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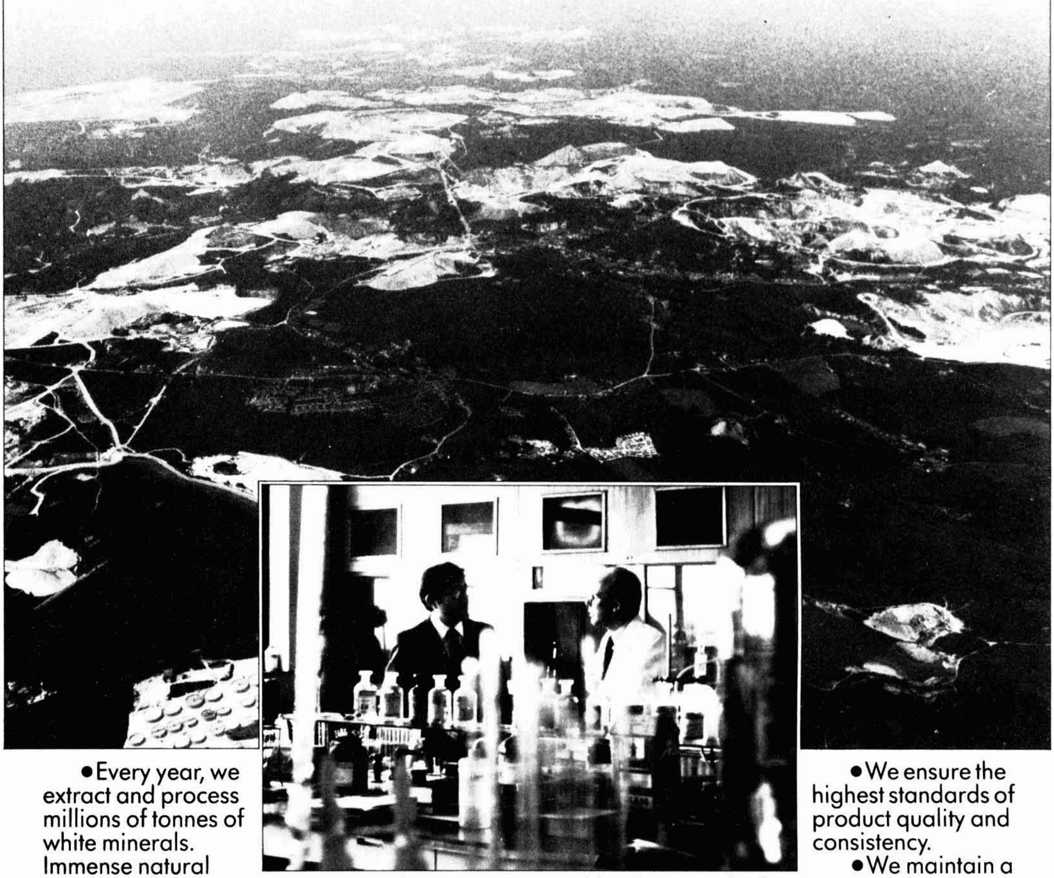
- New corrosion inhibitors for surface coatings based on fatty materials. Part I: laboratory evaluation
- Physico-chemical characterisation of aqueous shellac solutions and hydrosols for encapsulation
- Aspects of galvanic action of zinc-rich paints. Electrochemical investigation of eight commercial primers



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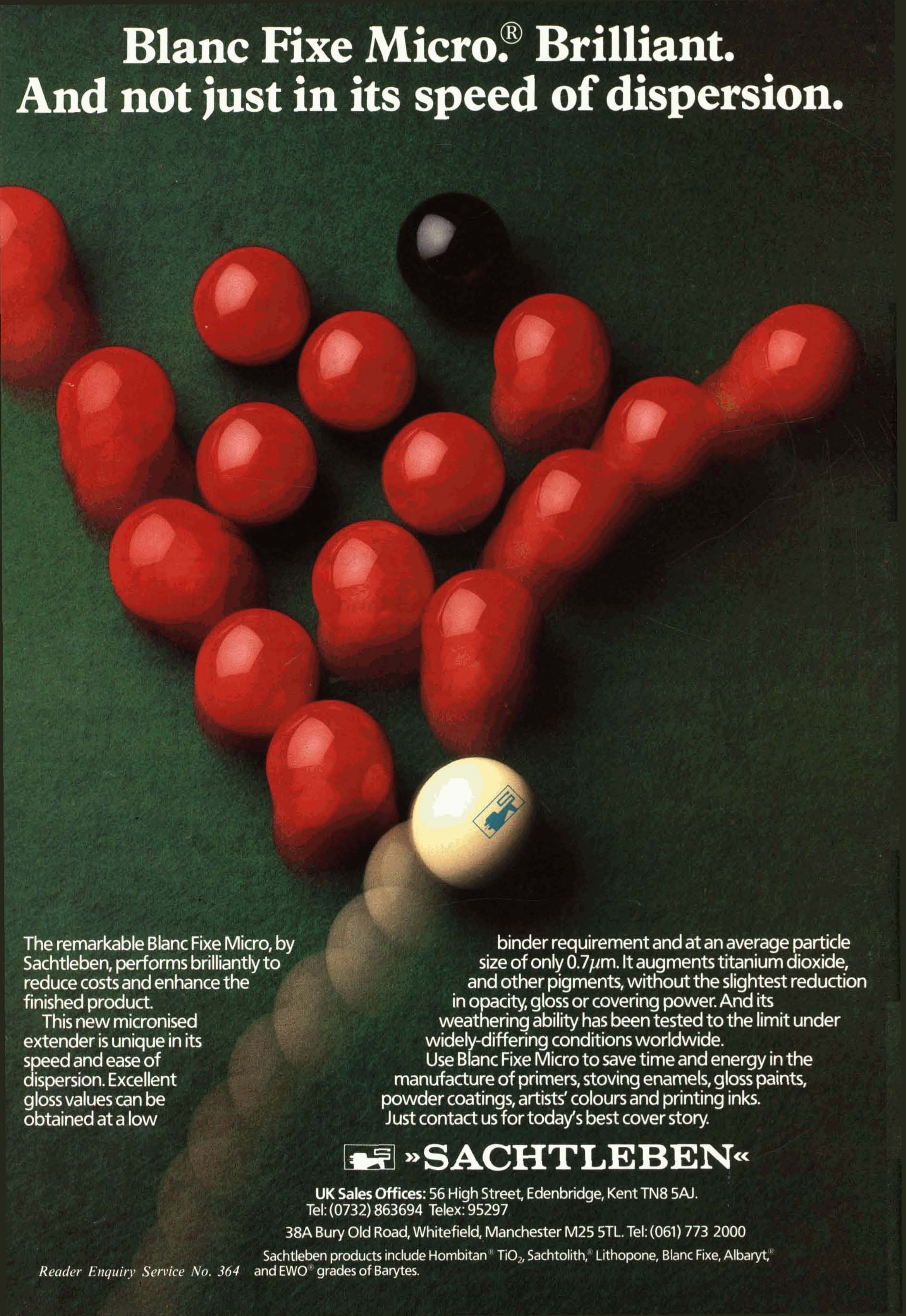
The list of Council members, committees of Council and section and branch committees for the 1984-85 session was given in full in the August 1984 issue of the Journal *following the appointment of Council at the AGM held on 13 June 1984 and of the committees of Council at the Council meeting on 11 July 1984. For ease of reference the names and addresses of the current Honorary Secretaries are shown above for both members and non-members wishing to contact any of the sections or branches.*



New corrosion inhibitors for surface coatings based on fatty materials. Part I: laboratory evaluation

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Laboratory of Polymers and Pigments, National Research Centre, Dokki, Cairo, Egypt

Summary

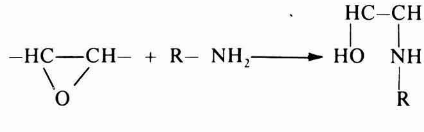
Linseed oil was epoxidized *in-situ* under well established conditions. The resulting epoxidized linseed oil was reacted with aliphatic amines (methyl amine, ethyl amine, propyl amine and butyl amine) and aromatic amines (aniline, o- ,m- ,p-chloroaniline, m- ,p-toluidine and o- ,m- ,p-anizidine) in sealed ampoules under an inert atmosphere at 130°C. The compounds so prepared were evaluated as corrosion inhibitors for surface coatings. It was found that aliphatic amine derivatives were superior in this respect and that their efficiency increased with decrease in chain length of the alkyl group. With respect to aromatic amine derivatives, their efficiency was directly related to the electron density on the nitrogen atom of the amino group.

Introduction

The presence of solid particles in anticorrosive paints may have various effects. Some are favourable, as in the case of zinc particles in the metal pigmented paints; others are potentially unfavourable, for instance if diffusing oxygen is prevented from reaching the point of contact between the pigment particles and the steel substrate, whilst being allowed access elsewhere, conditions favourable to a corrosion cell are set up. Many trials have been carried out to investigate the possibilities of unpigmented lacquers with inhibitive properties. Barouch[1] introduced a number of inhibitors into a medium oil alkyd resin. These inhibitors were N-dibenzyl amine, zinc naphthenate and heterocyclic tertiary amines. Rosenfeld[2] found that organic nitrites or chromates gave promising results. Many other organic compounds have been prepared and evaluated as corrosion inhibitors[3-5].

In the present work, new inhibitors were prepared from a cheap local source – linseed oil. Linseed oil was epoxidized *in-situ* with hydrogen peroxide and acetic acid, using Dowex 50W-8X as a catalyst[6]. The resulting epoxidized linseed oil was reacted with aliphatic and aromatic amines in sealed ampoules under an inert atmosphere[7] (nitrogen) at 130°C.

(a) With aliphatic amines:

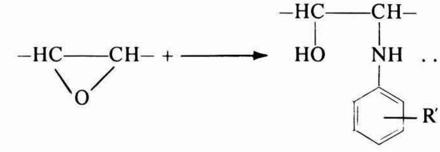


..... (1)

Where R = CH_3, C_2H_5, C_3H_7 or C_4H_9

The resulting products were viscous and yellow in colour. The intensity of colour increased with the chain length of the alkyl group (R).

(b) With aromatic amines:



....(2)

Where R′ = H, Cl, CH_3 or OCH_3

The resulting compounds were viscous. Their colour ranged from dark yellow to dark brown.

Reactions (1) and (2) stop at the formation of the secondary amine because of steric hinderance[8,9]; these were checked for by volumetric and NMR techniques[9].

Some of these compounds were evaluated, by the author, as corrosion inhibitors for overhead distillation towers, and it was found that the aromatic derivatives (except p-chloroaniline derivatives) gave better results than commercial ones[10,11].

The aim of the present work is to evaluate 13 compounds prepared as corrosion inhibitors for surface coatings.

Experimental

Materials

1. Linseed oil – alkali refined, IV 173.
2. Dowex 50W-8X – a sulphonated poly(styrene-divinyl benzene), dark yellow colour, particle size – 20+50 mesh.
3. Hydrogen peroxide – its strength was precisely determined by the sodium thiosulphate method[12] and was found to be 30 per cent.
4. Amines – both aliphatic and aromatic amines are of pure grade.
5. Resins:
    - (a) Laroflex MP 35 BASF, Ludwigshafen, Germany. It is a copolymer of vinylchloride containing about 25 per cent vinyl isobutyl ether, K value 32.36, chlorine content 42 ± 2.2 per cent, specific gravity 1.24 ± 0.03 and particle size 75 μm.
    - (b) Coumarone indene resin, R. H. Cole Ltd, England.
    - (c) Phenolic resin, Wresingl 265 (R552), Resinous Chemicals Ltd, England. It is a pure oil-soluble alkyl-phenol-formaldehyde resin, specific gravity 1.1.

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*El-Nasr Bottling Co., Dokki, Cairo, Egypt

(d) Rosin-modified phenolic resin, Wresinite 1(R 201), Resinous Chemicals Ltd, England.

6. Plasticizer – chlorinated paraffin N50, BASF, Germany.

7. Artificial sea water[13]. A representative sample was prepared by dissolving the following quantities of chemicals in one litre of water:

Component	Weight
Sodium chloride	27.26
Magnesium chloride	3.51
Magnesium sulphate	1.84
Potassium chloride	0.69
Sodium bicarbonate	0.11
Potassium bromide	0.09
Calcium sulphate	1.29

Techniques

1. Preparation of laboratory inhibitors – linseed oil was epoxidized *in-situ* with hydrogen peroxide and acetic acid, using Dowex 50W-8X as a catalyst[6]. The oxirane oxygen content of the epoxidized linseed oil was measured volumetrically, using HBr in acetic acid[14,15], and checked spectroscopically, by NMR[16]; it was found to be 5.5 per cent. The resulting epoxidized linseed oil was reacted with aliphatic and aromatic amines, under well established conditions[9], to explore the possibility of using these compounds as corrosion inhibitors.

2. Preparation of test panels – steel and glass panels were prepared for different tests according to ISO 1514/1983.

3. Viscosity[17] – the viscosity of the prepared varnishes were measured by Ford cup No. 4.

4. Drying time – the films were tested to touch dry[18]. The same films were used for surface dry tests[19].

5. Impact resistance[20] – the Gardner Impact tester was used.

6. Film thickness – thicknesses of films on steel panels were measured by a magnetic thickness gauge[21], while those on glass plates were measured using an Erichsen Thickness Gauge – 233.

7. Acid and alkali resistance tests – glass plates were used for both tests. In acid resistance tests, hydrochloric acid was used[23]. In alkali resistance tests, sodium hydroxide was used[24,25].

8. Corrosion scratch test[26] – 5 cm × 10 cm mild steel panels were degreased and then sanded. The panels were coated and left to dry for seven days. They were edged with wax and one face of each panel was scratched to the substrate with a sharp blade. The panels were exposed to synthetic sea water for 670 hours then washed with distilled water and dried. The amount of rusting on each panel was observed. The adhesion along the groove was determined in the usual manner.

9. Hardness – the Konig Pendulum Instrument was used. The hardness of the films was measured at 30 μm.

10. Adhesion test – carried out according to ANSI/ASTM D 3359 – 1976.

11. Water uptake[27,28] – plastic test panels were used. The coated test panels were immersed in tap water for 5, 9, 16, 20, 25 or 32 days. At the end of each period the panels were shaken and gently dried with filter paper, then weighed. The panels were then placed in a desiccator and constant weight was achieved.

12. Weight loss measurements[29] – steel panels of dimensions 3 cm × 1 cm were used. The weighed coated panels were immersed in a reaction chamber which contained sea water. After the specified periods, the panels were reweighed after removing the varnish film and corrosion products. The loss in weight was calculated in mg/g.

