sulfonate carrier; and the synthesis of an alkyd resin (R1) using acids C, pentaerythritol and phthalic anhydride was successful. R1 is soluble in a 70:30 mixture of water and 2-butoxyethanol. A varnish prepared from this solution dried completely in six hours but the resulting film was cloudy and flaked off. Since a film obtained from a toluene solution of R1 was satisfactory these defects could be due to the formation of high salt content areas incompatible with hydrophobic alkyd chains during the evaporation of water. It was therefore concluded that water solubilisation of alkyd resins by introduction of sulfonate groups in their chains had few advantages.

Alkyd resins solubilisation by trialkylammonium carboxylate groups

Depending on the nature of the functional groups attached to the resin backbone, solubilization can be obtained either in acid medium:



Or in a basic medium:



Cationic resins are mainly used for electrodeposited coatings. In the present work it was decided to prepare an anionic resin; some compounds of this type are described in various patents but, due to their short oil lengths, they must be oven-baked and cannot be used as architectural binders.

The introduction of carboxylic functions in a resin can be carried out by three main methods:

1. Use of the unsaturated groups in the resins.
2. Polycondensation of an acid bearing monomer during the formation of the chain.
3. Reaction of an anhydride with the hydroxy groups of pre-formed chains.

1. Use of the unsaturated groups in the resins

Some unsaturated oils have been water-solubilized after Diels Alder reaction of maleic anhydride with their conjugated double bonds²⁷; this reaction is carried out at 80°C. When no (or insufficient) conjugated double bond amounts are available maleic anhydride can react with the carbon in the α position of non conjugated double bonds, however this technique requires higher temperatures (200°C). This is the case for resins prepared from tall oil fatty acids, as only 22 per cent of their chains have conjugated unsaturated structure. In these conditions (200°), losses of carboxyl groups by decarboxylation of maleic anhydride take place²⁸, moreover at this temperature, maleic anhydride reacts also as an acid on residual hydroxyl groups, which results in crosslinked products. Preliminary studies showed that resins with good aqueous solubility could be obtained, but the viscosities of the resulting varnishes were too high for the desired applications (≈ 30 instead of max 10 dPas). Moreover, due to the consumption of double bonds during the reaction, their drying times were too long. For these reasons this method was not investigated further.

Another reaction the unsaturated groups involves copolymerizing the resin with acrylic or methacrylic acid, however formation of homopolymers cannot be avoided. These reaction products are compatible with the modified alkyd resin in aqueous solution but the resulting films are either opaque or cloudy.

2. Polycondensation of an acid bearing monomer during the formation of the chain

2,2-bis(hydroxymethyl)propanoic acid (DMPA) is particularly interesting as a reactant for the introduction of carboxylic groups into an alkyd resin during its formation, since due to steric hindrance, the carboxyl group is not reactive under the synthesis conditions; DMPA behaves as a diol with two primary hydroxyl groups. The resulting resin bears carboxyl

Table 1

Carboxyl contents (x_{COOH}) and average functionalities (\bar{f}) of the resins synthesized from DMPA (R2 to R6)

x_{COOH} (eq/kg)	0.7	1.07	1.60
* \bar{f}			
1.60	R2	R3	R4
1.80	R5	R6	
* $\bar{f} =$	number of reacted functional groups		
	total number of initial molecules		

Table 2

Maximum solvent content (% wt) of neutralized resin solutions in 80:20 (wt) water/ethoxyethanol mixture before loss of clarity. Influence of the nature of the tertiary amine.

	NN-dimethylethanolamine	Triethylamine	Morpholine
R1	30	50	50
R6	30	30	40

groups which can be neutralized in an aqueous medium by trialkylamines.

3. Reaction of an anhydride with the hydroxy groups of pre-formed chains

Trimellitic (TMA), or pyromellitic (PDMA), anhydrides can be reacted with the hydroxyl groups contained in the resin. Due to their high functionality and the high reaction temperature these compounds must not be introduced at the beginning of the synthesis due to premature crosslinking of the resin²⁶. They must therefore be reacted at a lower temperature $\approx 160^\circ\text{C}$, since in these conditions anhydride groups are more reactive than carboxylic groups. TMA behaves as a monofunctional compound and is fixed as a side group on the resin chains. However, PDMA which is a dianhydride leads, even in mild conditions, to gelled compounds if the stoichiometry is not carefully controlled.

Preparation and study of water-soluble alkyd resins with high fatty chain contents

These preliminary experiments and the results found in the literature led to the development of the two following methods for the preparation of water-soluble alkyd resins with high fatty chain contents:

1. Use of DMPA as a comonomer during the alkyd resin formation.
2. Fixation of TMA or PMDA on the polyester backbone of a preformed resin.

All resins were synthesized from tall oil fatty acids (62 per cent wt), pentaerythritol, phthalic anhydride and either DMPA, TMA or PMDA. The process is described in the experimental part of this paper.

To be of use the coatings prepared from these resins must have good aqueous solubility, low viscosities (≤ 10 dPas) and since high gloss is required, must be clear. The higher the amount of aqueous solvent added before the solution turns cloudy, the better the resin.

Use of 2,2 bishydroxymethylpropionic acid (DMPA)

By using different molar ratios of the reactants we obtained

Table 3

Final carboxyl contents of resins obtained by reacting R8 with different amounts of TMA

Resin	R9	R10	R11
% (wt) TMA	5.80	8.90	14.50
x_{COOH} (eq/kg)			
observed	0.71	0.97	1.50
theoretical	0.74	1.06	1.60

Table 4

Properties of paint films obtained from resins R7, R10, R11 and R12

Resin	R7	R10	R11	R12
Drying time (h)	16	16	7	30
Hardness (20 days)	61	80	113	59
Gloss 60°	90	90.5	90.5	93.5
Weatherability (h)	<90	<90	<40	<90

resins with various average functionalities \bar{f} and various carboxyl contents (Table 1).

Resins 2 to 6, after neutralization by morpholine exhibited a certain solubility in water, but the solutions were highly thixotropic. When more than 30 per cent (wt) of water was added, they turned cloudy. The addition of a cosolvent (ethoxyethanol EE) avoided these phenomena and the solubility of the resin was improved (Figure 1), the presence of a cosolvent was always necessary.

Figure 2 shows the influence of the average functionality \bar{f} and the residual carboxyl content x of the resins. An optimum trialkylammonium carboxylate content corresponds to each value of \bar{f} . Below this value the viscosity was high and the solubility low ($\bar{f} = 1.8$, $x_{\text{COOH}} = 0.7$ eq.kg⁻¹). At the optimum, the viscosity was low and the compatibility of the medium with water is satisfactory since it was possible to prepare c.a. 1:1 mixtures of resin/aqueous solvent, without separation or loss of clarity. A further increase in carboxyl content of the resin did not increase the solubility, but slightly increased the viscosity and decreased the amount of aqueous solvent which could be added before the medium became cloudy.

The resins 2 (R2) and 6 (R6) which possessed the best characteristics, were chosen for the following studies.

Choice of the neutralizing amines

Three tertiary amines were tested to neutralize the solutions of R2 and R6 in 80:20 (wt) mixtures of water and ethoxyethanol. The results are reported in Table 2.

Examination of table 2 shows that morpholine was more efficient than triethylamine or NN dimethylethanolamine. This was observed for all the synthesized resins. A slight excess (10 per cent wt) of amine was necessary to avoid phase separation on dilution.

Choice of the cosolvent

The cosolvent must be water soluble, compatible with the resin and volatile in order to allow room temperature drying of the resulting coatings. Alkylated derivatives of ethylene glycol are suitable compounds. Figure 3 showed that 2-butoxyethanol (BE) was a better cosolvent than 2-ethoxyethanol (EE) as it gave clear solutions with lower viscosities. This was probably due to the increased length of BE alkyl chain, which made this

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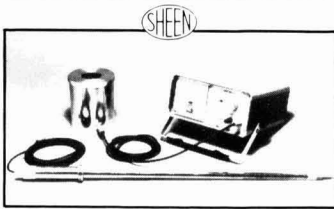
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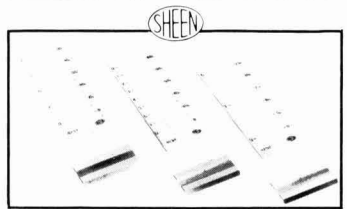
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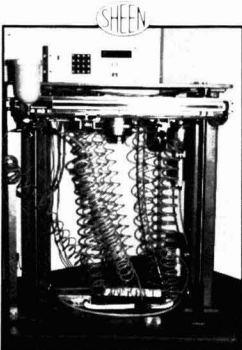
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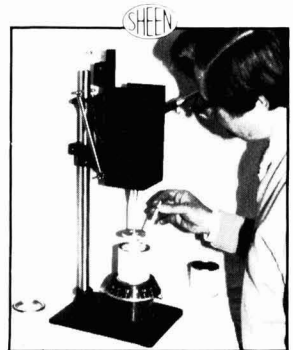
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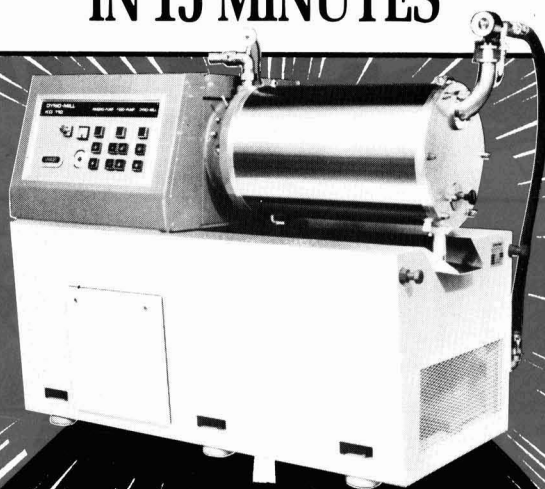
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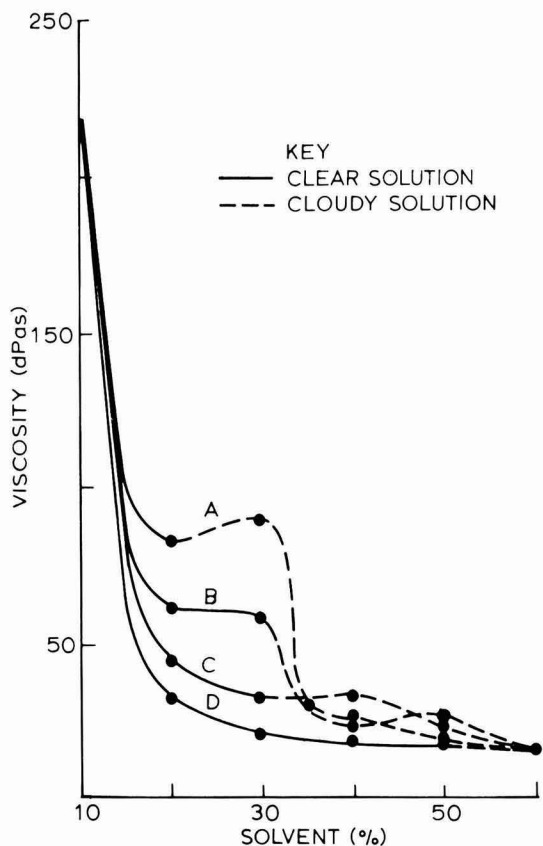