Table 1

Formula index of laboratory inhibitors (reaction products of epoxidized linseed oil with aliphatic and aromatic amines)

Amine	Index
Methyl	EP–L–MA
Ethyl	EP–L–EA
Propyl	EP–L–PA
Butyl	EP–L–BA
Aniline	EP–L–An
o-Chloro-aniline	EP–L–o–CA
m-Chloro-aniline	EP–L–m–CA
p-Chloro-aniline	EP–L–p–CA
o-Anizidine	EP–L–o–Az
m-Anizidine	EP–L–m–Az
p-Anizidine	EP–L–p–Az
m-Toluidine	EP–L–m–Tn
p-Toluidine	EP–L–p–Tn

Results and discussion

Linseed oil was epoxidized *in-situ* under well established conditions. The resulting epoxidized linseed oil (5.5 per cent oxirane oxygen content) was reacted with aliphatic (methyl, ethyl, propyl and butyl) amines and aromatic (aniline, o- ,m- and p-anizidine, m- and p-toluidine and o- ,m- and p-chloroaniline) amines. The reactions were carried out in sealed ampoules at 130°C under an inert atmosphere. The resulting compounds (13 in all) were added to neutral and acid media for evaluation as corrosion inhibitors. The formulae of the compounds are given in Table 1.

Preparation of anticorrosive varnishes

Anticorrosive varnishes were prepared by dissolving the chosen resins (a vinyl copolymer (Laroflex), coumarone indene, a phenolic resin and rosin/phenolic), in suitable solvents. The laboratory inhibitors were also dissolved in suitable solvents, taking into consideration the compatibility of the solvents used for the above mentioned resins with those used for inhibitors. Resins were blended, then thoroughly mixed and the required amounts of the laboratory inhibitors were added to prepare anticorrosive varnishes of suitable consistency for brush application. Typical formulations are shown in tables 2-5.

The formulated varnishes can be classified into four main groups, each of these can be classified into subgroups, according to the type of the inhibitor used.

Group I

This group comprises 16 varnishes and it is divided into four subgroups. The formulations of this group are given in Table 2. Chlorinated paraffin was used as a plasticizer. The ratio of Laroflex to coumarone indene used was 2.7:1, the result being a neutral medium.

The inhibitors used in this group were of the aliphatic type; they were the reaction products of epoxidized linseed oil with methyl, ethyl, propyl and butyl amines.

Table 2

Varnish composition of group I (neutral medium containing aliphatic inhibitors)

Varnish No.	Subgroup 1				Subgroup 2				Subgroup 3				Subgroup 4			
Composition	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Vinyl copolymer	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27
Plasticizer	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Coumarone indene	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
EP–L–MA (mg/100 g)	2.5	5.0	7.5	10.0												
EP–L–EA (mg/100 g)					2.5	5.0	7.5	10.0								
EP–L–PA (mg/100 g)									2.5	5.0	7.5	10.0				
EP–L–BA (mg/100 g)													2.5	5.0	7.5	10.0

Table 3

Varnish composition of group II (neutral medium containing aromatic inhibitors)

Varnish No.	Subgroup 1				Subgroup 2				Subgroup 3				Subgroup 4				Subgroup 5				Subgroup 6				Subgroup 7				Subgroup 8				Subgroup 9			
Composition	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52
Vinyl copolymer	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27	27
Plasticizer	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Coumarone indene	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
EP–L–An (mg/100 g)	2.5	5.0	7.5	10																																
EP–L–p–Tn (mg/100 g)					2.5	5.0	7.5	10																												
EP–L–m–Tn (mg/100 g)									2.5	5.0	7.5	10																								
EP–L–o–Az (mg/100 g)													2.5	5.0	7.5	10																				
EP–L–p–Az (mg/100 g)																	2.5	5.0	7.5	10																
EP–L–m–Az (mg/100 g)																					2.5	5.0	7.5	10												
EP–L–o–CA (mg/100 g)																									2.5	5.0	7.5	10								
EP–L–p–CA (mg/100 g)																													2.5	5.0	7.5	10				
EP–L–m–CA (mg/100 g)																																	2.5	5.0	7.5	10

Table 4

Varnish composition of group III (acidic medium containing aliphatic inhibitors)

Varnish No.	Subgroup 1				Subgroup 2				Subgroup 3				Subgroup 4			
Composition	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68
Phenolic resin	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30
Rosin-phenolic	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
EP–L–MA (mg/100 g)	2.5	5.0	7.5	10.0												
EP–L–EA (mg/100 g)					2.5	5.0	7.5	10.0								
EP–L–PA (mg/100 g)									2.5	5.0	7.5	10.0				
EP–L–BA (mg/100 g)													2.5	5.0	7.5	10.0

Group II

The same resin blend (in the same ratio) was used in preparing the 36 formulations of this group, i.e. they contained the same neutral medium of group I but they varied in the type of experimental inhibitor (Table 3). The inhibitors examined were the reaction products of epoxidized linseed oil with aniline, m- and p-toluedine, o- ,m- and p-anizidine and o- ,m- and p-chloroaniline.

Group III

Other resin blends, of an acidic medium, were used for preparing the 16 formulations of this group. They are shown in Table 4. The resins used were a phenolic resin and a rosin-modified phenolic resin in the ratio 3:1, respectively. The laboratory inhibitors used in this group are of the aliphatic type, as in group I. The aim of using an acidic medium was to examine the performance of the aliphatic inhibitors in the protection of steel.

Group IV

The formulations of this group number 36 and are shown in Table 5. In this group of varnishes, the same blend of resins used in group III was employed but aromatic inhibitors, as in group II, were added. The object of examining these formulations was to study the corrosion protection afforded by these inhibitors to steel in the presence of an acidic vehicle.

The laboratory tests (acid resistance, alkali resistance,

Table 5

Varnish composition of group IV (acidic medium containing aromatic inhibitors)

Varnish No.	Subgroup 1				Subgroup 2				Subgroup 3				Subgroup 4				Subgroup 5				Subgroup 6				Subgroup 7				Subgroup 8				Subgroup 9			
Composition	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104
Phenolic resin	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30	30
Rosin-phenolic	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
EP–L–An (mg/100 g)	2.5	5.0	7.5	10																																
EP–L–p–Tn (mg/100 g)					2.5	5.0	7.5	10																												
EP–L–m–Tp (mg/100 g)									2.5	5.0	7.5	10																								
EP–L–o–Az (mg/100 g)													2.5	5.0	7.5	10																				
EP–L–p–Az (mg/100 g)																	2.5	5.0	7.5	10																
EP–L–m–Az (mg/100 g)																					2.5	5.0	7.5	10												
EP–L–o–CA (mg/100 g)																									2.5	5.0	7.5	10								
EP–L–p–CA (mg/100 g)																													2.5	5.0	7.5	10				
EP–L–m–CA (mg/100 g)																																	2.5	5.0	7.5	10

Table 6

Test results of neutral varnish films containing aliphatic inhibitors (group I)

Varnish No.	Acid resistance	Alkali resistance	Corrosion scratch	Hardness (s)	Adhesion
1- 4	passed	passed	A*	80-83	good adhesion
5- 8	passed	passed	A	85-88	good adhesion
9-12	passed	passed	A	88	good adhesion
13-16	passed	passed	A	100-105	good adhesion

*Rust was in the groove only, without any spreading under the film. The film had good adhesion along the groove sides and no film defects.

Table 7

Test results of neutral varnish films containing aromatic inhibitors (group II)

Varnish No.	Acid resistance	Alkali resistance	Corrosion scratch	Hardness (s)	Adhesion
17-20	passed	passed	A*	75	good adhesion
21-24	passed	passed	A	72	good adhesion
25-28	passed	passed	A	93	good adhesion
29-32	passed	passed	A	97	good adhesion
33-36	passed	passed	A	90	good adhesion
37-40	passed	passed	A	93	good adhesion
41-44	passed	passed	B†	102	good adhesion
45-48	passed	passed	B	96	good adhesion
49-52	passed	passed	B	103	good adhesion

*Rust was in the groove only, without any spreading under the film. The film had good adhesion along the groove sides and no film defects.
†Steel surface had tarnished under the varnish film and had very slight rust spots, in addition to rust in the groove. No loss of adhesion was observed.

corrosion scratch test, adhesion and hardness) were performed on all the varnishes. The results obtained are summarized in tables 6-9. These tables indicate that the prepared varnishes (neutral and acidic) have good film properties, they are hard, possess high adhesion on steel surfaces and have good acid and alkali resistance.

With respect to the corrosion scratch test, it was clear that the films of both group I (neutral vehicle containing aliphatic inhibitors) and group II (neutral vehicle containing aromatic inhibitors) possessed good corrosion protection except those varnishes containing EP-L-o-CA, EP–L–m–CA and EP–L–p–CA in all concentrations, since tarnishing of the steel surface under the film and very slight rust spots near the scratch were observed. On the other hand, the results of scratch tests on group III (aliphatic inhibitors in acidic medium) show that films containing EP–L–MA and El–L–EA inhibitors were capable of protecting steel against corrosion at all concentrations (2.5-10 mg/g varnish), while those containing EP–L–PA showed limited efficiency in protecting steel, especially at inhibitor concentrations less than 7.5 mg/100 g of varnish. Films containing EP–L–BA failed to protect steel in all concentrations. The results of the tests of group III lead to the conclusion that the chain lengths of aliphatic amines play an important role in their efficiency in an acid medium, i.e. the efficiency of inhibition decreases with increasing chain length.

Table 8

Test results of acidic varnish films containing aliphatic laboratory inhibitors (group III)

Varnish No.	Acid resistance	Alkali resistance	Corrosion scratch	Hardness (s)	Adhesion
53-56	passed	passed	A*	78	good adhesion
57-60	passed	passed	A	76	good adhesion
61-62	passed	passed	B†	76	good adhesion
63-64	passed	passed	A	77	good adhesion
65-68	passed	passed	B	74	good adhesion

*Rust was in the groove only, without spreading under the film. The film had good adhesion along the groove sides and no film defects. †Steel surface had tarnished under the varnish film and had very slight rust spots, in addition to rust in the groove. No loss of adhesion was observed.

Table 9

Test results of acidic varnish films containing aromatic laboratory inhibitors (group IV)

Varnish No.	Acid resistance	Alkali resistance	Corrosion scratch	Hardness (s)	Adhesion
69-72	passed	passed	B†	77	good adhesion
73-76	passed	passed	B	75	good adhesion
77-80	passed	passed	C‡	70	good adhesion
81-84	passed	passed	B	78	good adhesion
85-88	passed	passed	B	73	good adhesion
89-92	passed	passed	B	71	good adhesion
93-96	passed	passed	C	76	good adhesion
97-100	passed	passed	C	77	good adhesion

†Steel surface had tarnished under the varnish film and had very slight rust spots, in addition to rust in the groove. No loss of adhesion was observed. ‡Corrosion occurred on steel surface under the film in the form of spots.

In the case of group IV (acidic medium and aromatic inhibitors), Table 7, films containing EP–L–An, EP–L–p–Tn, EP–L–o–Az, EP–L–p–Az and EP–L–m–Az inhibitors have only limited corrosion protection for steel, while varnish films containing EP–L–m–Tn, EP–L–o–CA, EP–L–p–CA and EP–L–m–CA show the least level of corrosion protection. The results also show that the electron density on the nitrogen atom of the amine plays an important role in the efficiency of the corrosion inhibitors.

Figures 1 and 2 show the time depedence of water uptake of the neutral and acidic varnish films containing aliphatic and

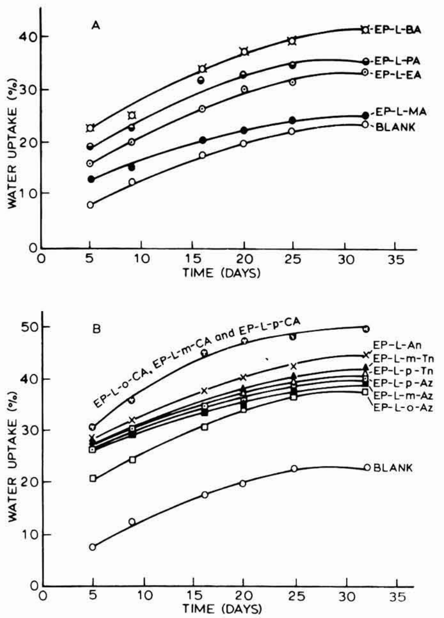

Figure 1. The dependence of water uptake of neutral varnish films containing aliphatic and aromatic inhibitors on time

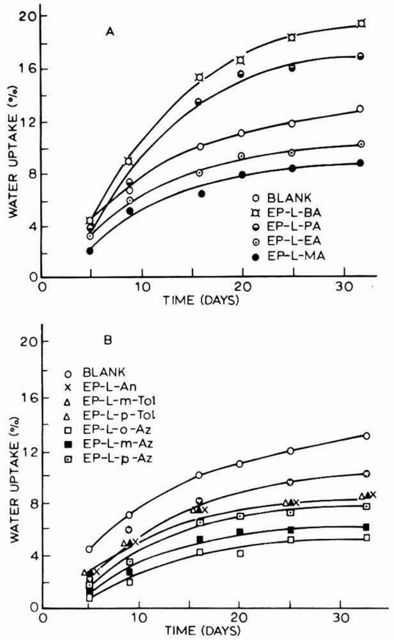

Figure 2. The dependence of water uptake of acidic varnish films containing aliphatic and aromatic inhibitors on time

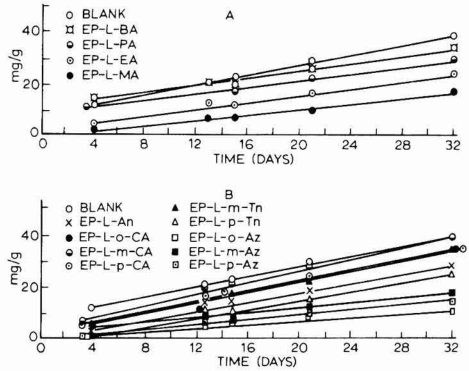

Figure 3. The dependence of weight loss of steel panels coated with neutral varnish films containing aliphatic and aromatic inhibitors on time

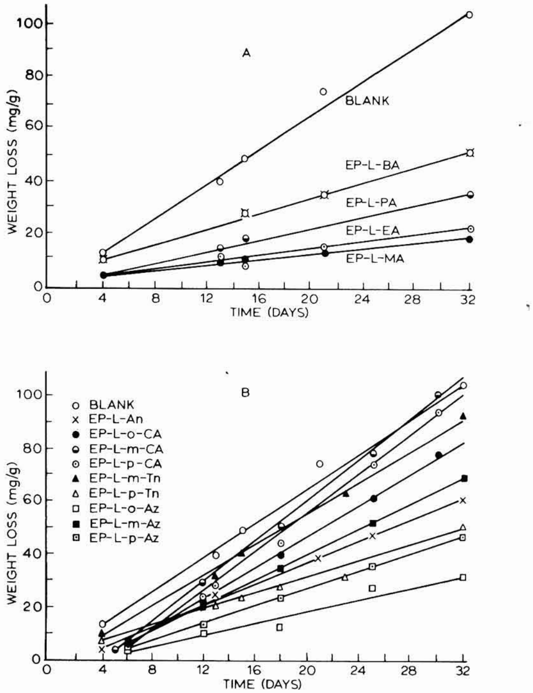

Figure 4. The dependence of weight loss of steel panels coated with neutral varnish films containing aliphatic and aromatic inhibitors on time

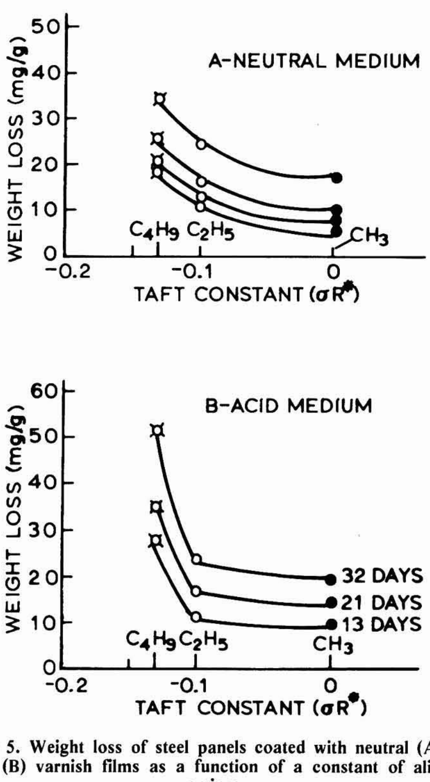

Figure 5. Weight loss of steel panels coated with neutral (A) and acidic (B) varnish films as a function of a constant of aliphatic amines

aromatic inhibitors. An initial examination of the graphs of figures 1 and 2 shows that the neutral varnishes (groups I and II) asborb much more water than the acidic ones (groups III and IV). However, much better results for corrosion protection were obtained on using the varnishes of groups I and II. This means that water uptake alone is not a true measure of corrosion protection. Also, it can be seen that not only does the type of varnish affect water uptake but also that the type of inhibitor influences this characteristic, as can be seen from the blank curves and the other curves of both figures. With respect to aliphatic amines (figures 1a and 2a), water uptake correlates with the fatty chain length of the amine and this phenomenon is dependent on time. Also, the electron density on the nitrogen atom of the aromatic amine (figures 1b and 2b) plays an important role in the water uptake of the medium.

The dependence of weight loss on time, obtained for coated steel panels immersed in sea water for 32 days, is illustrated in figures 3 and 4. It can be concluded that the weight losses in groups III and IV are always greater than those in groups I and II, even with blanks. Weight loss of the blanks of groups I and II (neutral varnish without inhibitors) was 40 mg/g steel after 32 days immersion in sea water, while it was 105 mg/g steel for groups III and IV (acid medium without inhibitors) under the same conditions. Also, it can be concluded from Figure 3a that corrosion protection decreases in the order EP–L–MA > EP–L–EA > EP–L–PA > EP–L–BA > blank. This means that the efficiency of the aliphatic inhibitors decreases with increasing chain length in both acidic and neutral varnishes. This phenomenon is shown very clearly in Figure 5, which illustrates the relationship between Taft[30] constants and weight loss of steel in both media. The relationship is not linear and the decrease in weight loss of steel is very sudden, especially for the acid medium, with decreasing chain length of the alkyl group of the amines used in the inhibitors.

In the case of the aromatic inhibitors, corrosion protection decreases in the order EP–L–o–AZ > EP–L–p–Az > EP–L–m–Az > EP–L–p–Tn > EP–L–An > EP–L–m–Tn = EP–L–o–CA = EP–L–CA > EP–L–m–CA = blank. This sequence would be expected except for the position of EP–L–m–Az. However, it can be concluded that the efficiency of the inhibitors increases with increase in electron density on the nitrogen atom of the amino group. This can be seen from Figure 6, where Hammett constants[31,32] for the p-substituted anilines are plotted against weight loss of steel. It is clear that the relationship is linear for both acid and neutral media for all periods of immersion. Figure 7 reinforces this, but the relationship between the basicity of the aromatic amines and weight loss of steel is not linear in an acid medium.

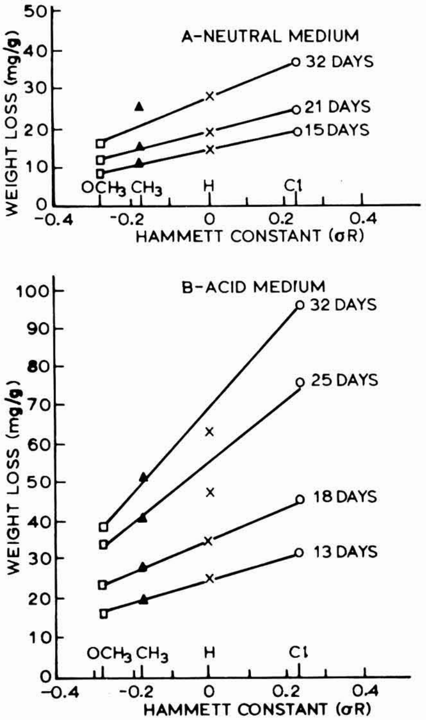

Figure 6. Weight loss of steel panels coated with neutral (A) and acidic (B) varnish films as a function of constant of aromatic amines

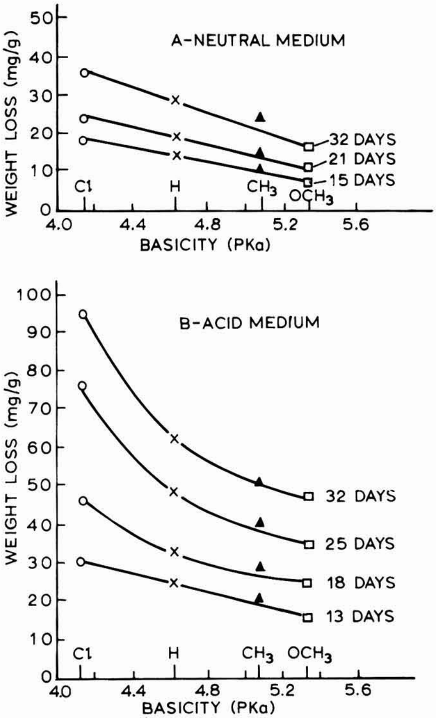

Figure 7. Weight loss of steel panels coated with neutral (A) and acidic (B) varnish films as a function of the basicity of aromatic amines

Conclusions

It is concluded that the reaction products of epoxidized linseed oil with aliphatic and aromatic amines can be used as corrosion inhibitors for steel surfaces. Aliphatic amine derivatives are effective in this respect, and as the chain length of the alkyl group of the amine increases, its efficiency as a corrosion inhibitor decreases.

In the case of aromatic amine derivatives, the electron density on the nitrogen atom plays an important role in their efficiency as corrosion inhibitors, i.e. the efficiency of the aromatic amine derivatives correlates with the electron density on the nitrogen atom of the amino group.

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References

1. Baruch, A. P., *Official Digest*, 1964, **36**(478), 1348.
2. Rosenfeld, I. L., "Ferrara Cong.", 1965, 69; "Moscow Cong.", 1966, 234.
3. Hackerman, N. and Makrides, A. C., *Ind. Eng. Chem.*, 1954, **46**, 523.
4. Nathan, C. C., *Corrosion*, 1956, **12**, 161 t.
5. Cox, P. F. and Riggs, O. L., *Corrosion*, 1964, **20**, 299 t.
6. Badran, B. M., El-Mehelmy, F. M. and Ghanem, N. A., *JOCCA*, 1976, **59**, 291.
7. Abdel-Bary, E. M., Badran, B. M. and Ghanem, N. A., *Europ. Polym. J.*, 1975, **11**, 399.
8. Lee, H. and Neville, K., "Epoxy Resins", *McGraw-Hill*, New York, 1957, 41.
9. Badran, B. M., Yehia, A. A. and Abdel-Bary, E. M., *Europ. Polym. J.*, 1978, **13**, 199.
10. Badran, B. M., Abdel-Fattah, A. A. and Abdul Azim, A. A., *Corrosion Sci.*, 1982, **22**, 513.
11. Badran, B. M., Abel-Fattah, A. A. and Abdul Azim, A. A., *Corrosion Sci.*, 1982, **22**, 525.
12. Vogel, A. I., "Quantitative Inorganic Analysis", 3rd edn., *Longmans*, London, 1966, 363.
13. Warmwell, F. J., *Iron Steel Inst.*, 1948, **160**, 250.
14. Durbetaki, A., *Analy. Chem.*, 1956, **28**, 2000.
15. "Tintative Methods", 2nd edn., *Am. Oil Chem. Soc.*, Chicago, I, 11, 1957.
16. Badran, B. M., *JOCCA*, 1978, **61**, 52.
17. ASTM, D 1200 – A052.
18. Gardner, "Physical and Chemical Examination of Paints", 12th edn., *Gardner Lab.*, 1962, 422.
19. Heaton, N., "Outlines of Paint Technology", 3rd edn., *Charles Griffin*, 1956, 356.
20. Ref. 18, p.147.
21. "Paint Technology Manuals", "The Testing of Paints", *OCCA*, 1965, 133.
22. Erichsen, 'Catalog for Paint Instruments", *Sudwig-Hamer*, W. Germany.
23. Indian Standard Specification (IS): 159, 1950.
24. Indian Standard Specification (IS): 158, 1950.
25. El-Awady, N. I., MSc, thesis, Cairo Univ., 1970.
26. "Modular Training Manual", "Selection and Industrial Training", *Adm. Ltd*, London, vol. III a, 1971, 267.
27. Funke, W., *JOCCA*, 1967, **50**, 942.
28. Jentles, J. K., *JOCCA*, 1963, **46**, 850.
29. Abou-Khalil, M. A., PhD, thesis, Cairo Univ., 1972.
30. Taft, R. W., "Steric Effects in Organic Chemistry", *John Wiley*, New York, 1956, ch. 13.
31. Hammett, L. P., *Chem. Rev.*, 1935, **17**, 125.
32. Jaffe, H. H., *Chem. Rev.*, 1953, **53**, 191.

Aspects of galvanic action of zinc-rich paints. Electrochemical investigation of eight commercial primers

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Summary

The cathodic protection of steel provided by zinc dust in eight commercial zinc-rich paints based on ethyl silicate and epoxy binders has been investigated by AC resistance-time, by potential-time, and by potentiostatic anodic polarization techniques. The AC resistance and potential were recorded for painted electrodes over 60 days of exposure to aerated 0.1 M sodium chloride solution at 25°C and pH 7, and anodic polarization curves were determined after periods of 3 and 60 days pre-exposure.

The AC resistance and potential of the epoxy-based zinc-rich paints increased much faster during exposure than did those of the ethyl silicate paints. A relationship exists between high AC resistance/potential and low galvanic action, as shown by anodic polarization measurements. After 60 days of exposure, limited galvanic action was still present in only two ethyl silicate paints. No simple correlation was found between cathodic protection and the zinc content of the commercial ethyl silicate or epoxy paints from either AC reistance-time, potential-time or anodic polarization measurements. The zinc content found in the paints was 83-93 wt per cent, which is rather low. At these concentrations it obviously was not only the zinc content itself that determined the electrochemical/electrical properties of the paint not even within a single class of binder, as ethyl silicate or epoxy. The duration of the corrosion protection given by both ethyl silicate and epoxy-based zinc-rich paints was, under immersed conditions, longer than their galvanically active time. Cathodic protective activity is probably accompanied by inhibitive properties of basic zinc salts formed on the steel surface and by high pH (≈9) within the paint film.

1. Introduction

The mechanism for corrosion protection given by zinc-rich paints is not fully understood. A review of different opinions is given by Evans[1-3]. Proposed mechanisms found in the literature can be summarized as follows:

1. Galvanic action from the couple – zinc particles in the paint film and the steel substrate.
2. Cathodic inhibition on steel from zinc salts formed due to corrosion of the zinc itself or due to galvanic action.
3. High pH within the paint film, determined by the zinc salts formed.

In an earlier investigation[4] we studied the duration of the galvanic action of zinc-rich paints by polarization measurements using a potentiostatic technique. The present paper reports on the cathodic protective activity of eight commercial zinc-rich paints based on ethyl silicate and epoxy binders.

2. Experimental

The AC resistance and electrode potential of painted steel electrodes and uncoated solid-zinc electrodes were determined as a function of exposure time in aerated 0.1 M sodium chloride solution at 25°C and pH 7 for 60 days. By a potentiostatic technique, anodic polarization curves were determined after 3 and 60 days of pre-exposure. The zinc content (wt per cent) and type of binder for the eight commercial zinc-rich paints investigated are given in Table 1.

2.1 Specimen electrodes

The electrode was a 14 mm diameter, 3 mm thick, metal disc with a soldered polyethene-covered electrical connector, all moulded in epoxy resin[4,5]. The disc material used as a substrate for the zinc-rich paints was cold rolled steel (SIS 1142); for reference, unpainted solid zinc metal discs, *pro analysi* grade, were used. A stainless steel (SIS 2343) disc was used for the counter electrode in the AC resistance measurements. The surfaces of the steel discs that were to be painted were blasted to Sa 3, after they had been painted the other disc surfaces were moulded in epoxy resin, thus completing the painted electrode. In order to cure the ethyl silicate, the painted electrodes were kept at room temperature and high relative humidity (≈90 per cent) for ten days, and after that at normal room temperature and relative humidity until used in the experiments.