Figure 1. Variations in the viscosity (20°C, method 2) of neutralized resin R2 solutions in water/ethoxyethanol mixture (solvent). Percentage (wt) of water in the solvent mixture: Curve A (100), B (90), C (80), D (70)

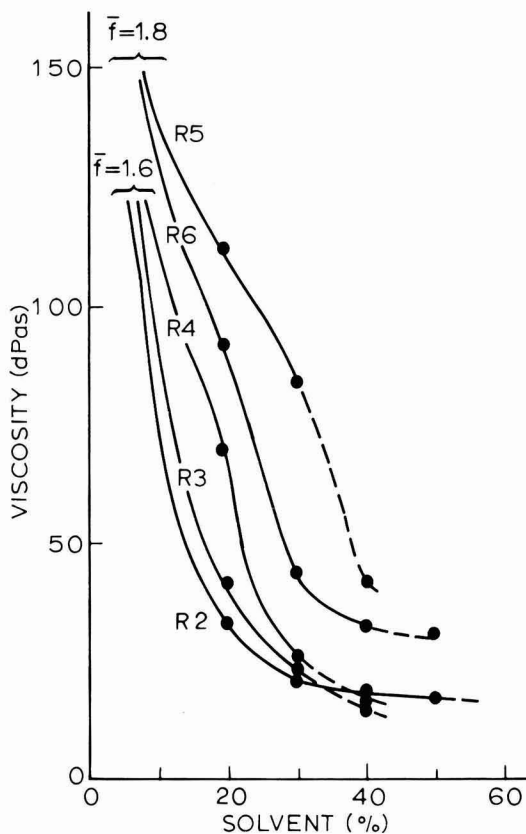


Figure 2. Alkyd resins obtained from DMPA: Variations in the viscosity (20°C, method 2) of their solutions against the amount (% wt) of solvent (70:30 (wt) water/ethoxyethanol). Influence of the average functionality \bar{f} of the resin, and of the residual carboxyl content (x). Symbols: see Table 1

compound more compatible with the fatty side chains of alkyd resins.

Optimisation of the drying time

The coatings prepared from these resins exhibited long air drying times (≥ 30 h). When 30 per cent of the tall oil fatty acids were replaced by dimeric fatty acids, the resins had a higher functionality (R7, $\bar{f} = 1.86$) leading to shorter drying times. Figure 4 shows that aqueous solubilities are very satisfactory as it was possible to obtain low viscosity (5 dPas) clear solutions in 87:13 water/BE mixtures with no more than 30 per cent solid content. The coating prepared from R7 showed an improved room temperature drying time (16 h), and the resulting dried paint films had satisfactory properties (Table 4).

Use of trimellitic anhydride (TMA)

As the trifunctionality of TMA at 200°C could lead to crosslinked compounds a two-step process was employed by reacting TMA at a lower temperature (170°C) with the excess hydroxyl groups of a preformed resin R8 ($\bar{f} = 1.82$, $x_{\text{COOH}} = 0.15 \text{ eq.kg}^{-1}$, $x_{\text{OH}} = 1.83 \text{ eq.kg}^{-1}$). Using different amounts of TMA the resins R9 to R11 described in Table 3 were obtained. However a fraction of the carboxylic groups resulting from the opening of the anhydride rings reacted with the residual hydroxyl groups of the resin resulting in a molecular weight increase and a carboxyl content which was lower than expected.

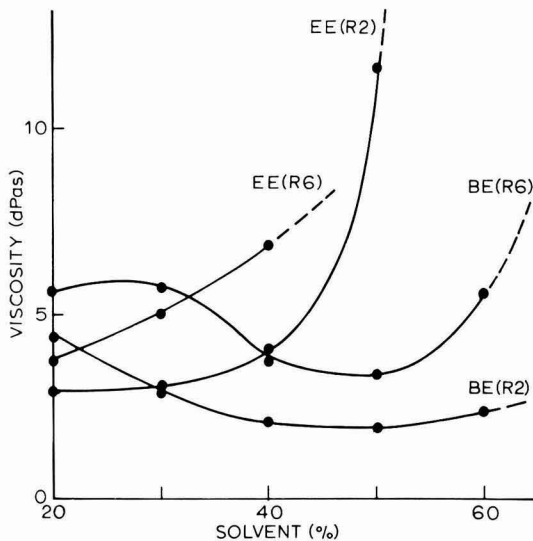


Figure 3. Dilution in water of 80:20 (wt) mixtures of resin R2 or R6 with a cosolvent: ethoxyethanol (EE) or butoxyethanol (BE). Variations of the viscosity (25°C, method 1) of resulting solutions against the total amount of solvent (water + cosolvent). See Figure 1 for key

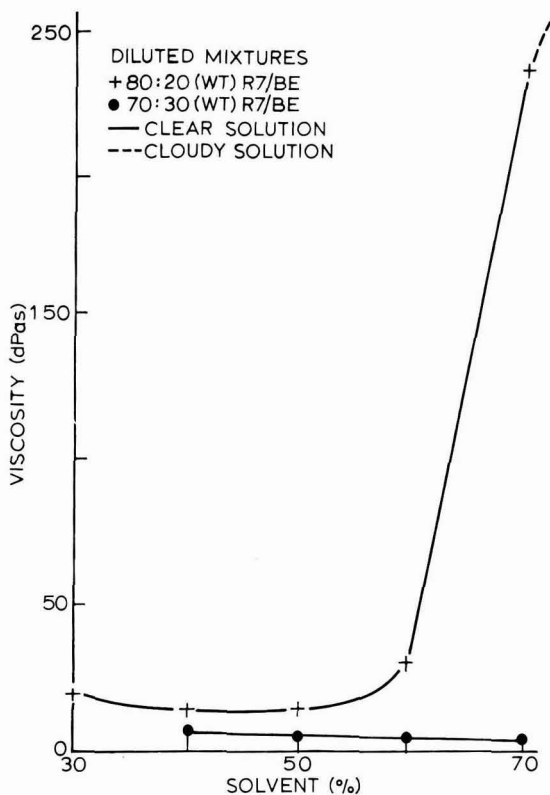


Figure 4. Diluted (in water) mixtures of resin R7 and butoxyethanol (BE), and the resulting variations in the viscosity (25°C, method 1) of the solutions against the total amount of solvent (water + BE).

After neutralization with morpholine (10 per cent excess) R9 ($x_{\text{COOH}} = 0,7 \text{ eq/kg}$) the resin failed to give clear aqueous solutions, even at low solvent content; as with DMPA modified resins a carboxyl content close to 1 eq/kg seems to be the optimum. Morpholine was found to be a better neutralization amine than NN dimethylethanolamine or tirethylamine, and 2-butoxyethanol a better cosolvent than 2-ethoxyethanol (R11, Figure 5). Clear coatings and paints prepared from these resins exhibited satisfactory air drying times and good film properties (Table 4; R10, R11).

Use of pyromellitic dianhydride (PMDA)

In order to obtain a resin with a carboxyl content equal to 1 eq.kg⁻¹, PMDA was reacted for 30 minutes at 170°C with the hydroxyl groups of a preformed alkyd resin. IR spectra show the disappearance of anhydride groups, and carboxyl group titrations gave contents close to the theoretical showing that, unlike TMA, PMDA reacted only through its anhydride groups. It was possible to obtain aqueous varnishes with satisfactory drying times, but their viscosities were too high (>15 dPas). To obtain lower final viscosities, the average functionality was decreased by replacing pentaerythrytol by a mixture of trimethylolpropane (TMP) and pentaerythrytol (R12). The resulting resin had a satisfactory aqueous solubility and a low viscosity (Figure 5). Varnishes and paints prepared from this resin have good properties (Table 4).

Experimental

Tall oil fatty acids were obtained from LOTIGIE (Paris, France) carboxyl content 3,54 eq.kg⁻¹. Composition (per cent

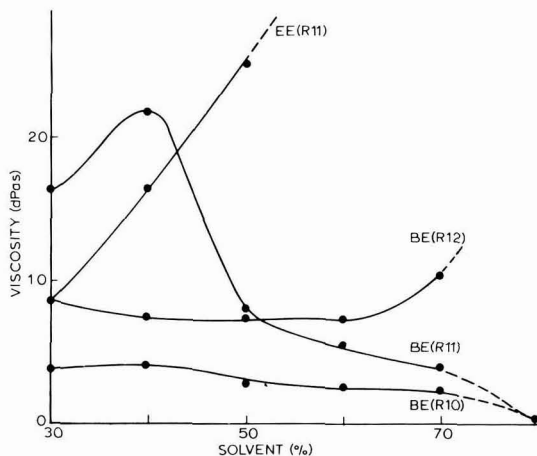


Figure 5. Dilution in water of resins R10, R11, or R12 mixtures with butoxyethanol (BE) or ethoxyethanol (EE) 70:30 (wt) resin/cosolvent; mixtures neutralized with 10 per cent excess morpholine. Variations of the viscosity of the resulting solutions against the total amount of solvent (water + cosolvent) present in the medium. See Figure 1 for key

wt) (Gas chromatography): hexadecanoic (0.2) 9-hexadecenoic (0.7), octadecanoic (0.7), 9-octadecenoic (36.0), 9, 12-octadecadienoic (39.4), 9, 11-octadecadienoic (22.1) acids, and unsaponifiables (0.9).

Sulfonated fatty acids (C): A 41.6 per cent aqueous solution of 1:1 mol mixture of 2-sodiosulfonato-octadecanoic acid methylester and 2-sodiosulfonato-hexadecanoic acid methylester was obtained from Union Générale de Savonnerie Co. (Marseille-France).

Dimer fatty acids (Empol 1022) were obtained from Unilever-Emery Co (The Neth.). Carboxyl content 3,50 eq.kg⁻¹ composition (per cent wt): monoacids (3), dimeric acids (75), trimeric acids (22).