2.2 AC resistance measurements

The AC resistance (frequency 1 kHz) of the painted steel

Table 1

Paint code and composition and a qualitative comparison of AC resistance and electrochemical properties of zinc-rich paints – AC resistance, electrode potential, and anodic polarization

Type of binder	Paint No.	Zinc content (wt %)	AC resistance	Electrode potential	Anodic polarization	
					3 days	60 days
Epoxy	1	90	0	–	0	0
	3	90	–	–	+	0
	6	92	–	0	+	–
	8	89	–	0	+	–
Ethyl silicate	2	83	+	+	+	+
	4	91	0	–	+	0
	5	87	+	0	+	0
	7	93	+	+	+	+

Property (+ positive, 0 medium, – negative) associated with corrosion protection given by cathodic protection from the zinc electrodes and the uncoated solid zinc electrodes were recorded during the 60 day exposure. The simple experimental apparatus was a glass cell with a Plexiglas lid and a holder with space for a specimen, counter and reference electrodes. The distance between the two surfaces of the specimen electrode and the stainless steel counter electrode was 10 mm. A Philoscope PR 9500 was used for the AC resistance measurements. The reported resistance is that of the cell; no compensation has been made for the resistance of the electrolyte. The results presented should therefore be used only for comparison between different samples.

2.3 Electrode potential measurements

The electrode potentials of the painted steel electrodes and the uncoated solid zinc electrode were recorded during the 60 day exposure using the same cell as for AC resistance measurements. The reference electrode was a saturated calomel electrode (SCE), and the potential was measured with a digital electrometer having high input impedance ($>2 \cdot 10^{14}$ ohm). The computerized measuring system has been described elsewhere[4].

2.4 Anodic polarization measurements

Anodic polarization curves were determined using a potentiostatic technique for electrodes of solid zinc metal and for steel electrodes coated with the zinc-rich primers. The potential sweep rate was 20 mV/min. The electrochemical measuring equipment was controlled by a minicomputer. The experimental set-up and the electrochemical cell used have been described elsewhere[4]. An SCE was used as a reference electrode. Before the anodic polarization curves were determined, the specimen electrodes were pre-exposed by immersion in aerated 0.1 M sodium chloride solution for periods of 3 and 60 days.

3. Results and discussion

The results from an investigation of cathodic protective activity of zinc-rich paints on steel were reported in an earlier paper[4]. From AC resistance and potentiostatic anodic polarization measurements it was found that an ethyl silicate zinc-rich paint had longer cathodic protective activity than an epoxy-based one, and for paints based on a single ethyl silicate binder with various zinc contents, the galvanic activity decreased with decreased zinc content.

In the present investigation the cathodic protective activity of eight commercial zinc-rich paints (four ethyl silicate and four epoxy-based) was studied.

The major objectives of the present investigation were:

1. To find out if an ethyl silicate-based zinc-rich paint generally has longer cathodic protective activity than an epoxy-based one.
2. To find out if there is a simple correlation between the zinc content in commercial zinc-rich paints and galvanic activity and its duration.
3. To compare the relationship of AC resistance and electrochemical (electrode potential and anodic polarization) properties of zinc-rich paints on steel substrates.
4. To evaluate the AC resistance and electrochemical measuring methods for use in practical paint formulation and testing.

When exposed to 0.1 M sodium chloride solution, the solid zinc metal electrodes – used for reference – were completely covered with a white corrosion product within a week. Also, the electrodes with zinc-rich paints based on ethyl silicate became more or less covered with this corrosion product due to the corrosion of zinc particles in the paint surface, but to a much lesser extent than the solid metal. Furthermore, the epoxy-based painted electrodes showed much lower corrosion of the zinc particles in the paint surface. The activity of the zinc surfaces was: solid zinc metal > zinc in ethyl silicate >> zinc in epoxy.

X-ray diffraction analysis of the zinc corrosion products found on zinc-rich paints and on hot dipped, galvanized areas[10] showed the corrosion products to be mixtures of several zinc compounds. $Zn_4CO_3(OH)_6H_2O$ and $Zn_5(CO_3)_2(OH)_6$ were identified, but although present, no chloride-containing compounds were identified. The non-polar ethyl silicate binder wets the zinc particles less effectively than the polar epoxy, and consequently the ethyl silicate-based coating is more porous and the active zinc surface is larger; this was discussed in an earlier paper[6]. This explains the greater tendency of the zinc particles to corrode at the surface of the ethyl silicate-based primer compared with the epoxy-based primer. Because it is difficult to investigate, little is known about the corrosion of zinc particles within the paint film and at the interface between a steel substrate and a paint film. The existence of corrosion products from zinc on a steel surface under the paint has been shown[7-9]. This problem will be treated in a separate paper[10].

The results of the AC resistance measurements on ethyl silicate-based zinc-rich paints are shown in Figure 1, and the corresponding results for epoxy-based paints are shown in Figure 2. Resistance values in the figures are the total values for the cell – specimen electrode, electrolyte, counter electrode – and should be used only for relative comparisons between the different samples. The resistance of the solid-zinc electrode cell is shown for reference, and as the increase in resistance with time was very low, there seems to be no reason to believe that the heavy increase in resistance of the painted electrodes was caused by a build up of zinc corrosion products on the paint surface. This opinion is confirmed by the fact that the ethyl silicate paints, having a much greater rate of paint surface zinc corrosion, show a much lower increase in resistance with time than do the epoxy-based ones. The reason for this difference is further discussed elsewhere[5,10]. No simple correlation was found between the increase in resistance with exposure time to electrolyte and zinc content.

From the electrode potential measurements shown in Figure 3 (ethyl silicate) and Figure 4 (epoxy) it can be seen that the solid zinc metal electrodes showed small fluctuations during the first week of exposure as the surface was covered with corrosion products; after that the potential became comparatively stable. The same figures show that the potential of the zinc-rich paints drifted towards a more positive value, more pronounced for epoxy than for ethyl silicate-based paints. Some came close to the potential of corroding steel, indicating a complete loss of cathodic activity of the zinc particles in the paint. No simple correlation was found between an increase in potential with time during exposure to electrolyte and zinc content in the commercial paints investigated. This seems to be valid for both ethyl silicate and epoxy-based paints. At the low zinc content used in the commercial paints, the zinc content is not in itself the only parameter determining the potential and the potential variation with exposure time when different vehicle formulations and incorporation of extenders are used. However, the ethyl silicate paint with the highest zinc content (93 wt per cent) was in best accordance with the solid zinc metal.

Anodic polarization curves for ethyl silicate and epoxy paints determined after three days pre-exposure in 0.1 M sodium chloride solution are shown in figures 5 and 6, respectively. The corresponding curves for 60 days exposure are shown in figures 7 and 8. After three days, the four ethyl silicate and three of the epoxy-based paints showed a definite cathodic protective property. For the fourth epoxy paint it was more dubious. After 60 days exposure there still seemed to exist some small cathodic protective activity for two ethyl

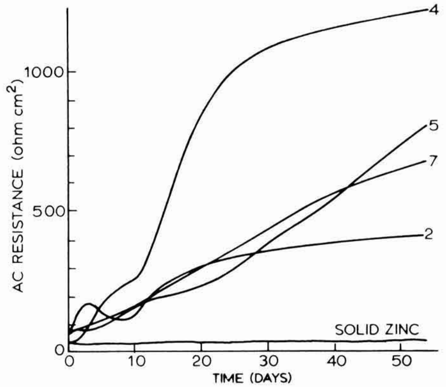

Figure 1. AC resistance of cells (specimen electrode, electrolyte, counter electrode) as a function of time during exposure in aerated 0.1 M sodium chloride solution at 25 °C and pH 7. Solid zinc metal and four commercial ethyl silicate zinc-rich paints on steel were the specimen electrodes

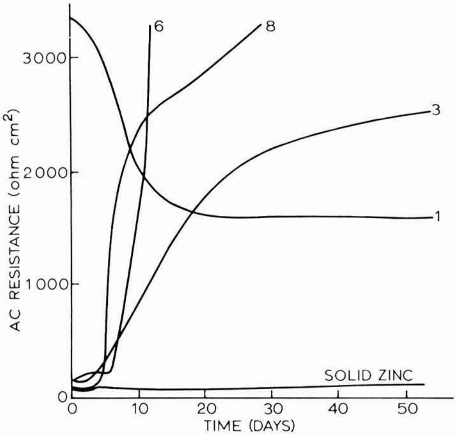

Figure 2. AC resistance of cells (specimen electrode, electrolyte, counter electrode) as a function of time during exposure in aerated 0.1 M sodium chloride solution at 25 °C and pH 7. Solid zinc metal and four commercial epoxy zinc-rich paints on steel were the specimen electrodes

silicate-based paints, numbers 2 and 7. It is doubtful if there remained any cathodic protective activity at all from the zinc particles in the rest of the paints, whether ethyl silicate or epoxy-based. No simple correlation was found between cathodic protection and zinc content. This seems to be valid for both ethyl silicate and epoxy. The ethyl silicate-based paints after a short exposure time (Figure 5) showed a maximum in the anodic polarization curve. A corresponding maximum was not found either for solid zinc metal or epoxy-based paints in the potential interval investigated. This is in agreement with earlier reported findings[4].

When the specimens were exposed to a salt solution, a great increase in paint film resistance/potential (corresponding to a lesser cathodic protective paint film) was shown by the anodic polarization measurements. No simple correlation was found between AC resistance, potential or anodic polarization and zinc content for either the eight commercial zinc-rich paints studied in this investigation or for the two paints in an earlier study[4], although the two ethyl silicate paints with the highest zinc content (93 wt per cent) showed the best cathodic protective activity. The zinc contents in the investigated paints are low compared with the values recommended in the literature[11] for the long lasting cathodic protection of steel. The only possible explanation for the odd behaviour of paint number 1, as shown in figures 2, 4 and 6, is an entirely different paint formulation or different paint raw materials were used. The zinc contents are, however, representative of those used in practice, since those ten paints are manufactured in more than 40 countries all over the world. It has been found[4] that ethyl silicate as a binder in zinc-rich paints gives much longer cathodic protection than epoxy; this result is confirmed by the present investigation.

Practical experience and laboratory bath tests[10] showed that the corrosion protection time for both ethyl silicate and epoxy-based zinc-rich paints, under immersed conditions, was longer than could be expected from only the cathodic protection by zinc of the steel substrate. Schuster[12] and Ross and Wolstenholme[13] pointed out that in addition to the cathodic protection there might be an inhibitive effect of the corrosion products of zinc. This can, according to Hatch[14], be compared to the situation in recirculating cooling water systems, where zinc salts markedly increase the cathodic polarization and thus serve as corrosion inhibitors. Their action, generally, is attributed to precipitation of basic zinc salts on cathodic areas as a result of the locally elevated pH. Protection is established rapidly by zinc salts but is not very durable.

The ten paints have been further investigated using potentiostatically controlled impedance measuring equipment[6]. The results confirm the difference in cathodic protective property between ethyl silicate and epoxy as binders in zinc-rich paints, as reported in the present paper.

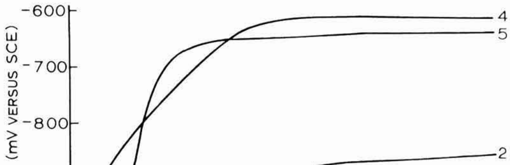

Figure 3. Electrode potential as a function of time during exposure in aerated 0.1 M sodium chloride solution at 25 °C and pH 7 for four commercial ethyl silicate zinc-rich paints on steel and for solid zinc metal

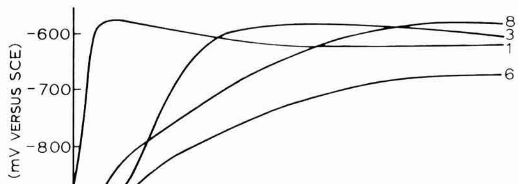

Figure 4. Electrode potential as a function of time during exposure in aerated 0.1 M sodium chloride solution at 25 °C and pH 7 for four commercial epoxy zinc-rich paints on steel and for solid zinc metal

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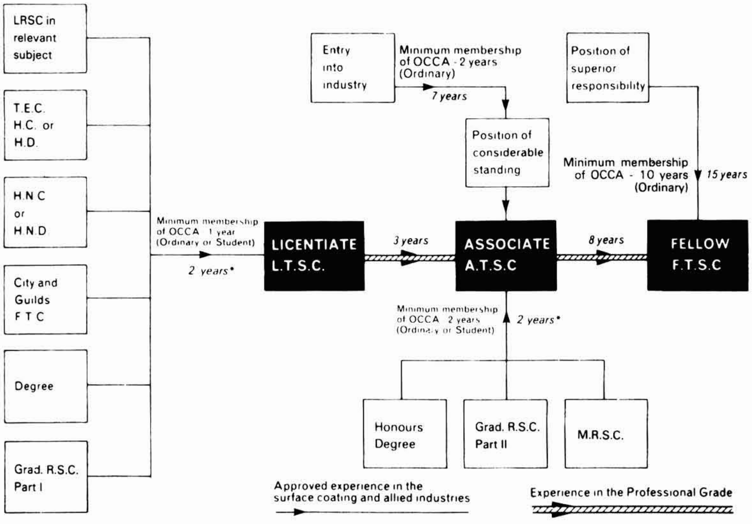

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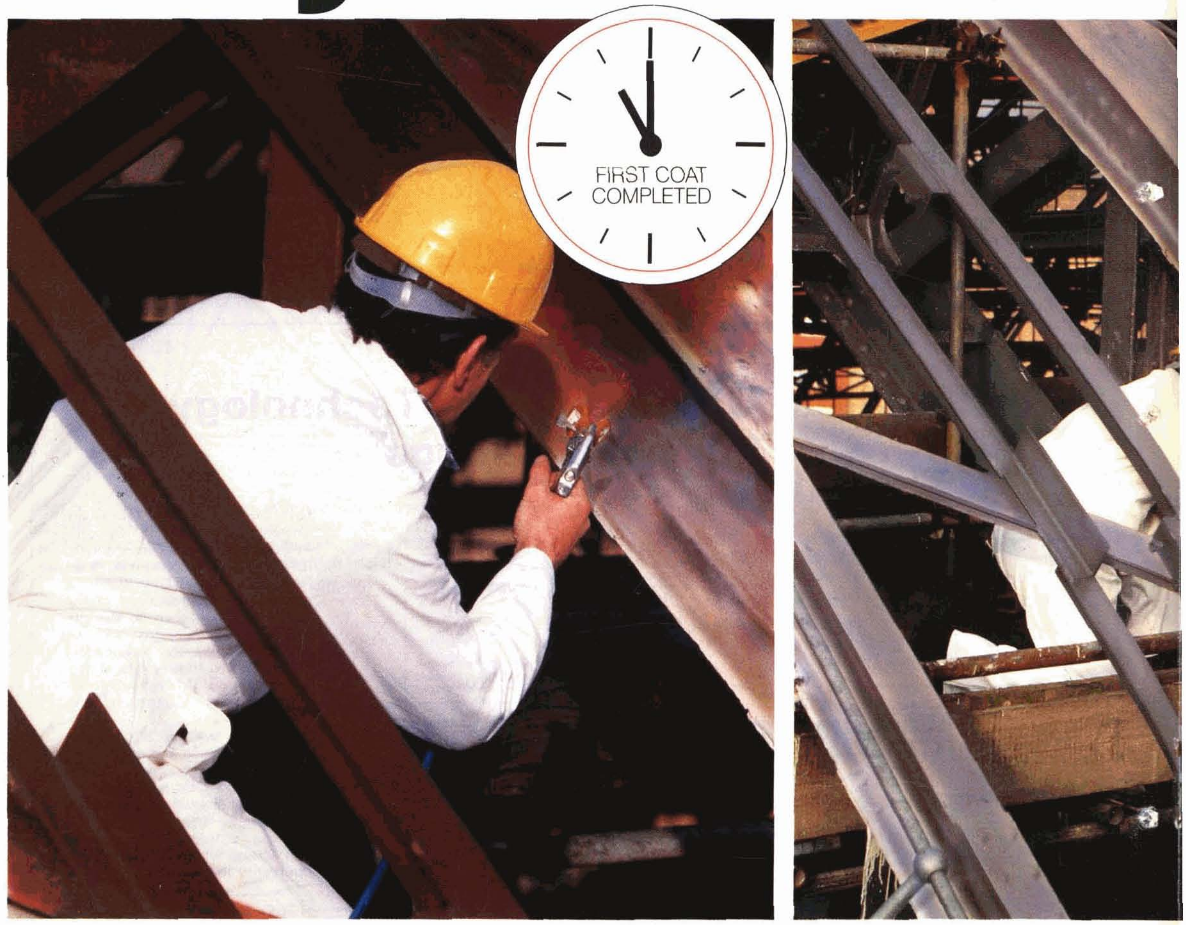

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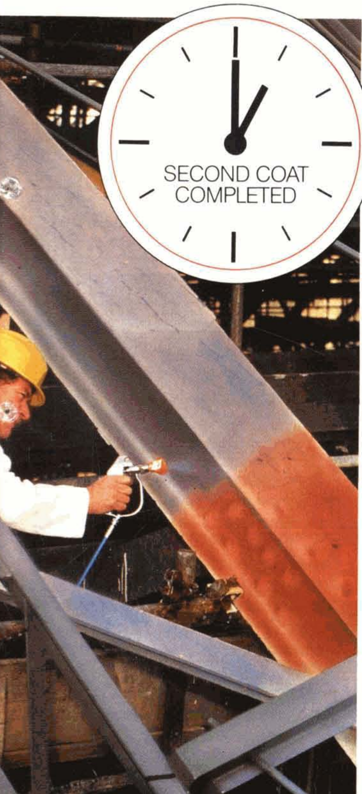

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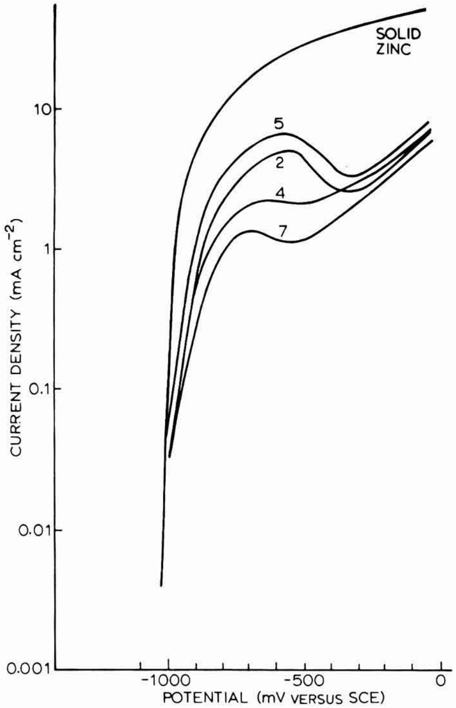

Figure 5. Anodic polarization curves for four commercial ethyl silicate zinc-rich paints on steel and for solid zinc metal. The electrodes were pre-exposed to aerated 0.1 M sodium chloride solution at 25 °C and pH 7 for three days before polarization

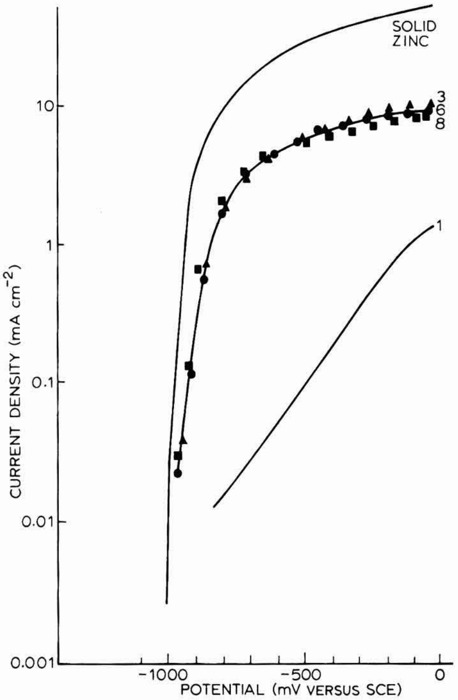

Figure 6. Anodic polarization curves for four commercial epoxy zinc-rich paints on steel and for solid zinc metal. The electrodes were pre-exposed to aerated 0.1 M sodium chloride solution at 25 °C and pH 7 for three days before polarization

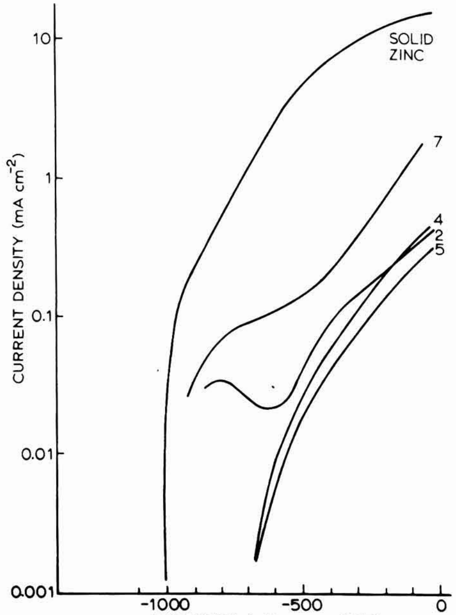

Figure 7. Anodic polarization curves for four commercial ethyl silicate zinc-rich paints on steel and for solid zinc metal. The electrodes were pre-exposed to aerated 0.1 M sodium chloride solution at 25 °C and pH 7 for 60 days before polarization

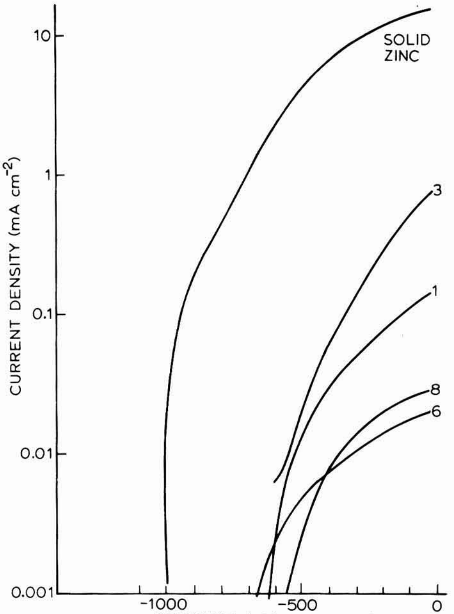

Figure 8. Anodic polarization curves for four commercial epoxy zinc-rich paints on steel and for solid zinc metal. The electrodes were pre-exposed to aerated 0.1 M sodium chloride solution at 25 °C and pH 7 for 60 days before polarization

AC resistance, potential, anodic polarization and potentiostatically controlled impedance measurement techniques are useful tools for the investigation of the total mechanism of, and the duration of the corrosion protection given by zinc-rich paints, i.e. not only the cathodic protection. The polarization techniques give more information than single AC resistance or potential measurements.

4. Conclusions

Conclusions drawn from the reported AC resistance/electrochemical investigations of eight commercial zinc-rich paints can be summarized as follows:

1. Commercial ethyl silicate-based paints give much longer cathodic protective action under immersed conditions (still present to some extent after 60 days, for some paints) than epoxy-based ones do. This was shown by AC resistance-time, potential-time, and anodic polarization measurements.
2. At the comparatively low zinc content (83-93 wt per cent) used in commercial paints, AC resistance, potential, or anodic polarization properties are not determined solely by the zinc content. No simple correlation was found for cathodic protection and zinc content, although the ethyl silicate paint with the highest zinc content (93 per cent) showed the best cathodic protection after 60 days exposure.
3. Corrosion protection given by zinc-rich paints lasts longer than their cathodic protective activity. The activity mentioned is probably combined with an inhibitive effect on the steel from the zinc corrosion products formed and from high pH within the paint film.
4. The surface corrosion rate of zinc was: uncoated solid zinc $>$ zinc in ethyl silicate $\gg$ zinc in epoxy.

   But the AC resistance increase was: uncoated solid zinc $\ll$ zinc in ethyl silicate $<$ zinc in epoxy.

   The rapid decrease in cathodic protective activity of epoxy-based paints is obviously not as a result of coating of the zinc particles in the paint surface by corrosion products. This opinion has been confirmed by potential-time and polarization measurements.
5. When the specimens are exposed to a salt solution, a great increase in paint film resistance/potential corresponds to lesser cathodic protection by the paint film, as is shown by reduced zinc dissolution from anodic polarization measurements.
6. Useful information on the total protective mechanism and the duration of corrosion protection given by zinc-rich paints can be obtained by combined electrochemical and electrode impedance (resistance) methods.

5. Acknowledgements

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6. References

1. Evans, U. R., "The Corrosion and Oxidation of Metals", *Edward Arnold*, London, 1960, 615.
2. Evans, U. R., "The Corrosion and Oxidation of Metals, First Supplementary Volume", *Edward Arnold*, London, 1968, 229.
3. Evans, U. R., "The Corrosion and Oxidation of Metals, Second Supplementary Volume", *Edward Arnold*, London, 1976, 304.
4. Lindqvist, S. Å., *Werkst. Korros.*, 1980, **31**, 524.
5. Lindqvist, S. Å. and Vannerberg, N.G., *Werkst. Korros.*, 1974, **25**, 740.
6. Mészáros, L. and Lindqvist, S. Å., "Eurocorr'82", Budapest, 18-22 October 1982, Section II, 147.
7. Mayne, J. E. O., *J. Iron Steel Inst.*, 1954, **176**, 143.
8. Newton, D. S. and Sampson, F. G., *JOCCA*, 1965, **48**, 382.
9. Moree, J. C., *Verfkroniek*, 1965, **38**, 364.
10. Lindqvist, S. Å., Mészáros, L. and Svensson, L., "9th Scandinavian Corrosion Congress", Copenhagen 12-14 September 1983, vol. 1, 199.
11. Mayne, J. E. O., *Trans. Inst. Met. Finishing*, 1964, **41**(4), 121.
12. Schuster, H. J., *Werkst. Korros.*, 1959, **10**, 490.
13. Ross, T. K. and Wolstenholme, J., *Corros. Sci.*, 1977, **17**, 341.
14. Hatch, G.B., in "Corrosion Inhibitors", ed. Nathan, C. C., *NACE*, Houston, 1973, 126.

Physico-chemical characterisation of aqueous shellac solutions and hydrosols for encapsulation

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Summary

The physico-chemical properties of aqueous solutions of regular (Bhatta) and dewaxed shellacs and their hydrosols have been studied. In both forms shellac has been found to possess a negative charge and show the reversibility phenomenon. The studies suggest that both types of shellac are suitable as a wall material and may be used in the form of hydrosol for simple and complex (pH) coacervation processes of encapsulation.

Introduction

Studies on the solution and sol properties of lac[1] have been in progress mainly at ILRI, Namkum since 1928. Since then the programme has contributed valuable results.

Rangaswami and Venugopalan[2] first reported in an account some of the physical propeties of shellac, in which the viscosity

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and solubility variations of shellac solution in common solvents have been given. They showed that both viscosity and solvent power are simultaneously important in rating a solvent. Verman[3] reported a study on the viscosity of lac and hard resin solutions and found that the viscosity of the latter is a little higher than the former. It has been noticed that viscosity decreases slightly with ageing of shellac solutions.

Bhattacharya[4] measured the viscosity of alcoholic solutions of shellac along with urea. Reasons of the variation in viscosity have been tentatively explained. Palit[5,6,14] did a considerable amount of work on the physical chemistry of resin solutions in which the characteristics of solution properties of shellac were the main theme and the nature of the shellac molecule in terms of colloidal aspect was dealt with in detail. Basu[7-11] investigated various properties of shellac solutions concentrating mainly on the molecular aspects. Basu[7,12] further studied properties by an optical method to elucidate the nature of shellac solution more clearly. The UV spectra of shellac in various alcohols and acids have also been studied by Basu[13], and certain behaviours of the shellac molecule in the different solvents are interpreted. He also studied the anisotropy of shellac molecules and indicated that shellac molecules are inherently anisotropic.

Basu[9] then investigated the nature of concentrated shellac solutions. Previously, Verman and Palit assumed the colloid-chemical property of shellac to be associated with the presence of micelles. However, Basu considered the existence of clusters of molecules.

The original postulation[1,4], that shellac solutions in organic solvents are of a colloidal nature, has been proved, on systematic study[5], to be incorrect. In fact, in the dilute non-aqueous solutions (in organic solvents), shellac is molecularly dispersed.

As such, lac is insoluble[1] in water. However, due to its acidic character it dissolves in aqueous solutions of caustic alkalies, ammonia, borax, organic bases etc. Lac behaves like a colloid, in dilute aqueous solutions, and its hydrosols can be prepared[1].

Palit[14] reported a new method of preparation of the hydrosols of shellac by dialysing its aqueous ammoniacal solution, and studied their properties. A hydrosol of shellac can also be prepared by continuously pouring an alcoholic solution of shellac into water and evaporating the alcohol in vacuum.

Gidvani[16,17] mentioned that when shellac dispersions in ammonia are heated in the open, the ammonia volatilizes leaving behind a colloidal solution of shellac. Colloidal solvation of shellac has also been examined, by Shpenzer *et al*[18,19].

Thus we see that the solution and sol properties of shellac play a very important role in the theoretical understanding of shellac molecules in a dissolved state. This understanding is of use in finding practical applications for commercial purposes such as in the processes of encapsulation[20], which is a rapidly expanding technology encompassing several areas including paint technology[21]. This study has been undertaken with this practical objective in view.