Trimellitic anhydride (TMA) (anhydride content 97.5%: AMOCO-CHEM-Co, USA), Pyromellitic dianhydride (PMDA) (99.7%, HULS-France), Pentaerythritol (PE) (95%: SNPE, France) Trimethylolpropane (TMP) (99%: BAYER, F.R.G.), isophthalic acid (IPA) (95%, AMOCO CHEM-Co, USA), phthalic anhydride (PA) (99.7%, PCUK, France) dimethylolpropanoic acid (DMPA) (96%, TROJAN POWDER-Co, USA) were used without further purification.

Analytical grade solvents were purchased from PROLABO (France).

Measurements

Carboxyl contents were determined by titration with aqueous N/10 KOH.

Hydroxyl contents were determined by acetylation with excess acetylchloride in pyridine (60°C, 30 min), and back titration with N/2 alcoholic KOH.

Viscosity: The viscosimeters used were either a Nouryvisko ball viscosimeter (Method 1) or a Rotovisco RV3 (HAAKE) Method 20.

GPC: waters 6 000 A pump with 100 Å + 2 × 500 Å + 10⁴ Å μ styragel columns.

IR: Perkin Elmer 577

Table 5
Composition of resins 2 to 12 reaction mixtures – weights (g) of reactants for 100 g final resin

Resin number	2	3	4	5	6	7	8	9	10	11	12
Tall oil fatty acids	62	62	62	62	62	49.6	68.58				62
Dimeric fatty acids						12.4					
DMPA	9.55	13.40	21.49	9.55	13.40	13.40					
TMA								5.80	8.90	14.50	
PMDA											10.90
PE	18.93	15.01	6.88	15.38	11.57	11.82	21.43				7.48
TMP											14.90
PA	15.35	15.42	15.47	19.38	19.34	19.06	16.36				9.88
Resin 8								94.20	91.10	85.50	

Gas chromatography: GIRDEL 75 FSI with chromosorb G DMCS "fatty acid" column (3 m).

Drying time: ASTM D 1640.

Gloss at 60°: AFNOR Pr T 30-064.

Hardness: AFNOR T 30-016.

Weatherability: AFNOR T 30-053-3rd.

Synthesis

Reactors (4 000, 250, 150 cm³) fitted with a stirring device, reflux condenser or distillation column, nitrogen inlet and diffuser, electronic temperature regulation and monitor, and sample withdrawing outlet, were used throughout.

Resin 1

Tall oil fatty acids (63.35 g, 0.1487 mol) sulfonated fatty acid aqueous solution (104.21 g, 0.0743 mol, fatty acids) and silicone antifoaming agent (0.2 g) were slowly heated to 140°C until water elimination was complete. Afterwards pentaerythritol (28.77 g, 0.14 mol.), phthalic anhydride (26.49 g, 0.1184 mol.) and tetrabutoxytitanium catalyst (0.2 g) were added and the mixture heated to 200°C for 16 h.

Residual carboxyl content: 0.27 eq/kg¹.

Resin 2 to 7

The reaction mixture (Table 5) was slowly heated to 205°C and this temperature maintained until the expected carboxyl content was obtained (7 to 10 h).

Resin 8 to 12

All reactants with the exception of the anhydride were slowly heated to 220-230°C until the carboxyl content was below 0.18 eq/kg. (6 to 20 h). The mixture was cooled to 150°C and the amount of anhydride introduced and reacted at 170°C until the total disappearance of anhydride peaks in the IR spectra. (1 h for TMA, 30 min for PMDA). See Table 5 for reaction mixture composition.

Coatings

Resin (40 g) was mixed with 2-butoxyethanol (17.14 g) and neutralized with 10 per cent excess morpholine. 1.33 g of 6 per cent Co drier and 1.33 g of 10 per cent Zr drier were added, and the mixture weight adjusted to 100 g with distilled water.

Paints

The varnishes were mixed with Ti oxide RN-59 pigment (pigment/binder ratio = 0.8) and diluted with water to achieve a viscosity (Method 2) of 10 dPas. The properties of resulting films are reported in Table 4.

Conclusion

The introduction of carboxylic groups to an alkyd resin, either during the formation of the chains (use of DMPA) or by chemical modification of the preformed chains by different anhydrides, is an easy way to obtain water-soluble alkyd resins after neutralization by a tertiary amine with oil lengths above 60 per cent. The resulting products are soluble in water and exhibit air drying times and application conditions similar to classic alkyd resins. Moreover these syntheses do not require any modification of existing resin plants, only some minor changes in the processes.

[Received 23 January 1984]

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short communications

Synthesis of glyceryl monoaleuritrate

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Summary

Glyceryl monoaleuritrate (III) has been synthesized by reacting aleuritic acid (I) with glycerol (II) at $225 \pm 5^\circ\text{C}$ for one hour and 15 minutes in the presence of zinc dust as catalyst.

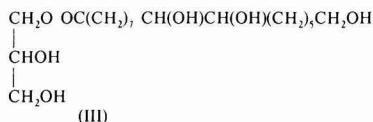
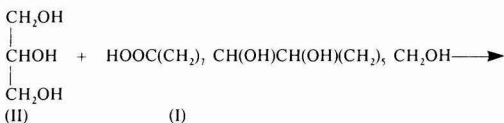
Introduction

The monoesters¹⁻⁴ of glycerol such as glyceryl monopalmitate and monostearate are widely used as emulsifiers in the food⁵ and cosmetic industries⁶.

These esters are generally prepared by the combination of glycerol with long chain fatty acids. However, for use in food industry fatty acids must be edible. These monoesters due to their hydrophilic nature, help in its emulsification and its stabilisation. Besides the above mentioned uses, some of the monoesters like glyceryl monopalmitate also find use in the preparation of cakes, cake mixes, whipped toppings and bread.

Aleuritic acid (9,10,16-trihydroxy palmitic acid), the major constituent acid of shellac⁷ is believed to be present to the extent of ≈ 40 per cent⁸. As aleuritic acid is readily available and also reasonably cheap it prompted us to synthesise the hitherto unknown glyceryl monoaleuritrate. Its synthesis followed by its application in various fields augers well for the aleuritic acid market.

Scheme of the synthesis



Experimental

Materials used

Aleuritic acid was prepared by alkaline hydrolysis of shellac, recrystallised from water, m.p. $100-101^\circ\text{C}$ (TLC pure).

Glycerol, AR grade, BDH.
Zinc dust, LR grade, BDH.
Solvents for TLC and purification, AR grade, BDH.
Ether and other reagents, LR grade, BDH.

Preparation of glyceryl monoaleuritrate

Aleuritic acid⁷ (30.4 g, 0.1 mole) and glycerol (9.2 g, 0.1 mole) were placed in a three-necked flask and the mixture was heated to $225 \pm 5^\circ\text{C}$. Zinc dust (1.2 g) was added as a catalyst and temperature was maintained at $225 \pm 5^\circ\text{C}$ for one hour and 15 minutes with constant stirring. The product was then poured into ice-cold water and extracted with ether. The ethereal layer was washed sequentially with aqueous sodium bicarbonate solution (25 ml; 5 per cent) and water. The extract was then dried over anhydrous sodium sulphate and removal of the solvent afforded a solid, which on crystallisation from ethylacetate gave white needles of glyceryl monoaleuritrate, m.p. $63-65^\circ\text{C}$, yield 80 per cent.

The Rf values of aleuritic acid and glyceryl monoaleuritrate were found to be 0.40 and 0.11 respectively (solvent system, ethylacetate : acetic acid, 100 : 1, v/v), while the Rf values of the same products in another solvent system (chloroform : methanol : acetic acid, 90:10:2, v/v) were 0.35, and 0.10, respectively. The IR spectrum : 3380 cm^{-1} (hydroxyl), and 1730 cm^{-1} (ester carbonyl).

Found : C, 60.20; H, 10.50. $\text{C}_{19}\text{H}_{38}\text{O}_7$, requires C.60.31; H, 10.52 per cent.

Discussion

The preparation of a monoester of this nature, using glycerol having three free hydroxyl groups and aleuritic acid with three hydroxyl groups at different positions, two of which are vicinal and the third along with the carboxyl group at the two terminal C atoms of the long chain acid, poses a problem for the preparation of pure monoester. There was every possibility that the aleuritic acid molecule might react among themselves to form an interester resulting in polymerisation besides the formation of mixture of mono-, di- and triesters.

Obviously special efforts were made to avoid such complications. The reaction was carried out under different conditions of time, temperature and molar ratios of the reactants and also in the presence of different catalysts but in all the cases a mixture of mono-, di- and triesters of glycerol were obtained. However, when zinc dust was employed as the catalyst, a pure monoester of glycerol was obtained.

Acknowledgements

The authors are thankful to Dr B. B. Khanna, Head, Division of Chemistry, and Dr T. P. S. Teotia, Director, for their keen interest in the progress of this work. They are also grateful to Mr M. Ekka and Mr U. Sahay for their valuable technical assistance.

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next month's issue

The Honorary Editor has accepted the following papers for publication in the March issue.

Dispersion of cuprous oxide in antifouling paints; coefficients defining particle shape and size by C. A. Giudice and B. del Amo

Styrenated alkyd resins based on maleopimaric acid by A. K. Vasishtha and M. C. Shukla

An evaluation of the falling rod viscometer and the inkometer for the testing of news inks by G. Pangalos and Prof. J. M. Dealy

occa meetings

Transvaal Section

Patent Rights

On 14 August 1984, the Section was addressed by Mr Jan Lamprecht on the subject of "Patent Rights".

The first law in South Africa covering Patent Rights was promulgated in 1860 with revisions in 1916, 1972 and 1978. The law defines what may be patented and makes provision for legal redress. Patents are granted for 20 years but they must be licensed every three years, and they remain vulnerable to attack in court for their duration. Trade Marks have an indefinite period of validity provided they are used and registration is renewed.

The advantages and disadvantages of Patent Rights to both the holder and users of patented products have stimulated development, and in countries where patents have been abolished, a concomitant decrease in development has occurred.

After a lively question and answer session, a vote of thanks to the speaker was proposed by Mr Peter Gate and endorsed by the audience.

The development of coatings for wine tanks

At a meeting held at the KWV Wine Centre on 26 September 1984, Mr Marais de Villiers of KWV (South African Co-operative Wine Producers Association) delivered an address on the subject "The development of coatings for wine tanks".

It may seem strange that there is a need for coated tanks in an industry that last year used 2,000 tons of stainless steel. When, however, it is appreciated that 70 Co-ops within KWV have tank capacity of 7.5 million hectalitres, of which 40 per cent are concrete, the need for lining becomes clear.

In 1979 KWV estimated that to replace concrete tanks with stainless steel would cost R31 million. Therefore, a research programme on the lining of concrete tanks was initiated.

The problems at that stage were poor resistance to alcohol, and that the materials used depended on imported technology. Coatings must comply with a number of factors, they:

1. Must not contaminate the contents
2. Must be smooth and glossy
3. Must be acceptable to, for example, FDA
4. Must be versatile and be resistant to materials ranging from moss to potable spirit.

Testing panels using "blind" organoleptic evaluations were found to be faster and more reliable than GC or LC methods.

By 1982 two prototype coatings were formulated, and after exhaustive testing, the KWV Politank lining material was produced. While the formulation cannot be divulged, the Politank system may be described as a standard bisphenol-A epoxy resin modified with a multi-functional epoxy novolac and cured with a cycloaliphatic diamine. The pigmentation is 40 mesh 316 stainless steel flake.

Following an active question period and the vote of thanks proposed by Mr Peter Quorn, the audience were treated to a fine selection of the wine producers art provided by KWV, our hosts for the evening.

Alternatives to bridge painting

On 16 October 1984, a joint meeting of OCCA and the South African Corrosion Institute was held at the Science Park, Kelvin, in Johannesburg. The speaker was Dr R. R. Bishop of the British Transport and Road Research Laboratory, who was visiting South Africa to attend the ISO Committee Meeting of ISO/TC35/SC12 of which he is chairman. Dr Bishop presented his paper "Alternatives to Bridge Painting". (See *JOCCA*, November 1981).

Although bridge construction methods in South Africa differ widely from those of the UK, this lecture generated a great deal of interest among the audience, as evidenced by the hearty question and answer period.

A vote of thanks was proposed by Mr Gordon Munro, Vice Chairman of OCCA, Transvaal.

Marketing – the unconventional approach

At a meeting held at Science Park, Kelvin on 23 October 1984, Mr Neville Marks, marketing director of Plascon-Evans Paints Ltd, presented a lecture entitled "Marketing – the unconventional approach".

Marketing was defined as the creating of an atmosphere conducive to the successful selling of a product or service. Effective advertising must be geared to fulfil a need, solve a problem, or satisfy a desire. The product must be able to perform to the advertised standard, and therefore product knowledge training was considered essential as were clearly defined marketing policy parameters and objectives. Production and distribution facilities must be capable of meeting the increased demand created within the range of successful marketing campaigns. Market research, advertising media selection, and retail sales strategies were illustrated.

Following an interesting discussion period, Mr Eric Timm proposed the vote of thanks. Plascon-Vans Paints Ltd, were our hosts for the evening.

B. Bailey

Manchester Section

Coatings and the chip, microprocessing our industry

On Friday 28 September 1984 the Manchester Section one day symposium entitled "Coatings and the chip, microprocessing our industry" was held at the University of Manchester Institute of Science and Technology.

After the official welcome by Mr G. R. Robson Chairman of the Manchester Section OCCA, the morning session was chaired by Mr Camina of the PRA.

The introductory lecture was by Dr M.G. Hartley of UMIST. Dr Hartley set the scene for the day by giving an introduction to the current situation via a historical treatment of computers. Starting with the Chinese Abacus to Babbage's digital computer using purely mechanical methods in the 19th century, he showed how scientific, commercial and military calculations could be made. Electricity enabled electronic switching to be utilised, but the early computers were large and cumbersome, 50 feet long with a 200 kW power consumption but with only the capability of a modern home computer. The connection between Ferranti and UMIST and later ICL was explained in computer development and Dr Hartley rounded off with the current situation of vastly lower cost for hardware but a requirement for efficient usage by sharing of software to keep costs reasonable.

Introduction to computer technology

The speaker was Mr G. T. Eady of Ault and Wiborg. Mr Eady's explanation of the binary system enabled even the uninitiated to understand the simplicity of the basic theory involved in BYTES and BITS.

He then developed the technology for introducing languages that the computer can understand, and the methods of storing information using magnetic tapes, floppy discs and metal discs, explaining the difficulty of access with tapes and greater storage and rapidity of search with discs. Information displayed on VDU's can be printed out by daisywheel or dot-matrix printers.

Questions were asked about voice simulation, bubble memory and relative stability of storage of the various systems with the risk of loss of material at various stages.

Use of computers and research and development of coloured molecules

The speaker was Dr Bamfield of ICI Organics Division. Dr Bamfield equated molecular designed with research and gave examples of the computer support required for such a vast undertaking. In molecular modelling, a wide range of molecular orbital programmes including 'ab initio' could be dealt with, using both high and low grade graphics.

A chemical data bank enables rapid location of samples from 70,000 stored at Blackley and 400,000 stored within ICI. In colour chemistry, colour and colour strength, maximum absorption wavelength and absorption and extinction coefficients, can be predicted for organic molecules. Examples showing the close correlation were given. The colour differences for different crystal forms of the same molecule, e.g. alpha and beta copper phthalocyanine were explained.

Automated paint manufacture

The lecturers Mr W. Ollett and J. Boyden, covered the automated control and handling system for paint manufacture at Crown Paints.

The system was designed to eliminate human error, give better utilisation of plant and to improve consistency and production rate; all of which would lead to less waste and hence more profit. A production of 25 million litres per year with the computer involvement in pigment, liquid and mill base storage, handling and distribution was demonstrated.

The process of paint manufacture was taken through each stage (the print shown was too small to be easily readable on the slide). Using a series of loops the clearance of mixer empty, mixer clean steps enabled mixing operations to be carried out. The control tasks include monitoring of weigh tanks for liquids and bulk powder with coded signals being received from relevant valves and weight and volume indicators.

The software for the system took five years to write and the information and processing instructions are designed to eliminate short cuts by operators. Batch progress is monitored by MIMIC checking the line progress, VDU's are used and these can control the process, but it is normally automatic. Pigment addition involving fluidisation requires a sequence of switching of blowers, fluidizers, and powder/air valves, and this is achieved by one signal. The paint additives are manually added and batch correction is through VDU instructions. Questions on the performance of the system were asked, and only transistor failures in 2 years operation had resulted in no disasters and this showed the overall benefits of such a system.

Use of microprocessors in colour quality control

Mr K. McLaren of ICS Ltd, introduced the subject of colour measurement using the relationship of the reflectance curve to colour. He reviewed the progression from manual spectrophotometers to the recording types.

As all the information required is contained in the spectral reflectance curve, this could be converted into Tristimulus xyz values. The computer enabled calculation of these figures to be rapidly achieved. Tristimulus Colorimeters gave results directly, and the original 1969 instrument is still working. Further developments were discussed enabling colour difference and match prediction to be achieved if information

storage is available. Costs could be repaid in 12 months. The aspects of quality control were good in textiles, fairly good in gloss paints but poor in emulsion paints. Examples of each were given.

The three dimensional concept of colour from Fursius to Munsell introduced the problems of colour difference tolerances. Numerous objections can be catered for and the modern systems Cielab, JPC and MS series of modified equations were dealt with. The inability to measure wet films was explained.

Robotics

Mr M. Podmore of Dainichi-Sykes Robotics Ltd, dealt with the development of Robots, illustrating the degrees of freedom and degree of control now available.

Air, hydraulic and electric power sources were discussed showing their relative costs and limitations of speed, control payload and hazards resulting in electric drives now being dominant as well as being more compatible with electronic control. Enclosure of vulnerable parts within a positive pressurised air bag increases safety.

The relationship of end of arm tooling with specific jobs was explained enabling spot welding, arc welding, paint spraying, stacking etc. to be achieved. Control by sensors at each joint enable the position and direction of movement to be reported to the computer and requisite signals for action to be made.

Point to point programmes are prepared for each positional point, giving the necessary clearance for fairly straight lines. For continuous path, the actual path and the speeds required are programmed and positioned; information is stored at all stages.

The path may be determined using a teaching arm, the operator leading the robot through the pattern and all information being stored. Details of payload and reproducibility were then given. This shows that many repetitive, hazardous and tedious work can be safely done by Robot.

Use of microprocessors in sales and marketing

Mr T. A. Dalton, of England and Bell, explained the uses of voice response systems to give parts availability and in relaying orders. These use the telephone system overnight and are used, mainly by banks and larger firms.

The use of microprocessors enables sales literature and sales support to be readily available using portable equipment.

This would revolutionise the sales rep as detailed information could be stored with rapid access and be more quickly updated than conventional printed literature.

On market modelling, the more rapid processing of reports and price changes reduces the risk in making decisions and increases the availability of the right product at the right time. Mr Dalton concluded with the alternate definition of OCCA "only computers can answer".

Sources of assistance

Mr Heard of DTI, outlined the availability of consultancy services to tailor-make computer services for individual requirements.

Details of the financial assistance available to firms and

details of the limitations were given. Leaflets giving these details were available at the DTI stand.

Future role of computers and micro processors

Having chaired the afternoon session, Dr Hartley of UMIST crossed the platform to discuss the increasing roles for micros. Firstly: standing alone. Secondly: as part of an intelligent instrument. Thirdly: linked to a network for information exchange with access to a data bank, stock control etc.

He reviewed the future with regard to cost-decreasing for memory i.e. unit cost per bit enabling larger data banks to be established and with regard to special purpose, semi custom chips as transducers, analogue/digital specials and speech synthesisers.

The explicit software will become more expensive for newer technology, character recognition, speech synthesis image processing, robotics and networking.

A discussion on the 1984 approach to the "Quality of life", uniformity, security hazards, jobs deskilling and jobs, best brought the afternoon session to a close.

Mr Gordon Robson closed the meeting by expressing the gratitude of the delegates to all those who had made the symposium a success.

R. G. Handley

London Section

Problems encountered in can making

The first meeting of the 1984-85 season was held on Thursday 20 September 1984 at the new venue the Pearly King in Bow, London E3.

Mr Alan Waller of Nacanco Limited, assisted by Mr Peter Tye, gave a very informative talk on the problems encountered in can making. His theme was that a close understanding between the can maker and paint manufacturer was needed.

Mr Waller referred to the problems encountered in the 1950's in packaging water based products and in particular to the positioning of handle rivets in relation to the level of the paint inside the can. Generally corrosion was less when the handle rivet was below the paint level. He had found that corrosion was at its worst when water had a high level of chloride ions, and that the nitrate levels could aggravate the situation; this being relevant during periods of water shortage. The advantages of welded seams which, because of the method of construction give less exposed metal; and the pressure reducing effect of raised panel caps, were also mentioned.