Material and methods

Shellac solution

Two types of shellac, regular (Bhatta) and dewaxed (DL, dewaxed lemon), were used and solutions at three concentrations were prepared by dissolving the shellac resin in aqueous ammoniacal solution by slight warming on a water bath.

Shellac hydrosol

Shellac hydrosols were prepared by the following two methods.

Dialysis method

Hydrosols were prepared by the slightly modified method of dialysis as proposed by Palit[14], using thin cellophane paper for the sac (bag). Aqueous ammoniacal solutions in a cellophane bag were dialysed against an equal amount of water at 30°C which was changed twice a day. The concentration of the resin in sols was adjusted.

Condensation method[16,17]

Hydrosols of shellac were also prepared by a condensation technique. Shellac solutions were made up in methylated spirits (95 per cent v/v) and poured into an excess of boiling water (five fold), the solvent being removed by gently boiling the water.

Physico-chemical characteristics

Various physico-chemical characteristics of these shellac solutions and hydrosols were determined, as outlined below.

Concentration of resin

The concentration of the resin was determined by evaporating to dryness a known volume (10 ml) of the sols/solutions and then heating for three hours at 100 ±1°C until a constant weight was obtained.

pH value

The pH value of solutions and sols was determined with the help of a pH meter (Phillips model PR – 1904 M) by using glass and reference electrodes.

Specific conductivity

The specific conductivity of sols/solutions was measured at 30°C by a conductivity measuring bridge (Phillips model – 9500/90).

Relative density[22]

The relative density of sols/soltuions was measured at 30°C by an RD bottle fitted with a thermometer.

Relative viscosity[22]

The relative viscosity of solution/sols was determined at 30°C by a commonly used Ostwald type viscometer.

Nature of charge[23,24,34]

The nature of the charge on the shellac resin in solutions/sols was ascertained by the boundary movement method using a Burton tube and applying a known DC current (100 V) from a power supply unit (Apalab model – MVH 5/10) using a silver electrode. The same was also determined and confirmed using acrylamide disc-gel electrophoresis apparatus (model AIMIL 75024) and the above power supply unit.

Reversibility[25]

The reversibility phenomenon of the sols/solutions was determined by using N/2 HCl and N/2 NaOH solutions.

Ammoniacal strength

Ammoniacal strength was estimated by the method adopted by Palit[14].

Results and discussions

Results and observations made regarding various physicochemical characteristics of aqueous solutions and

Table 1

Physico-chemical characteristics of aqueous solutions and hydrosols of regular (Bhatta) shellac

Sl. No.	Characteristics	Aqueous solutions of regular (Bhatta) shellac			Hydrosols of regular (Bhatta) shellac (dialysis method)			Hydrosols of regular (Bhatta) shellac (condensation method)		
		I	II	III	I	II	III	I	II	III
1.	Appearance	hazy, wax floats on top on standing	hazy, wax floats on top on standing	hazy, wax floats on top on standing	trans-parent, very slightly hazy in reflected light	trans-parent, very slightly hazy in reflected light	trans-parent, very slightly hazy in reflected light	milky and opaque	milky and opaque	milky and opaque
2.	Concentration of resin (% w/v)	4.82	9.24	13.10	3.25	4.02	5.49	0.25	0.31	0.5
3.	Concentration of ammonia (N)	0.52	0.49	0.49	0.025	0.018	0.019			
4.	Sp. conductivity (mhos/cm at 30°C)	7.89×10^{-3}	1.37×10^{-2}	1.45×10^{-2}	9.02×10^{-4}	12.63×10^{-4}	12.63×10^{-4}	1.62×10^{-4}	1.66×10^{-4}	1.34×10^{-4}
5.	pH	9.8	9.2	9.3	7.3	7.2	7.2	4.2	4.5	4.5
6.	Relative density (30°C)	1.007	1.015	1.021	1.015	1.017	1.019	1.001	1.001	1.002
7.	Relative viscosity (30°C)	1.09	1.37	1.67	1.20	1.25	1.30	1.01	1.01	1.02
8.	Nature of charge	negative	negative	negative	negative	negative	negative	negative	negative	negative
9.	Reversibility	reversible	reversible	reversible	reversible	reversible	reversible	reversible	reversible	reversible

Table 2

Physico-chemical characteristics of aqueous solutions and hydrosols of dewaxed (DL) shellac

Sl. No.	Characteristics	Aqueous solutions of dewaxed (DL) shellac			Hydrosols of dewaxed (DL) shellac (dialysis method)			Hydrosols of dewaxed (DL shellac (condensation method)		
		I	II	III	I	II	III	I	II	III
1.	Appearance	clear	clear	clear	trans-parent	trans-parent	trans-parent	milky and opaque	milky and opaque	milky and opaque
2.	Concentration of resin (% w/v)	4.81	9.55	15.34	3.20	4.10	5.60	0.24	0.32	0.49
3.	Concentration of ammonia (N)	0.52	0.49	0.49	0.028	0.019	0.019			
4.	Sp. conductivity (mhos/cm at 30°C)	7.89×10^{-3}	1.37×10^{-2}	1.52×10^{-2}	9.02×10^{-4}	12.63×10^{-4}	12.63×10^{-4}	1.62×10^{-4}	1.66×10^{-4}	1.34×10^{-4}
5.	pH	9.9	9.2	9.2	7.4	7.3	7.3	4.1	4.5	4.4
6.	Relative density (30°C)	1.007	1.016	1.026	1.014	1.017	1.020	1.001	1.001	1.002
7.	Relative viscosity (30°C)	1.07	1.38	1.70	1.20	1.26	1.31	1.01	1.01	1.02
8.	Nature of charge	negative	negative	negative	negative	negative	negative	negative	negative	negative
9.	Reversibility	reversible	reversible	reversible	reversible	reversible	reversible	reversible	reversible	reversible

hydrosols of regular (Bhatta) shellac and dewaxed (DL) shellac are given in tables 1 and 2, respectively. It has been observed from the appearance of shellac solutions that wax, which is associated with shellac[1], produces haziness in Bhatta shellac solutions, whereas DL shellac solutions are transparent. Similar observations have been made of hydrosols of Bhatta shellac. It has been observed that on dialysis some part of the wax is removed during the process[33], thereby producing a hazy dialysate, the haziness being more pronounced as the concentration increases. However, this does not produce any adverse effect during the process.

Aqueous ammoniacal solutions of both types of shellac have been found to be conductive and this property is related to the pH and not to the concentration of the resin. The viscosity of the solutions has been observed to be related to their concentrations.

Aqueous shellac solutions possess a negative charge, which is in agreement with the observations made by Wiedernman[26], Pandey[27], and Banerjee *et al*[34]. This negative charge is obviously due to the presence of the COOH group in the shellac molecule[1].

The reversibility phenomenon[25] has been observed in aqueous ammoniacal solutions of regular (Bhatta) and dewaxed (DL) shellac, which is characteristic of its colloidal and hydrophilic nature. Shellac hydrosols prepared by dialysis are conductive and this property is related to the pH. Hydrosols thus prepared are almost neutral and this observation is in good agreement with Palit[14]. The charge[28] on both types of shellac resin colloid has been found to be negative in nature. Thus, shellac may be considered as a carboxyl-colloid. This is in agreement with Lewis *et al*[29] and is a pre-requisite of a wall material (hydrophilic colloid) for suitable encapsulation in pH coacervation[20] (aqueous phase separation). The reversibility phenomenon[30] in shellac hydrosols has also been observed, which further signifies the suitability of shellac as a wall material in the pH coacervation process[25].

Further, it has been observed that shellac hydrosols may be concentrated or diluted to the desired extent, provided a minimum concentration of ammonia remains so that it is not transformed into a gel. Palit[14] also made similar observations. Preparation of thicker hydrosols works in favour of the encapsulation purpose.

It has been observed that there is no difference in the physico-chemical characteristics of the sols of dewaxed and regular (Bhatta) shellacs (tables 1 and 2). Therefore, regular shellac being cheaper than dewaxed shellac, regular may be used for preparing shellac hydrosols by using the dialysis technique and may be utilised as carboxyl-colloid for encapsulation by pH coacervation[25] (di-complex process). The concentration of resin in hydrosols can be adjusted and these hydrosols show similar characteristics. Thus, shellac solutions of 5-15 per cent w/v may be used for the preparation of hydrosols. However, solutions of 10 per cent w/v resin concentration may be preferred, as molecular clusters which exists in shellac solutions (Basu[8-11]) may grow bigger and more stable at higher concentrations. The cluster formation, of course, results from the influnce of secondary valence forces, and in dilute solutions the shellac molecules are so far apart that the van der Wall's forces cannot operate. The greater the concentration of shellac, the less is the distance between dissolved molecules and consequently the greater is the association or cluster formation (Basu[8-11]).

On analysing the physico-chemical characteristics of hydrosols prepared by the condensation technique, it has been observed that the pH is considerably lowered, which is not desirable for encapsulation[20,25]. The conductivity, also, is not sufficient. In addition, these sols are milky and opaque and extremely dilute. The concentration of resins was insignificant. Further, these sols cannot also be concentrated. This is in agreement with the observation made by Wolff[15]. Thus, all these characteristics suggest that the shellac hydrosols prepared by the condensation technique are not suitable for encapsulation.

Conclusions

It may thus be concluded that the ordinary shellac hydrosols prepared by the dialysis method possess all the desired physico-chemical characteristics[20,25] and, therefore, may be suitably used as a wall material as such and in conjunction with gelatin on a par with other negatively charged polymeric materials or polyelectrolytes[31] such as polysaccharide, gumarabic, etc. Further, any type of shellac hydrosols may be used as a carboxyl-colloid in a simple or di-complex coacervation system for encapsulation[20,25]. Aqueous ammoniacal solutions of shellac may also be used in fluidised bed encapsulation.

Acknowledgement

The authors wish to express their thanks to Dr T. P. S. Teotia, Director of the Institute, for his encouragement and kind permission to publish the results; to Dr B. B. Khanna, Head, Division of Chemistry, for his keen interest in the work; and to Dr A. Pandey, Scientist of this Institute, for his help in determining the specific conductivity.

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References

1. Bose, P. K., Sankaranarayanan, Y. and Sengupta, S. C., "Chemistry of Lac", *Indian Lac Research Institute*, Namkum, 1963.
2. Rangaswami, M. and Venugopalan, M., *Indian Lac Res. Instt.*, 1928, Bull. Nos. 1 and 2.
3. Verman, L. C., *Lond. Shellac Res. Bur.*, 1937, Tech. Paper No. 11.
4. Bhattacharya, G. N., *Ind. Lac Res. Instt.*, 1940, Bull. No. 42.
5. Palit, S. R., *J. Ind. Chem. Soc.*, 1940, **17**, 308, 537, 663; *Ind. Lac Res. Instt.*, Bull. Nos. 39, 4 and 44.
6. Palit, S. R., *J. Ind. Chem. Soc.*, 1942, **19**, 207, 253, 266, 414 and 434; *Ind. Lac Res. Instt.*, Bull. Nos. 46, 48, 51, 52 and 53.
7. Basu, S., *J. Ind. Chem. Soc.*, 1947, **24**, 148, 157 and 263; *Ind. Lac Res. Instt.*, Bull. Nos. 67, 68 and 70.
8. Basu, S., *Sci. Cult.*, 1948, **14**, 279.
9. Basu, S., *Sci. Cult.*, 1948, **14**, 119.
10. Basu, S., *Sci. Cult.*, 1948, **13**, 508.
11. Basu, S., *J. Ind. Chem. Soc.*, 1948, **25**, 103; *Ind. Lac Res. Instt.*, Bull. No. 75.
12. Basu, S., *J. Phys. Coll. Chem.*, 1950, **54**, 1358; *Sci. Cult.*, 1948, **14**, 79.
13. Basu, S., *Ind. Lac Res. Instt.*, Ann. Rep., 1947-48, 22.
14. Palit, S. R., *J. Ind. Chem. Soc.*, 1940, **17**, 6, 375; *Ind. Lac Res. Instt.*, Bull. No. 40.
15. Wolff, H., *Chem. Umschau*, 1916, 92.
16. Angelo Bros, "Shellac", *Angelo Bros*, Cossipore, Calcutta, 1965.
17. Gidwani, B. S., "Natural Resin Plastic Monograph", 1946, 46, 39.
18. Shpenzer, N. P., Gurvich, T. B. and Talmud, S. L., *Chem. Abst.*, 1969, **71**, 62, 430.
19. Shpenzer, N. P., Ketslei, N. Y. and Talmud, S. L., *Chem. Abst.*, 1969, **71**, 72, 139.
20. Vandagaer, J. E., "Microencapsulation Processes and Applications", *Plenum Press*, New York, 1974.
21. Makorskoya, T. A. and Stolyarova, V. A., *Lako Krasosechnye, Ikh Primenenie Materiale*, 1980, **1**, 11-13; *Continent Paint Resin News*, 1980, **18**(5), 106.
22. Palit, S. R., De, S. K., "Practical Physical Chemistry", *Science Book Agency*, Calcutta, 1971.
23. Burton, E. F., "The Physical Properties of Colloidal Solutions", *Longman, Green & Co.*, London, 1921, 16-18.
24. Ornstein, L. and Davis, B. J., "Disc Electrophoresis", *Distillation Products Industries, Division of Eastmand Kodak Co.*, 1959.
25. Kruyt, H. R., "Colloid Science", *Elsevier Publishing Co. Inc.*, New York, 1949, II, 251-520.
26. Viedermann, *Elkhricität*, 1893, **I**, 1007.
27. Pandey, A., PhD thesis, Ranchi University, 1972.
28. Overbeek, J. T. G. and Bungenberg De Jong, H. G., "Colloid Science", Kruyt, H. R., ed., *Elsevier Publishing Co.*, New York, 1949, II, 184-186.
29. Lewis, W. K., Squires, L. and Broughton, G., "Industrial Chemistry of Colloidal and Amorphous Material", *MacMillan Co.*, New York, 1943, 250.
30. Bungenberg, De Jong, "Colloid Science", Kruyt, H. R., ed., *Elsevier Publishing Co.*, New York, 1949, II, 244, 275, 325 and 339.
31. Bixler, H. J. and Michaeles, A. S., "Encyclopedia of Polymer Science", Mark, H. F. and Gaylord, N. G., ed., *Interscience Publishing Co.*, New York, 1969, 10, 765.
32. Roschchin, N. I., Shvetsov, G. M., Mina, S. A., Efimova, L. S. and Bril, A. S. (Nauchno-Proizuod Obedin), *Progress Khim-Farm. Zh.*, 1978, **12**(6), 131; *Chem. Abst.*, 1978, **89**(8), 135, 766.
33. Banerjee, P. K., Srivastava, B. C. and Kumar, S., *JOCCA*, 1984, **67**(6), 146.
34. Banerjee, P. K., Srivastava, B. C. and Kumar, S., *JOCCA*, 1984, **67**(8), 217.

next month's issue

The Honorary Editor has accepted the following papers for publication in the February issue:

Synthesis and study of water-soluble alkyd resins with high fatty chain contents *by D. Caron, A. Fradet and E. Marechal*
Comments on the galvanic action of zinc-rich paints *by S. Å. Lindquist, L. Meszaros and L. Svensson*
Synthesis of glyceryl monoaleuritate *by M. Mukherjee, R. N. Majee, S. Kumar and S. N. Mukherjee*
Coal tar and bitumen modified protective paints pigmented with aluminium flakes *by G. Cinti, A. Papo and G. Torriano*

We were delighted to hear from Past President Norman Bennett, who was President at the last Edinburgh Conference in 1959, about the possible derivation of the word Dragonara in the island of Malta G.C. where he now resides in retirement and we feel sure it will be of interest to readers of the Journal. The Dragonara Hotel in Edinburgh is the location of the 1985 Conference.