Considerable plant investment by Nacanco has led to improved can making ability, and closer control of the print and coatings application in the most modern tin-printing works in the world. In production areas no large stocks of cans are held. Flat printed plate is held in stock and made into cans when required for despatch. The plate is stored at a controlled temperature and is handled carefully to avoid scratching which could lead to electrolytic corrosion taking place.

In the lively discussion period which followed, the topics covered were: the nature of the corrosion encountered, whether square or round cans were favoured for solvents, changes in

materials of construction to meet future legislation requirements and various aspects of alternative can design.

In his note of thanks to the speakers Mr Norman Locke said he was pleased that there should have been such a good attendance for this meeting of the 1984-85 session. He thanked them for their excellent presentation and for the way in which the wide ranging questions had been answered.

K. H. Arbuckle

Midland Section

Electrostatic spraying

The second lecture this session of the Midland Section was held on 15 November 1984 at the Clarendon Suite, Stirling Road, Edgbaston, Birmingham.

Members and guests heard Mr R. P. Winter of Binks Bullows Ltd, give a talk entitled "Electrostatic Spraying".

Mr Winter began his talk by outlining the principles of electrostatics. Historically electrostatic forces were observed more than 2,000 years ago when it was noticed that if amber was rubbed with other dry substances then it had the ability to attract feathers and other light objects.

In electrostatics the voltages are very high in the order of 30-150 kV, but the current is low only about 75-150 micro-

amps. The lethal current density is in the region of 15,000 micro-amps. The speaker continued his talk by describing some of the equipment used saying that one of the earliest systems used was known as the Ransburgh No. 1 process. Here the paint was sprayed from a conventional gun at fairly low pressure through a high voltage grid. The next stage was the development of rotating discs and bells. This type was sometimes called the "Pure electrostatic" system. The disc or bell rotates between 1,500-3,000 rpm and was used mainly on automatic equipment because at that time operators were concerned about the safety of the equipment. To demonstrate the use and advantages of electrostatic spraying Mr Winter then showed a short film.

To conclude his talk the speaker outlined the current developments in finishing technology. Although high speed discs and bells have been on the market for 7-8 years it is only during the past 2-3 years that the paint technology has caught up with the equipment potential. The high speed discs and bells are driven by air turbines at speeds up to 60,000 rpm. The viscosity and conductivity of the paint is not so important here as with the other equipment. The speaker showed numerous slides to illustrate these latest developments. As to the future Mr Winter thought that more use would be made of robotics.

After a short question time the meeting was brought to a close with a vote of thanks proposed by Mr H. J. Clarke and endorsed by the audience in the usual manner.

B. E. Myatt

news

Berger Britain

A luncheon was held at the Savoy Hotel, London, on 20 November 1984 to celebrate the first anniversary of the reorganisation of the Berger Group and to announce a new corporate identity for the nine companies in Great Britain and Ireland - Berger Britain. The guest speaker was the Rt. Hon. David Steel, MP, Leader of the Liberal Party.

The guests were welcomed by Bill Collins, supervising director of the companies within Berger Britain - Arthur Johnson (Guiseley) Ltd, Berger Decorative Paints, Berger Elastomers, Berger Industrial Coatings, Berger Ireland, Berger Traffic Markings, Cuprinol, PGW Holdings Ltd, and Resinous Chemicals Ltd. In his address he pointed out that during the four year period 1979-83 the UK paint market declined by 7% in volume, manufacturers' profits by 32 per cent and return on assets from 20 per cent to 12 per cent despite an increase of 13 per cent in productivity. In real terms paint is about 8 per cent cheaper now than it was three years ago. Although only 50 per cent of Berger sales are in paint manufacture, to succeed in such highly competitive markets it has been necessary to specialise to meet the needs of different customers - big is no longer beautiful for its own sake. Customers are better off dealing with smaller companies that really understand their requirements, and this is what Berger Britain provides - each company in the group now has its own separate



David Steel (left) is seen here speaking at the luncheon in company with Bill Collins, supervising director of Berger Britain and Dominik von Winterfeldt of Hoechst UK, the parent company of Berger.

management and is focussed on selected markets. In addition there is a new central management team comprising the individual company managing directors and others responsible for specific functions, such as purchasing, on behalf of all nine companies. Capital expenditure of £6.5m has been initiated, e.g. on a custom-built, specialist sealant production plant for Berger Elastomers, on an advanced plant for Berger Decorative Paints and on a new factory for Berger Industrial Coatings. As regards research and development, Berger benefit from being part of Hoechst, the second

largest chemical company in the world, and as such are part of the biggest paint group in the world.

Summing up, the Berger Britain concept is to take advantage of size in areas where it is appropriate, but to develop the smaller independent units necessary to succeed in increasingly specialised markets.

T.A.B.

Crown Paints

On 4 December 1984 Crown Paints Industrial Division held a press briefing on their plans for the future. In his introduction Mr John Asher, Director of the Division, said that although Crown are best known in the decorative paint market they also have an industrial arm which is equally successful in its own markets. Thus in print finishing Crown first developed adhesives which made possible the lamination of oriented polypropylene film to board, whilst in coil coating they are probably the UK market leaders.

Until about 20 years ago industrial finishes were almost all hand-spray applied, low solids materials requiring lengthy stoving at relatively high temperatures. Competitive demands for quality and efficiency resulted in new methods of application and curing eg coil coating, curtain coating, powder coating, radiation curing and induction curing. As

regards products there has been a marked trend towards high solids types, water based types and low temperature curing types, some of them two-pack materials.

The newer processes are achieving an increasing share of the industrial finishing market. Thus powder coatings now hold about 5 per cent of the market and are expected to reach about 10 per cent in the early 1990s: radiation curing now holds about 1 per cent and is expected to achieve about 3 per cent in the next decade. Low-energy coatings (typically two-pack chemical curing systems), now hold 10 per cent of the market and are expected to reach about 17 per cent in the early nineties. Water-based coating materials are often more expensive than the corresponding solvent-based materials, generally require greater skill and control during application, but overall show a slight economic advantage: their market share has grown from about 2½ per cent in 1976 to 9 or 10 per cent now, and is expected to reach about 16 per cent in the early 1990s.

Crown are amongst the market leaders in these new technologies, and their position has been achieved by co-operating closely with plant designers and manufacturers as well as with users. In many cases this has led to appreciable economic savings overall and in some cases to considerable reductions in the space required for the finishing operations.

Mr Geoff Dodd, Technical Manager, discussed the main factors leading to development of the new technologies, covering methods of application and curing as well as the formulation of new materials. These include the oil crises of the early and mid 1970s, the three-day week in 1974 and the Health and Safety at Work Act of 1974.

The oil crises has focused attention on the costs of solvents and resins as well as on the fuel costs of stoving systems, and led to the development of higher solids products, solvent free materials (eg powder coatings), water-based products for a wide range of substrates and applications, whilst radiation curing systems, particularly ultra-violet curing, are specially suitable for heat sensitive substrates.

The three day week emphasised the need to reduce energy consumption, which led in part to the introduction of catalysed systems, particularly two-pack isocyanate curing systems of 60 or 80 per cent solids content – these requiring the lowest curing energy of any thermally cured coatings.

The Health and Safety at Work Act with its attendant Directives on Solvents (1973) and Paints (1977), the 1981 Lead Regulations and the EEC Labelling Regulations (coming into force in 1986) have made manufacturers and users more aware of the potential hazards of paint, although the speaker commented that there is sometimes

a tendency to pay more attention to potential carcinogens than to the usually major danger – flammability. There are, of course, some chemical hazards with paint, and these have led to the restriction or removal of some raw materials, eg benzene, asbestos, lead compounds, glycol ethers, chlorinated solvents and some isocyanate prepolymers.

These apparently adverse factors have spurred the development of a number of products and processes, and Mr Dodd highlighted Crown's achievements in powder coatings, radiation curing, water-based finishes and coil coatings. The first three reduce energy costs and improve both appearance and performance whilst the fourth removes completely the paint shop section of the production line. He spoke of their patented continuous loop process for emulsion manufacture – this is useful for small production runs and especially in new overseas factories where the high capital cost of conventional batch production plant can be avoided. Summing up, Mr Dodd said that when developing a new product for a customer Crown consider not only the end specification of the paint (i.e. its performance) but also environmental factors (ecology), health and safety (trade unions, personal prejudices, etc.), equipment and unit overall cost. The end result is to enable the customer to reduce costs or improve quality – or both.

T.A.B.

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BP Chemicals to expand ethyl acetate production facilities

BP Chemicals is to expand its ethyl acetate production facilities at Hull. The project due to be implemented in April 1985 will bring the total acetate capacity on the site from 80,000 tpa close to 100,000 tpa.

The expansion is based on the adaptation of the existing ethyl acetate unit using technology developed by BP Chemicals R & D department. A smaller multi-purpose ester unit on the Hull site has been used in recent months to establish the new process for main plant application. The feedstocks used will continue to be captively produced synthetic ethanol and acetic acid. The project includes the erection of additional storage for finished product.

In addition to ethyl acetate, BP Chemicals at Hull produces n-butyl acetate and isopropyl acetate.

Reader Enquiry Service No. 31

KleenTech buys coating specialist

Ensecote Ltd, a leading industrial surface coating and preparation company, has been acquired by KleenTech International, the fast growing specialist cleaning, maintenance and engineering services group.

"Ensecote's anti-corrosion, anti-abrasion and anti-fouling products and services, together with its unique stress relieving services, complements a number of our key service areas," said KleenTech's chairman and managing director, Eddie Ashby.

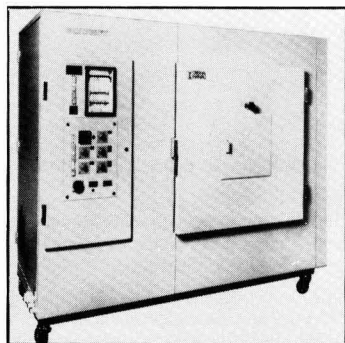
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products

New accelerated weathering tester

A new accelerated weathering tester, which it is claimed gives good simulation of the ultraviolet range of global radiation and is low cost, is now available.

The Heraeus original Hanau Soltest has been specially developed for weathering tests



The Heraeus Soltest accelerated weathering tester

in which the effect of UV radiation at various humidities and temperatures, or combined with rain cycles, is of particular importance.

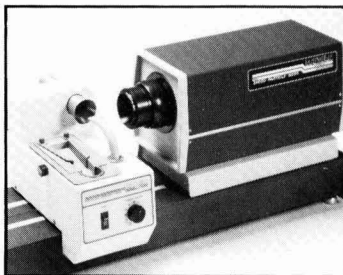
With its 16 special fluorescent tubes the Soltest provides high intensity UV radiation while keeping energy costs low. In addition it can be used without the radiation function, in which case it operates as a climate cabinet. By altering the combination of fluorescent tubes the special distribution can be varied, which means the radiation settings can always be optimised for each individual test as it is carried out.

The Soltest is also said to offer a wide range of temperature settings.

Reader Enquiry Service No. 33

Dry powder particle analysis

The Malvern 2600D and 2600HSD Particle Size Analysers can now be equipped with a dry powder feeder accessory which disperses the powder and presents it to the analyser in its dry form. The result is claimed to be a quicker, simpler analysis and, in some cases, claimed to give a more accurate result due to the elimination of any possible effects of the dispersing liquid. It also eliminates the need - in the case of water soluble powders - to resort to using organic liquids which are often unpleasant to handle and more expensive.



Dry powder feeder mounted on a Malvern 2600D Particle Size Analyser

Particles in the size range 0.5 - 300 microns can be measured and in the case of low density materials, the upper limit may be considerably higher. A typical measurement is claimed to take only ten seconds and require 1-5 cm³ of sample.

Reader Enquiry Service No. 34

meetings

Coatings for concrete

The Paint RA in association with the Building Materials Working Part of Committee of Directors of Research Associations will be organising a two day symposium on 20-21 March 1985 at The Royal Kensington Hotel,

London. It is designed to benefit users who need to select coatings for specific applications and paint manufacturers and their suppliers who need to be familiar with the state of the art.

About sixteen papers in four sessions will be presented on:

- Coatings using particular binders, i.e. polyurethane or polymer emulsion
- Coatings giving special properties, i.e. anti-condensation or multi-colour
- Coatings for aggressive atmospheres, i.e. marine or industrial
- Coatings for reinforced concrete and for the reinforcing bars
- Repair of damaged reinforced concrete
- Biocidal coatings and maintenance
- The size of the market.

Reader Enquiry Service No. 35

people

Mr R. J. Fletcher has been appointed sales manager of Diamond Shamrock Process Chemicals Ltd following the reorganisation of Diamond Shamrock's UK operation.



Mr W. E. I. Galloway has been appointed managing director of W. Canning Materials Ltd. The company is a leading manufacturer and supplier of industrial and speciality chemicals for the surface finishing industry.



Ian Patterson has been appointed sales manager of Solrec Ltd, the solvent recovery specialists.



As part of its plans for expansion Sheen Instruments has made a number of appointments to its technical sales staff. **Gordon Mitchell** has been appointed technical sales manager and will provide field support to three new area representatives. **Ram Subramanian** has joined the company as sales manager, UK North. Based in Grimsby, Mr Subramanian is a graduate chemist with 20 years experience in the UK surface coatings industry and was previously with the Paint Research Association, Laporte and Blundell-Permoglaze. **Keith Gregson** will cover the North West area, based in Sale, Cheshire. He has spent most of his career with Ferranti. **Gerry Howells** has joined the company from Esso and will cover central and SW England from his home base near Basingstoke.



Chris Russ has been appointed Southern Regional sales manager for Binks-Bullows Ltd, one of Britain's leading spray painting equipment manufacturers.

OCCA Conference 1985 – New substrates, new materials, new problems?

OCCA's Biennial Conference will be held in Edinburgh at the Dragonara Hotel from 26-29 June 1985 under the general title "New substrates, new materials, new problems?"

The Technical Sessions will take place in the Riverside Suite at the Dragonara Hotel on Thursday and Friday, 27 and 28 June.

The Hon. Research & Development Officer (Mr J. R. Taylor) has arranged the following lectures for the Conference:

Session I

Thursday 27 June 9.15 am–12.15 pm

Chairman: Mr J. R. Taylor (Hon. Research & Development Officer)

Keynote address

By Dr G. C. Fettis (ICI Ltd)

The role of adhesion in corrosion protection by organic coatings

By Prof. Funke (Stuttgart University)

Cataonic electrodeposition primers over zinc coated steels (paper presented on behalf of FSCT)

By Dr Clifford Schoff (PPG Industries)

An improved technique for the measurement of adhesion based on a blister method

By Dr Scantlebury and Dr Ebsener (UMIST)

Paint adhesion to galvanized steel surfaces

By Dr Van Ooij & Dr Groot (Akzo & Sikkens Paint Co.)

Formulation of solvent-based paints pigmented with titanium dioxide

By Mr McNutt (DuPont, USA)

Session II

2.00 pm–4.30 pm

Chairman: Mrs A. Gibson (Vice-President, OCCA)

Colour acceptance parameters, HLB values and effect on weathering

By Mr H. G. Stephens (Kemrez, Australia)

Progress in timber finishing in Britain

By Mr R. Woodbridge and Mr E. A. Hilditch (Berger Paints Ltd)

New materials for the furniture industry

By Mr A. J. Sparkes (Furniture Research Association)

Oxo-acetates – new solvents for high solids paint formulation

By Dr M. A. Taylor and Dr Jan de Greef (Esso Chemicals Ltd)

Advances in the technology of water-based inks and coatings for the printing and packaging industries

By Mr G. H. Hutchinson (Croda Ltd)

Session III

Friday 28 June 9.15 am–12.15 pm

Chairman: Dr L. Valentine

Plastics as a painting substrate (paper presented on behalf of the SLF)

By Mr M. Risberg (Wilhelm Becker, Sweden)

Pigments

By Dr Val de Kodar and Dr Eckherl (Ciba-Geigy, Switzerland)

The effect of titanium dioxide pigments on the cure of thermosetting film

By Mr T. Entwistle (Tioxide International)

Novel corrosion inhibitors

By Mr Z. Kalewicz (France)

Internal stress and film formation in emulsion paints (paper presented on behalf of FATIPEC)

By Dr Y. Perera (CORI, Belgium)

Surface study of pigments

By Prof. C. H. Rochester (Dundee University)

Water-based coatings for plastics

(Polyvinyl Chemie, Holland)

Session IV

2.00 pm–4.15 pm

Chairman: A. McKendrick (Chairman, Scottish Section)

Metals and metal ions in pigmentary systems

By Dr W. E. Smith (Strathclyde University)

New developments in self-polishing anti foulings

By Dr C. M. Sghibartz (Joten-Henry Clarke, Norway)

Performance of a novel corrosion inhibiting pigment of low toxicity, in ferrous metal primers,

applied to marginally prepared surfaces

By Mr P. A. Draper (South Africa)

Polyurethane resins in moisture curable surface coatings

By Mr A. H. Hughes (Baxenden)

Recent advances in vinylidene chloride copolymers for use in surface coatings

By Mr B. Elgood (Scott Bader Ltd)

Further summaries of papers and biographies of authors

Paint adhesion to galvanized steel surfaces

Dr W. J. van Ooij and Dr R. C. Groot

The mechanism of paint adhesion deterioration on hot dip galvanized steel during natural weathering has been studied. Variables were, the paint resin, the pigment system, and the source of the galvanized steel. The binder vehicle, the type of anticorrosive pigment and the surface composition of the zinc all had a pronounced effect on the performance of the system since they determined the rate of corrosion of the zinc, as was elucidated by analysis of the paint-zinc interfaces. Chemical degradation of the paint itself was a negligible factor in the adhesion loss mechanism.

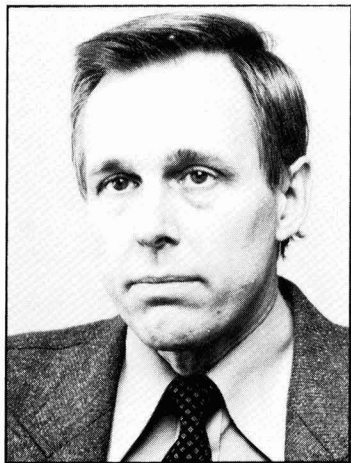
The best zinc system had either a uniform oxide film consisting of largely Al_2O_3 or a ZNO film completely devoid of Al_2O_3 . Irregular Al_2O_3 distributions were found to expedite the corrosion of zinc under paint by an electrochemical mechanism.

Biographies

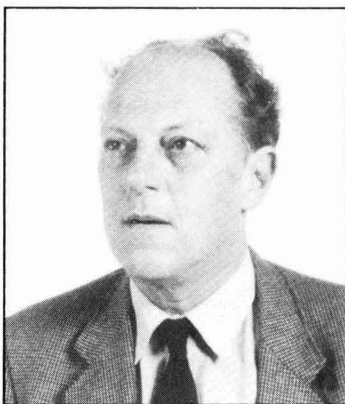
Dr van Ooij obtained his PhD in physical and inorganic chemistry at the technological university at Delft (Neth.).

Subsequently he spent two years at Iowa State University (USA) as a postdoctoral fellow. Since 1973 he is manager of a small research group at the corporate research laboratories of Akzo at Arnhem (Neth.), where he applies surface analysis techniques to industrial problems in such areas as adhesion, corrosion, polymers, coatings, catalysis, fibres, etc.

He has authored or co-authored over 70 scientific papers, holds a number of patents, and has organized international conferences and short courses on applications of surface analysis.



Dr W. J. van Ooij



Dr R. C. Groot

Dr R. C. Groot obtained his PhD in colloid chemistry at the University of Utrecht, carrying out most of the work at the University of Southern California (Chemistry Department). In 1965 he joined Akzo as a research worker in the field of lubrication of textile fibres and headed the research group dealing with lubrication and imparting antistatic properties of textile fibres in 1969. Since 1975 he has been managing research in the coatings industry (Akzo Coatings - Sikkens BV) in the fields of trade sales and is presently managing research in the field of protective coatings.

The role of adhesion in corrosion protection by organic coatings

W. Funke

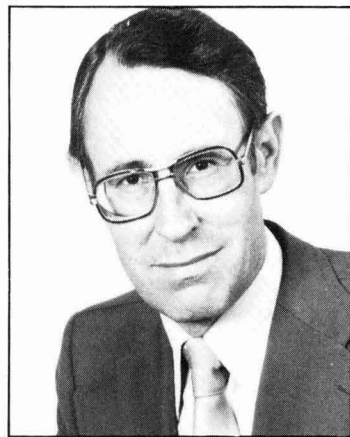
In the role of organic coatings in general, and especially in the corrosion protection of metal surfaces, adhesion in presence of water ("wet adhesion"), is the most important and a decisive property. Adhesion theories provide considerable theoretical background for understanding the basic mechanisms involved, however, they are still of little help in improving adhesion of organic coatings and explaining practical defects. Moreover, statements such as "polar groups are good for adhesion", or "there is no paint film impermeable to water", are so common and much repeated in the literature, that only little notice has been paid to some incongruities existing in the interpretation of the facts in practice.