Dragonara

Sir – The name Dragonara probably originated in Malta, where in the district bounding Sliema to the north there is a peninsula about quarter of a mile in length and beyond it a reef area of the same length, in which most of the rocks are just submerged.

At the sea end of the peninsula a banker built a magnificent colonnaded house which became known as the Dragonara Palace. This was purchased with the land in the '70s by an American hotel group and later sold to a Group who renamed the hotel "Dragonara".

The derivation of the name, according to a local legend, comes from the sound made by the violent winter storms rushing over the slightly submerged rocks and a member of the academic staff at the University suggested that there are several sites in Malta and in the sister island Gozo which all have a common feature in that they are coastal and have a sea cave with a hole in the top through which water is ejected from rough seas but, as to the origin of the name, he was not able to state with certainty that the name came originally from Malta.

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Norman Bennett

11 November 1984

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occa meetings

Trent Valley Branch

Haloflex – recent advances in water-based coatings

The first technical meeting of the Trent Valley Branch on 25 October 1984 was generously sponsored by Imperial Chemical Industries PLC. Prior to the meeting a buffet was enjoyed by 25 members and guests followed by a slide-assisted paper presented by Mr R. G. Humphries, technical sales manager of ICI Mond Division, on the product, Haloflex.

In recent years there has been a steady trend towards water-based trade paints, particularly in the decorative field. When compared to solvent-borne coatings they offer several advantages including: low odour, non-flammability, freedom from toxicity, easy clean up of equipment and less problems with waste disposal. Water-based paints are thus environmentally and technologically more acceptable than their solvent-based counterparts.

In the anticorrosive primer field, water-based coatings have made little impact despite the steady advance in latex technology. This is largely due to a number of recognised limitations in paints formulated from the acrylic-rich copolymers specially developed for this application.

Typical problems encountered include a marked tendency to flash rust, poor results in accelerated laboratory tests such as salt spray and humidity due to the high permeability of the latex, inadequate chemical reistance and poor adhesion to metal surfaces.

Against this background ICI Mond Division have introduced a series of vinyl acrylic copolymer latices which overcome many of the existing problems. These are becoming well established and accepted in the market place and this paper traced the development of the copolymer, its formulation into a wide range of industrial paints and discussed a selection of worldwide case histories.

With the aid of slides and specimen panels, the applications and versatility of a group of these polymers sold under the trade name Haloflex were demonstrated.

Paints based on these new vinyl acrylic latices have already been adopted for a wide range of applications such as structural steel, galvanised steel, rust conversion coatings, fire retardant coatings, barrier coatings, masonry, asbestos cement and wood coatings, and in some cases an effective water-based primer may be the only type suitable for the particular environment and for the method of cleaning involved.

Particular benefits claimed for Haloflex include; reduced fire risk, easy clean up, fast dry, versatile in use (good adhesion to substrates, good intercoat adhesion), tough, flexible film, easy to use (one pot, air drying, brush, roller or spray application), chemical resistance (acids, alkalis and oils), and fire retardant (contains 64 per cent chlorine).

Development work is continuing in the ICI laboratories to improve still further the performance of primers and top coats based on the two existing Haloflex grades.

The technology of the production of the vinyl acrylic copolymers is such that it is a practical proposition to design other polymers tailor-made for specific market applications. Several such polymers are already under evaluation in various world markets.

A very lively and interesting discussion period followed the paper, and the vote of thanks proposed by Jonathan Bourne was enthusiastically endorsed by the 25 members and visitors present.

J. C. Ellis

London Section

The interpretation of viscometric measurements of flocculated suspensions

The second technical meeting of the 1984-85 session was held at the Pearly King, Bromley High Street, on Thursday 18 October 1984.

Despite the somewhat daunting title of "The interpretation of viscometric measurements of flocculated suspensions", the presentation of his paper by such an acknowledged expert as Norman Casson of the Bank of England Printing Works certainly held people's attention as he expanded his theme.

He emphasised the importance in any viscosity research that all units should be clearly defined and measurements kept as simple as possible. He described how the relationship of the viscosity of a suspension to the volume fraction of the dispersed solid can be described by an empirical equation which contains only one material constant. Mr Casson reviewed the results of his own interpretation of this, leading to what he believed was a more meaningful equation and supporting this by the practical results of his work.

After a lively and supportive question time a vote of thanks to the speaker was proposed by Mr D. Smith.

D. B. Bannington

Irish Section

Preservatives

The first meeting of the current session was held on Wednesday 4 September 1984 at the Ashling Hotel, Dublin, when Dr Karl Wagner of Riedel-de Haen of Hanover, W. Germany, presented a lecture entitled "Preservatives".

Dr Wagner assured the meeting that there are more than 100,000 different species of micro-organisms which may be encountered, and which are likely to cause deterioration of timber, paper, leather, paint etc. upon which they all tend to flourish.

Dr Wagner listed the most commonly found algae and fungi upon buildings, and gave details of recommended biocides for dealing with the problem.

After a lively discussion Mr D. Pountain proposed a vote of thanks to the speaker.

Control of quality of organic pigments

The second meeting of the current session was held on Thursday 11 October 1984 at the Ashling Hotel, Dublin, when Dr Simon Lawrence of Ciba Geigy gave a lecture entitled "Control of quality of organic pigments".

Dr Lawrence detailed the numerous tests which his company employ for pigments mainly used by the paint and printing ink industries.

After a lively discussion Mr P. McCrudden proposed a vote of thanks to the speaker.

R. G. Somerville

Thames Valley Section

Chemical pre-treatment

The above meeting was held at the Crest Motel, Beaconsfield on Thursday 18 October 1984.

The speaker, Mr C. Bates of Pyrene, in his introduction outlined the history of Pyrene and its involvement in chemical pre-treatment of metals including steel, aluminium, dipped galvanised and electro-deposited zinc, and various combinations of these metals. He then went on to list the various chemical preparation that were available and emphasised that the majority are still based on metal phosphate compounds but some are chromate-based and others are complex compounds of metals and organics.

As Mr Bates saw it, most people in the metal pre-fabrication industry are well aware of the advantages to be gained by chemical pre-treatment of steel. The in-factory treatment of articles fabricated from light gauge cold reduced steel is well documented in extensive pattern literature and a multiplicity of proprietary processes exist, many of which can be monitored and controlled by advanced electronic equipment. This is in contrast to a totally different situation which exists with the treatment of heavy gauge structural steel where chemical pre-treatment has found little application and is still considered something which can be dispensed with as thoroughly impracticable in the current maintenance practices in the construction industry.

The problems outlined were those of how to deal with structural steel outside of the factory environment and can be listed as follows:

1. The shear size of the objects being treated.
2. The relatively small number of items to be treated for a particular project.
3. The need to remove mill scale.
4. Difficulties of handling application and effluent disposal of chemicals in a non-factory environment.

For these reasons amongst others the use of chemical pre-treatment in the structural area has been limited.

On-site pre-treatment has been limited to some use of phosphating applied by hand lance, and brush applied T Wash for galvanised steel developed some years ago by British Rail's laboratories at Derby. Increased attention is now given to widening the scope of chemical pre-treatment into the structural field.

The speaker then went on to list how Pyrene would deal with the following metal substrates:

1. Galvanised steel

Examples were given of paint systems applied to freshly galvanised surfaces without pre-treatment compared with those applied over pre-treated galvanised steel and the consequent loss of adhesion of paint films applied over untreated galvanised steel.

For many years T Wash has been available as a mordant wash containing phosphoric acid solvents and copper. The presence of copper having the added advantage of darkening the coating and making it obvious which area has been coated, but it is less desirable on electro-chemical grounds. T Wash has given good service over the years but the market now recognises the need for something more advanced and happily the speaker was able to announce that such pre-treatments are now available.

2. Hot rolled steel

In the past it has only been treated under factory conditions but now due to the ever increasing use of both dry and wet blasting techniques, specially formulated pre-treatments at this stage have now been introduced and to date their performance has been encouraging. Possible areas of application will include ships plates, freight containers and storage tankers.

Finally, it should be mentioned that there is an ever increasing use of aluminium for structural work and this in itself necessitates the use of a chemical pre-treatment as an assurance against premature adhesion failure of the applied paint systems.

In conclusion, the speaker saw nothing but advantages both for the paint manufacturer and the paint user, and it can be concluded that the information given did not fall on deaf ears. A vote of thanks was given by Mr Peter Watson.

J. A. Gant

Midlands Section

A review of liquid resin systems for UV curing

The first lecture of the Midlands Section was held on Thursday 18 October 1984 at the Clarendon Suite, Stirling Road, Edgbaston, Birmingham. Members and guests heard Mr K. O'Hara give a talk entitled "Liquid resin systems for UV curing – a review".

Mr O'Hara commenced by describing some of the classes of resins that are used in UV curing. Vinyl-based prepolymers comprise the largest group of compounds used. They account for the biggest volume and find the most widespread use in terms of the types of applications for which they are used. These vinyl-based prepolymers could be divided broadly into two main types:

1. Unsaturated polyester
2. Acrylics

In the area of wood coatings, polyesters are still widely used. The acrylics may be used as prepolymers or modified with epoxy, urethane, polyester or silicone.

The production of these resins requires careful monitoring as it is necessary to maintain the right conditions in the reactor to suppress the free radical addition polymerisation reaction which is both rapid and exothermic.

To conclude his talk Mr O'Hara described some of the curing methods used in UV curing. The use of these systems is steadily increasing and new applications are continually being found. Throughout his talk the speaker showed numerous slides to illustrate the various raw materials that are used, the chemical reactions that take place, and typical formulae of UV curing systems.

The meeting closed with a vote of thanks proposed by the Chairman, Mr E. Wallace.

B. E. Myatt

Becker acquires Valspar

The sale by Cookson Group plc of Goodlass Wall & Co. Ltd together with Alexander, Fergusson & Co. Ltd to A. B. Wilh Becker of Sweden introduces a major new force into the highly competitive UK paint industry.

Goodlass Wall have, for many years, been among the leading producers of surface coatings in Britain. Becker has been established in Sweden for more than 100 years and manufactures consumer paints and industrial coatings, which are sold throughout Scandinavia, France, Germany, Spain and Canada – as well as in the UK.

Goodlass Wall and Alexander, Fergusson are manufacturers of decorative paints (Valspar brand) and industrial coatings. They will operate as autonomous subsidiaries of Wilhelm Becker Ltd, and will complement Becker's existing UK operations.

Reader Enquiry Service No. 1

Paint companies combine to build new plant

Two of Britain's fast growing paint companies are joining forces to build a new factory which will have the most advanced production equipment in Europe and heralds a new concept in the paint industry. The £6 million project in Buckingham is a half-share joint venture between the C. H. Industrials PLC Group and Petrofina (UK) Ltd, who are planning ahead for major expansion of their two companies, Cementone-Beaver and Sigma.

The two companies have formed a subsidiary, Buckingham Coatings, which will start production in early 1986 and have a maximum capacity of 23 million litres of paint a year. This new company will then supply both Cementone-Beaver and Sigma with their paint product ranges, allowing substantial scope for expansion in a highly cost-effective manner. Both companies stress that although they will have a joint manufacturing plant, their respective marketing companies will continue to be competitors and that the deal is in no way the beginning of a takeover by either side.

John Kinder, C.H.I. group managing director, explained: "This joint scheme will enable both of us to increase our sales while holding back on overhead costs – an ideal situation".

Reader Enquiry Service No. 2

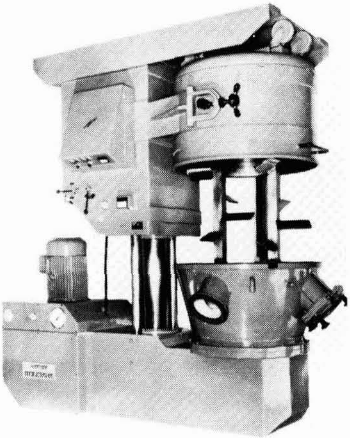
Hunt Chemical acquires Advanced Products Inc.

Hunt Chemical Corporation, a wholly owned subsidiary of the Olin Corporation, has announced its acquisition of Advanced Products Inc., California.

Advanced Products is a manufacturer of conductive inks and coatings used in the electronics and aerospace industries. Hunt Chemical is primarily a manufacturer of image-forming specialty chemicals for the microelectronics, printed circuit, photofinishing, graphic arts and electrostatics industries.

Reader Enquiry Service No. 3

products



The high powered planetary mixer from Molteni

High powered planetary mixer

Improved mixing of highly viscous products such as sealants, adhesives, mastics and other advanced polymers with difficult rheological characteristics is claimed to be made easier with the latest Molteni planetary mixer, available from D. H. Industries Ltd.

The Molteni PH 500 planetary mixer has been developed specifically by Molteni of Milan to simplify the manufacture of modern polymer-based materials and is said to employ a number of features which distinguish it from traditional general purpose mixers such as those commonly used in the paint industry.

Different blade formations and types can be fitted, depending on whether powders with liquids, slurries or pastes are being mixed. All surfaces in contact with the product are highly polished and bevelled or dished, wherever necessary, to facilitate easy cleaning. Mixing vessels are also available with machined walls for acceptance of pressure plates used in filling presses.

The Molteni planetary mixer is manufactured in material specifications capable of meeting most corrosive conditions and includes a stainless steel version. The machine can also be fitted with a vacuum cover where dehydration or deaeration is necessary. Explosion proof electrics can be added, where specified, for use in low flash areas.