Considerable experimental evidence indicates, that defects of organic coatings related to metal corrosion, like for example blistering, are always preceded by a significant reduction of adhesion as a consequence of water penetrating into the coating/metal interface. Accordingly, it is very important to measure or evaluate wet adhesion and to apply organic coating systems which resist the interfacial action of water.

As a testing method the simple adhesive tape tear-off test is recommended; and for improving wet adhesion, enhancing the barrier properties of the film, and optimizing the cooperative action of adhesive bonds at the coating/support interface, are suggested.

Biography

Dr Funke received his PhD from the University of Stuttgart in 1956. Since that time he has been an associate of the Research Institute for Pigments and Paints; and since 1969, Professor of Polymer Chemistry at the University of Stuttgart. Dr Funke's research interests include the structure of crosslinked polymers; the synthesis of crosslinked macromolecules and microgels; and the mechanism of cross-linking emulsion polymerisation. In the paint field Dr Funke and his co-workers are studying the mechanisms of coating defects related to metal corrosion; testing corrosion protection by organic coatings, and corrosion protection by the barrier principle.



Professor W. Funke

Cationic electrodeposition primers over zinc-coated steels

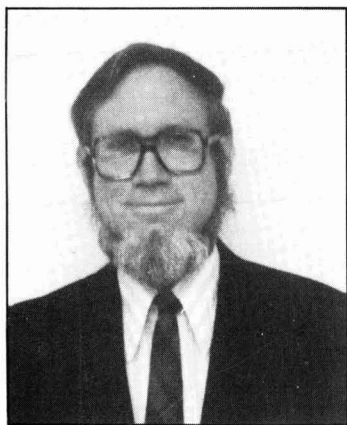
Clifford K. Schoff and Huey-Jyh Chen

The automobile industry is turning more and more to zinc coated steels to improve corrosion protection. Considerable amounts of galvanised and zinc alloy coated steels presently are used in production, and their proportion of the steel contained in an automobile will rise in the future. These surfaces, along with the rest of the vehicle, normally are primed with cationic electrodeposition primers and the results have been excellent. However, from the literature and occasional problems seen in the past, we know that under certain conditions cationic electrodeposition primers can give pits and craters over zinc alloy and galvanized steels. This is of concern to us and to our customers. Therefore, we set out to recreate the problem, determine the mechanism of

defect formation, and devise process or paint changes that would prevent the occurrence of the craters.

We reproduced the problem over a variety of zinc coated steels and applied a wide range of physical and analytical techniques to characterise the defects. Conventional cationic electrodeposition primers have a high tendency to crater, but the newer, high build, good flow primers, are quite resistant to cratering. There are two mechanisms for formation of the defects: electrical discharge (dielectric failure of the wet film) in the bath, and gassing. Defects are associated with the zinc surface, but with high build primers, the craters occur mainly with deposition over the zinc alloys.

Craters can be prevented or greatly reduced by: using high build cationic electrodeposition primers rather than conventional ones; using smoother substrates, and controlling AC ripple so that there are no high voltage spikes.



Dr Clifford K. Schoff

Biographies

Dr C. K. Schoff is a senior research associate and head of the Physical Chemistry Group at the Coatings and Resins Research Center of PPG Industries in Allison Park, Pa. He received his BS and MS degrees in chemistry from the University of Idaho. He holds a PhD in polymer chemistry from the University of St. Andrews and did post-doctoral research at Glasgow University and at Princeton. He has been with PPG since 1974. His research interests include the rheology of fluid paints, the mechanical and thermal properties of paint films, the physical chemistry of cationic electrodeposition, and resin-solvent interactions. He currently is the president of the Pittsburgh Society for Coatings Technology. He is also on the Executive Committee of ASTM Committee D-1 on Paints and Related Materials and is chairman of sub-committee D01.24 on Physical Properties of Fluid Paints.

Dr Huey-Jyh Chen is a Research Associate

in the Physical Chemistry Group at PPG's Research Center in Allison Park, Pa. She received a BS degree in chemistry from the National Taiwan Normal University in Taipei, Taiwan and a PhD in physical organic chemistry from the Illinois Institute of Technology in Chicago. She did post doctoral research at IIT, the University of Toronto, and the University of Chicago. Before coming to PPG in 1979, she did research for Rohm and Haas, Spring House, Pa., and the Carnegie Mellon Institute of Research in Pittsburgh. Her work at PPG has centred on cationic electrodeposition, the development of electrochemical techniques, and the synthesis and testing of corrosion inhibitors.

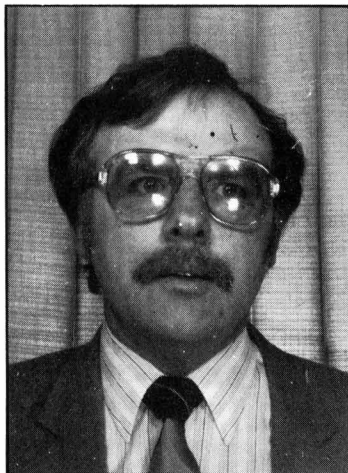
Polyurethane resins in moisture curable surface coatings

A. H. Hughes

The chemistry of moisture curing polyurethanes is covered in brief. The advantages and disadvantages of moisture curable coatings is outlined, followed by examples of the type of application in which these coatings are used. Anti-corrosion coatings are covered in more detail along with vehicle underbody seals, a completely new application. Typical formulations are given for a number of different coatings and the method of manufacture is covered in detail.

Biography

A. H. (Tony) Hughes BSc ATSC, was born in a Lancashire village outside Wigan in 1948. Having attended Wigan Grammar School and studied maths, physics and chemistry at 'A' level, he later obtained a BSc(Hons) in Applied Chemistry from the University of Salford.



A. H. Hughes

He joined Donald Macpherson & Co. Ltd as a technologist in Resin Development in 1972 and progressed via section leader and group leader to technical co-ordinator to the Resin Unit. He left Donald Macpherson in

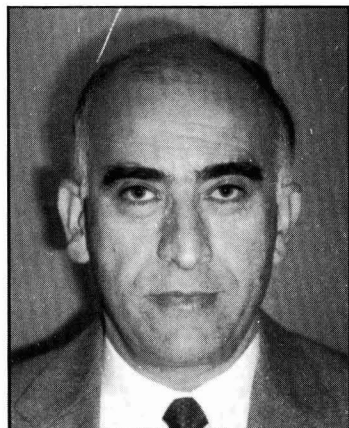
1977 to join the Baxenden Chemical Co. Ltd. as research and development manager and was made technical manager at their newly set-up Speciality Chemicals Division in 1979. This Division has seen spectacular growth as a supplier of urethane and acrylic intermediates to the surface coating, elastomers, building products and adhesive industries. Tony Hughes joined the Manchester Section of OCCA in 1973 and was accepted into the professional grade in 1977.

Internal stress and film formation in emulsion paints

D. Y. Perera

The different stages occurring in the film formation of emulsion paints were examined in the light of the development of internal stress. The development of internal stress (due to coalescence, loss of volatiles) may, in addition to a stress relaxation, also induce filler/binder dislocations, micro(fissures), loss of adhesion, etc. - all processes which affect the film integrity.

The way in which the coating components (pigmentation, organic solvents) affect the development of internal stress, the evaporation kinetics and thus the film formation, was also investigated. The results obtained were explained by considering, on the one hand, the internal stress components (elastic modulus, internal strain, Poisson's ratio) and on the other hand a number of fillers properties (specific surface area, nature of interacting sites), binders (acid/base character, T_g), and solvents (plasticizing effectiveness, molecular structure).



D. Y. Perera

Biography

Dr Dan Y. Perera has been head of department at the Coatings Research Institute (Belgium) since 1971. He received his doctorate degree from the University of Technology (T.H. Delft, The Netherlands). His field of activity and publications include: moisture transport in organic coatings and porous building materials, adhesion, mechanical properties and internal stress.

International Liaison



An Informal Luncheon Meeting of ICCATCI took place during the FSCT Convention at Chicago on 25 October 1984 and shown in the photograph above are (standing; from left to right) Carlos E. Dorris, Howard Jerome, William Mirick, A. Clarke Boyce, William H. Ellis, Joseph A. Bauer, Harry Griffiths (BPVLC) and Frank J. Borrelle. (Seated; from left to right) Toyohiko Yoshida (JSCM), Frank Redman, Donald J. Morris, E. C. (Ted) Saultry (OCCAA), Terry Johnson, Amleto Poluzzi (FATIPEC), Kenjiro Meguro (JSCM) and Robert H. Hamblin. Mr Redman, Mr Morris and Mr Hamblin represented OCCA and the remainder, unless otherwise designated, were from FSCT

Report of Council meeting

A meeting of the Council took place at 2.00 pm on Wednesday 31 October 1984 at the Great Northern Hotel, King's Cross, London N1, with the President, Mr C. N. Finlay, in the chair. There were 27 members present.

Council learnt with regret of the death of Mr E. Armstrong, a former member of Council who had twice been Chairman of the Hull Section and had received a Commendation Award.

The President welcomed to their first Council meeting Mrs E. Stretton, Mr R. Barrett and Dr C. Crawford and it was noted that Mr J. Inshaw had replaced Mr R. Wright as the Thames Valley Section Representative.

It was reported that Mr F. Morpeth had agreed to serve as Chairman of the Exhibition Committee and that Mr A. Lageu would be the Association's nominee on BSI Committee BDB/7/2.

Details were given of the arrangements for the Association's Conference at Edinburgh 26-29 June 1985, including the technical papers for the four sessions and registration fees. It was hoped that the brochure giving the list of lectures and the social programme, together with the registration form would be despatched to members in the New Year and that full details of summaries of papers and biographies of lecturers would be appearing in the January 1985 issue of the *Journal*. The chairmen of the Northern Sections were asked to consider if it would be possible to arrange coach parties from their sections for those wishing to apply for daily registration; a note would be added to the registration

form so that members wishing to avail themselves of this facility could indicate this when registering for the Conference. Arrangements were in hand for over-night accommodation at the Pollocks Halls of Residence.

The half-year accounts and estimates for the second half of the year were adopted by the Council and it was reported that the subscription rate for non-members wishing to purchase the *Journal* for company libraries etc. had been raised to £60 and that increases had been made in the advertising rates for the *Journal*.

Details were given of the removal from the Register of the names of members still in arrears with their subscriptions and Council were pleased to note that the total removed was less than in October 1983.

It was reported that Mr G. Robson, Chairman of the Manchester Section had, in the course of his business activities in the Far East, compiled a list of potential members and companies who might be interested in subscribing to the *Journal* and had passed this to the Director & Secretary. The President thanked Mr Robson for the considerable trouble which he had taken in this matter and hoped it would lead to increased membership in the Far East.

It was reported that the Second Volume of the Surface Coatings Manual prepared by OCCA Australia, would be available shortly and that details and application form had appeared in the Association's *Journal*.

Council were reminded that the Jordan Award would normally be presented at the

Edinburgh Conference and that applications by members under 35 years of age should be sent to the Director & Secretary by the end of 1984.

It was reported that one Associate had been transferred to Fellowship and two Associates had been admitted to the Professional Grade.

Reports were received from sections on their activities. The President reported on his visit to the South African Division Symposium and to the FATIPEC Congress at Lugano.

It was reported that it had been agreed with the South African and New Zealand Divisions that since the Vice Presidents resident in these two countries were automatically Chairman of the Division concerned, that their term of office should be marked by a Past Divisional Chairman's badge. This would also apply to those Vice Presidents who had already served in this capacity.

The President reported on his discussions at Lugano with FATIPEC representatives since an earlier discussion had taken place at OCCA 35 regarding the possibility of the Association and FATIPEC joining in an exhibition to be held at the same time as the next FATIPEC Congress at Venice in late September/early October 1986. Council approved the suggestion, subject to satisfactory arrangements being made. This did not preclude the holding of the Association's biennial exhibition earlier in 1986, though it was agreed with FATIPEC that it would not be held in London. Mr Morpeth reported on the discussion which had taken place at the Exhibition Committee and the possibilities which were being investigated by the Director & Secretary and

himself for an exhibition to be held in May 1986 at a provincial centre.

It was reported that lectures were being arranged in the New Year on career opportunities etc. at universities in the West Country in which the Association would participate.

There being no other business, the President thanked members for their attendance and declared the meeting closed at 4.40 pm.

London Section

Ladies Night

The London Section Ladies Night was held on Friday 7 December 1984 at the Royal Chace Hotel, Enfield. Some two hundred and twenty members and guests attended.

The Section's top table guests were: the President (Mr Cecil Finlay) and Mrs Finlay; Mr Alan Stacey (President of Mitcham Polymer Club) and Mrs Stacey; Mr Roland Staples (President of the Birmingham Paint Varnish and Lacquer Club) and Mrs Staples; Mr Robert Hamblin (Director & Secretary); the Chairmen of Bristol; Manchester, Midlands, Newcastle and West Riding sections and their ladies, and long serving London Committee member Mr Herbert Worsdall and guest.

After an excellent meal, the Section Chairman, David Sharp, proposed the Toast to the "Ladies and Guests", the response being in the capable hands of the President.

Following the speeches, the President conferred Honorary Membership on Herbert Worsdall for services to the Association. (See separate report.) The Section Chairman said that it was fitting that the award was made at a function that Herbert had organised until recent years. In a brief speech Herbert thanked the Association and section for the Honour.

After the formalities dancing commenced and continued until 1 am.

A tombola organised by Karen Canterford (wife of the Section's Social Secretary) proved most successful.

Just before "Auld Lang Syne" a presentation was made to Barbara Sharp, the Section Chairman's wife in recognition of her support to David during his chairmanship.

B. A. Canterford

News of Members

Honorary Membership conferred upon Herbert Worsdall

As reported in the account of the London Section's Ladies' Night, Honorary Membership of the Association was conferred upon Herbert Worsdall, a long

serving and well known member of the Association at both Association and Section levels for his outstanding contribution to the Association over a long period.



Shown above are (left to right) Mr R. H. Hamblin (Director & Secretary), Mr C. N. Finlay (President), Mr H. C. Worsdall and Mr F. D. H. Sharp (Chairman, London Section)

The scroll of Honorary Membership reads as follows:

At the meeting of the Council held on 22 February 1984, it was unanimously agreed to confer Honorary Membership upon Herbert Charles Worsdall, in recognition of the outstanding service given by him both to the Association and to the London Section, of which he was Chairman (1957-59). He also served the Section at various times as Honorary Secretary, Honorary Social Secretary and Honorary Programmes Officer, as well as serving on its Committee.

His service on the Council has been in many capacities - as Representative of both the London and Ontario Sections, Elective Council Member, Vice President and on Committees and Working Groups of the Council, including the Exhibition and Technical Education Committees and the Technical Education Working Group.

The conferment of Honorary Membership thus expresses the admiration of the members for the way in which he has at all times discharged his duties and upheld the prestige of this Association.

President Honorary Secretary
Director & Secretary

Midland Section

Golf Competition

The Midland Section held a golf competition at Maxstoke Park Golf Club on Tuesday 2 October 1984. Twenty-seven members and guests enjoyed a good days golf with Mr Sid Harris becoming the first winner of the Chairman's Trophy kindly donated by the immediate past Chairman of the Midland Section Mr D. Kimber. Various companies

donated prizes and all the JOCCA players received a prize.

The prizes were presented after dinner and the Chairman Mr E. Wallace proposed a vote of thanks to the staff and the club professional of Maxstoke Park Golf Club.

B. E. Myatt

Obituaries

Dr M. Ellinger

J. R. Taylor writes:

It is with some sadness that the death on 9 October 1984 of Dr Marianne Ellinger is recorded. She was a keen supporter of OCCA for some 25 years after she took residence in the UK.

Marianne Ellinger (nee Gardonyi) was the daughter of a successful mechanical engineer. She was born in Hungary in 1915 and enjoyed a happy childhood and adult life until 1939. She obtained a PhD Degree in Chemistry at the P. Pazmany University of Science, Budapest, and married a fellow student who owned a paint factory, where they worked in their early married life.

During the 1939-45 war years she lost both her brother and husband in a concentration camp. The paint factory was confiscated by the authorities and not returned after the war. She was employed as head of technical service for the Hungarian Paint Industry in 1946.



Dr M. Ellinger

On coming to the UK in 1956, she later obtained British citizenship and often remarked on the benefits of life in the UK. She joined GIPaints, now Ault and Wiborg Paints Ltd, as research chemist and was subsequently employed in the Electrodeposition Laboratory at Perivale.

Marianne Ellinger was a most keen paint technologist and was a member of the London Section of OCCA. She was a regular attender at local meetings and she had given papers at both OCCA and FATIPEC conferences. She acted as the European Representative of DSET Weathering Organisation and travelled extensively world-wide. She spoke several

languages fluently and acted as a technical translator to several technical journals. She retired from Ault and Wiborg Paints Ltd but was retained as a consultant until the time of her death.

She believed in physical fitness and was a keen swimmer. It is tragic that in her last year of life a stroke paralysed her and prevented her from speaking. Even during this period in hospital the writer can vouch for her continued interest in OCCA activities since she attempted to read JOCCA and

often indicated her approval or disapproval of a paper.

In her death Dr Ellinger has remembered OCCA and has bequeathed a sum of money from her estate to be set aside in memory of her husband and father to assist junior members of OCCA and to be known as the "Ellinger-Gardonyi Award".

Dr Ellinger did not leave a family but will be missed by members of OCCA who had the privilege of knowing her.

Mr Joe Kidd

It is with regret that we record that Joe Kidd of Cape Section, an Ordinary member since 1965, died of a severe heart attack.

Joe was well known and respected throughout both the industry and the Association in South Africa and will be much missed.

CLASSIFIED ADVERTISEMENTS

APPOINTMENTS

SURFACE COATING PRODUCTS

South of England

A successful medium sized company, active in the coatings industry, is seeking a technically experienced person to lead its expansion in this field.

This is a ground floor opportunity and it is anticipated that you will be totally responsible for the technical aspects of the paintmaking business. The development and successful introduction of new products will be prime objectives.

Applicants should have current experience of industrial paintmaking in the United Kingdom and be familiar with recent advances in the Industry. A high level of customer involvement will be required and knowledge of product application and problem solving is essential. It is unlikely that anyone under the age of 30 will have the necessary experience.

Conditions of employment are excellent and the rewards and prospects will be decided entirely by your own abilities and success.

Please write with full c.v. and details of current salary to:

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HEMPEL'S MARINE PAINTS (SAUDI ARABIA) W.L.L. LABORATORY MANAGER

We are the largest paint company in Saudi Arabia, with an extensive line of paints and speciality coating serving trade-sales, industrial finishes and marine segments.

The candidate must have the following qualifications:

1. B.S. with major in chemistry
2. Minimum eight years of experience in paints and coatings industry
3. Strong product development background in trade sales paints
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We offer:

1. Tax free salary of 30,000 US dollars, per annum
2. Two nineteen days each paid vacation for family including air fare
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6. Minimum three years contract extendable for additional two years

Submit an extensive resume to **Mr Per Berthelsen, Corporate Personnel Department, Hempel's Marine Paints, Skibsfarve-Fabrik A/S, PO Box 280, Lundtoftevej 150, Denmark.**

CONSULTANCY SERVICES

Consultancy and Translation Services

A range of advisory services is available in the fields of Adhesives, Surface Coatings, Flexible Abrasives, and Health and Safety.

Technical Translations also provided from the French.

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Telephone Potters bar (0707) 53118

Consultancy offered

A well qualified materials protection consultant has a limited amount of time available. Experienced in formulation and testing of protective systems and also in inspection both in the UK and overseas.

Box Number 548

APPOINTMENTS

Research and Development Chemist

George M. Whiley is a leading manufacturer of metallic and pigment hot stamping foils which are used on a wide range of consumer and industrial goods.

Based in a custom-built factory at Livingston only ten miles from Edinburgh, we now require a Chemist to join our small Research and Development team. Reporting directly to the Technical Director, you will undertake laboratory and pilot production work on new products and product improvements. This will include investigating and assessing new materials, coating phenomena and manufacturing problems. This wide remit demands a combination of laboratory work and production involvement in this highly specialised field.

Essentially candidates will be experienced in ink, paint or adhesive technology and have at least 3-4 years post HNC or Graduate industrial experience. In addition a familiarity with laquer coating techniques, pigments and resins, would be advantageous.

We offer an attractive benefits package including a salary negotiable upon experience and ability, together with real career potential. If you require to move to the Edinburgh area, which enjoys a wealth of social, cultural, housing and educational facilities, then generous relocation assistance is available.

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