Reader Enquiry Service No. 4

New colorimeter

Hunter Associaties Laboratory, Inc., USA, has announced the introduction and availability of the new D25-PC2. The D25-PC2 allows users to select one or more of HunterLab's five specially designed optical sensors and combine them with the lightweight, compact Epson HX-20 Laptop computer. The result is said to be an affordable colorimeter that allows any user to design a system that fits the demands of their particular application.

Standard features include one decimal place data, automatic standardization, transfer standardization, XYZ and one additional colour scale, system diagnostics and a built-in printer, to name a few. Of equal importance is that options such as Hunter L,a,b; CIELAB, haze; contrast ratio; YI D1925-70; WI E313-73; component colour difference and total colour differences can be selected separately, not in packages.

The D25-PC2 may be ordered without the Epson computer and connected directly to the user's own computer via an RS-232 port found on most computers in the market today.

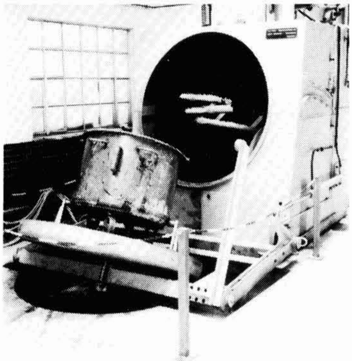
Application flexibility is another key feature of the D25-PC2. Users can choose from the field-proven, low cost optical sensors that include D25A and D25AA for flat opaque surfaces and materials of small particle size; the D25L for non-uniform surfaces and large particle size specimens; and the D25M for textured surfaces, fibres and chips.

The D25P sensor is used for transmission and haze measurements of film and liquids and the measurement of opaque materials where the specular light can be included or excluded.

Reader Enquiry Service No. 5

The HunterLab D25-PC2 Colorimeter shown with the Epson HX-20 computer (lower right) and (clockwise) the P, A, L, and M optical sensors

news



F-ex01, the explosion proof cleaning plant for mixing bowls and storage containers

Automatic vessel cleaning plant

Now available in the UK from Jenag Equipment is a fully automatic, explosion proof vessel cleaning system known as the F-ex01, which is said to offer significant improvements in cleaning efficiency and standards of safety.

The machine can be tailored easily to suit different scales of operation and is designed for use with fresh water, caustic, detergent and flammable or non-flammable solvents.

Equipped with its own hydraulic lifting and tipping turntable for maximum safety, the machine automatically transfers and locks the container into the machine, where it is rotated horizontally on its own axis throughout the washing and rinsing cycles. A centrifugal brushing action cleans the inside of the vessel whilst, simultaneously, a rotating buffing brush scrubs the outside surface. Following the rinsing cycle, the container is returned automatically to its original position, ready for removal from the turntable and immediate re-use.

Each cleaning cycle lasts for between 2-5 minutes, depending on degree of contamination. Separate reservoirs are incorporated in the machine for cleaning and rinsing solvents respectively and, where high toxicity levels are involved, a solvent fume extraction system is fitted to conform to factory health and safety regulations.
Reader Enquiry Service No. 6

Computerised system for laboratory automation and data management

Recently introduced by the Laboratory Automation Operation of Beckman is CALS (Computer Automated Laboratory System), which can be interfaced with a variety of analytical instruments.

CALS offers complete software packages for data acquisition, data reduction, data management, data storage and retrieval. Data may be acquired in analogue or digital form from intruments such as chromatographs and spectrophotometers. Alternatively, data may be entered manually via a keyboard. The acquired data can then be analysed using stored methods, and results reported according to user-specified formats.

The laboratory management package provides complete sample tracking facilities including log-on, label printing, work list generation, scheduling of analytical tests, test validation and approval, and reporting of data. A certificate of analysis may be generated and many other features such as an audit trail are designed to comply with GLP requirements.

Archiving of data may be achieved using magnetic tape storage or by communication with a central site computer.

The system, based on Hewlett Packard 1000 Series computers, is easily expandable in terms of hardware and software. For instance, optional software for auto-analyser links, simulated distillation analysis and gel permeation chromatography is available.
Reader Enquiry Service No. 7

literature

RSC launches CBNB

On 1 January 1985 the Royal Society of Chemistry (RSC) launched a new database covering vital technocommercial information to aid chemical and allied industries.

The material covered in the "NewsBase" is drawn from major journals and other serials containing news about the Chemical Industry. About 70 per cent of the journals covered are published in Europe, the remaining 30 per cent consists mainly of major US and Japanese journals to give balance to the coverage. Other sources of information and company literature, Government and EEC literature and market research and stockbroker reports are supplied directly to RSC.
Reader Enquiry Service No. 8

New product guide

George Meller Limited, suppliers of specialised pumps, valves, compressors, turbines and instrumentation to the process and pipeline industries, have issued a completely revised and updated version of their "Product Guide", which provides brief details on each item in their comprehensive equipment range.

Copies of the new leaflet are available through the reader enquiry service.
Reader Enquiry Service No. 9

bsi news

BSI publications are obtainable from: BSI, Sales Department, Linford Wood, Milton Keynes MK14 6LE (orders by post only).

All sales enquiries by telephone/telex should be directed to the new Enquiry Section at Milton Keynes on 0908 320066 (Telex 825777).

British Standards

The publications listed below are new and revised British Standards. An existing BSI publication bearing a number identical to one announced below is automatically withdrawn. In all other cases of supersession, details are given in the appropriate announcement and a corresponding entry appears in the "Standards withdrawn" section.

NOTE: The symbols used to denote the degree of correspondence between British Standards and International Standards have been altered to implement a new system agreed internationally and documented in ISO/IEC Guide 21/Addendum 1-1983.

The new symbol "≡" indicates an identical standard,

the symbol "=" indicates a (technically) equivalent standard, and

the symbol "≠" indicates a related standard.

The meaning of these three different levels of correspondence is defined in page xxiii of the 1984 BSI Catalogue but it refers to the symbols used before this change was implemented. The 1985 BSI Catalogue will introduce the new symbols.

BS 508:
Butan-1-ol for industrial use
BS 508: Part 2: 1984 ≠ISO 755/1, =ISO 755/2
Method of test
Instructions for sampling and test reports, and methods for distillation characteristics, aldehydes and ketones content and acidity. Also gives cross-references to relevant general methods. Partially supersedes BS 508: 1966 which is being amended.
4 page Gr 2

BS 1595:
Propan-2-ol (isopropyl alcohol) for industrial use
BS 1595: Part 2: 1984
≠ISO 756/1, =ISO 756/2, =ISO 756/3
Methods of test
Instructions for sampling and test reports, and methods for distillation characteristics, aldehydes and ketones content, acidity and a


SURFACE COATINGS
FORMULATION TESTING

test for miscibility with water. Also gives cross-references to relevant general methods. Partially supersedes BS 1595: 1965 which is being amended.
4 page Gr 3

Special announcements

ISO 1250: 1972 Mineral solvents for paints – White spirits and related hydrocarbon solvents
ISO/TC 35 "Paints and varnishes" has reported that an ad hoc Working Group which was considering the revision of ISO 1250 could not reach agreement on certain of the requirements to be specified in a new edition of the standard. Also it reported that "in some of the most important user countries, the standard is not used at all".

Because of the difficulties in agreeing the "aromatic" content of different grades of white spirit and because of the apparent lack of international use, the Secretariat of ISO/TC 35 has decided to propose the withdrawal of ISO 1250. Comments on this proposal are invited and these should be sent to the Secretary of PVC/3, Mr W. G. Cobbett, at Head Office, by 23 November 1984.

Even if ISO 1250 is withdrawn, the corresponding British Standard, BS 245: 1976 "Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes" will be maintained, subject to its review by the Technical Committee.

British Standards reviewed and confirmed

2621-5: 1979
Glycerol (glycerine)

5711:
Methods of sampling and test for glycerol.
5711: Part 0: 1979 General introduction.
5711: Part 1: 1979 Sampling and test methods: general.
5711: Part 2: 1979 Methods of sampling.
5711: Part 3: 1979 Determination of glycerol content.
5711: Part 4: 1979 Determination of density and relative density.
5711: Part 5: 1979 Determination of alkalinity or acidity; volumetric method.
5711: Part 6: 1979 Determination of ash: gravimetric method.
5711: Part 7: 1979 Determination of sulphated ash: gravimetric method.
5711: Part 8: 1979 Determination of water content: Karl Fischer method.
5711: Part 9: 1979 Calculation of matter (organic) non-glycerol (MONG).
5711: Part 10: 1979 Determination of arsenic content: silver diethyldithiocarbamate method.
5711: Part 11: 19J9 Determination of propane-1,3-diol content; gas chromatographic method.
5711: Part 12: 1979 Determination of chloride content.
5711: Part 13: 1979 Limit test for chloride.
5711: Part 14: 1979 Limit test for organic chloride.
5711: Part 15: 1979 Determination of heavy metals content.
5711: Part 16: 1979 Determination of iron content.
5711: Part 17: 1983 Limit test for lead.
5711: aart 18: 1979 Detection of sugars.
5711: Part·19: 1979 Assessment of odour.
5711: Part 20: 1979 Test for reducing substances.
5711: Part 21: 1979 Determination of saponification equivalent.
5711: Part 22: 1979 Additional methods of test (non-specification).

New work started

British Standards X 33 Cold curing epoxy paint primer for aeronautical purposes
Will specify the requirements for a chromate pigmented epoxy primer suitable for application to a suitably pre-treated metallic and composite material surface.

Specification for varnish for aeronautical purposes
Will specify the requirements for a water resistant varnish for aeronautical purposes for application to wood and certain plastics.

occa conference

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?"

Below we include some of the summaries of the papers to be presented and the biographies of their authors. Further details will be published in forthcoming issues of the *Journal.*

Summaries of papers and biographies of authors

Dr G. C. Fettis

Keynote address

Dr G. C. Fettis

The coatings industry has been faced with new problems due to the introduction of new substrates and new materials, particularly in the last five years. The changes should be viewed as beneficial because the challenge they present is leading to the development of new technologies and helping revitalise the industry.

The need for new products has brought fresh impetus to research spending which is on the increase. In part this is the reason behind the growth by acquisition of a number of paint companies which are becoming increasingly internationally based to maximise market place benefit from the research spend.

Biography

Born in 1935 in Edzell in Scotland Dr G. C. Fettis was educated at the Bo'ness Academy and Edinburgh University where he obtained a 1st Class Honours degree in Chemistry. He gained a PhD on "The Gas Phase Reaction Kinetics of Fluorine and Bromine Atoms".

He joined ICI General Chemicals Division (now Mond Division) as a chemist in 1960 and later became a group manager in Research Department in 1966 where he was responsible for the development of the ICI process for vinyl chloride production. In 1969 he became an assistant works manager in ICI's Castner Kellner Works, responsible for the production of acetylene from carbide, chlorinated hydrocarbons, sodium and sulphur compounds production.

In 1973 he moved to the Paints Division of ICI as works manager of the Stowmarket Paint Factory and in 1978 became division research manager. Now responsible for research worldwide in the ICI Paints Group of Companies, he is also a member of the International Steering Committee which guides strategy for the ICI World Paint Group.

Effect of titanium dioxide pigments on the cure of thermosetting film

T. Entwistle

Summary

In the past the rate of cure of thermosetting paint films has generally been little affected by the grade of titanium dioxide pigment used. However, with the increasing pressure to reduce environmental pollution due to solvent emission the use of high solids systems has become more widespread. This necessitates the use of lower molecular weight resins, together with catalysts to assist the crosslinking, and it has been found that with some such systems the rate of cure can be affected by the grade of pigment.

In addition, in order to conserve energy, there is an increasing tendency to lower the stoving temperature, particularly with powder coatings, and again it has been found that the grade of pigment can affect the rate of cure.

Examples of both systems will be given and how the resultant differences in cure can affect film properties.

T. Entwistle

Biography

Mr T. Entwistle has been in the paint industry for over 35 years, 15 years in the laboratories of paint manufacturing companies and the last 21 years in the Technical Service Department of Tioxide UK Ltd. Currently he is section manager responsible for advice on the use of TiO_2 in industrial paints, powders and printing inks. From 1969 to 1973 he was seconded to an associate company, South Africa Titan Products as technical service manager.

He has been a member of OCCA since 1953. Whilst in South Africa he served as Hon. Secretary of the South Africa Section and currently represents the South Africa Division on Council. He is currently a Vice President and is a member of the Finance Committee and has previously served on the Professional Grades and Publications Committees.

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

G. H. Hutchinson

Summary

For many years, water-based inks have attracted the attention of the printing and packaging industries. In the last decade, several factors have stimulated the interest, namely the growing attention to environmental pollution problems, health and safety legislation, eliminating residual print odour from food packaging printing and the conservancy of organic solvents. The influence of these factors on the progress of water-based inks and allied products is discussed, and some existing and potential applications for the products in appropriate printing processes are described.

Recent years have seen steady advances in the technology of water-based inks and clear coatings for printing paper and board substrates. More knowledge of ink and paper relationships, for example, the influence of smoothness and porosity of the substrate on print quality, is being gathered and this is fundamental to the achievement of the best printing results. More difficult problems remain in the formulation of water-based inks for printing impermeable plastic and metallic substrates, not least the high energy requirements for ink-drying at high printing speeds and the problems of achieving film-forming properties comparable to those from the best available organic solvent based inks.

Developments in the relevant materials technology are discussed with emphasis on the pigment and vehicle, also the likely trends in drying techniques involving use of Infra-Red, Microwave and Radio Frequency electric heating.

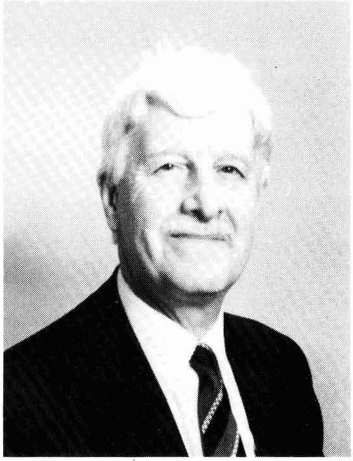
Biography

G. H. Hutchinson, CChem, MRSC, FInstPet, FTSC, aged 65, is a consultant with Croda International Inks Federation.

In his early career he engaged in research and development on refined and processed drying oils and varnishes for paints and printing inks. Mr Hutchinson has some 25 years experience of printing inks. He became research and development director of A. B. Fleming, Edinburgh in 1969 and after the acquisition of Flemings by Croda International, technical director of Croda Inks Ltd from 1971. He was appointed deputy president of Croda International Inks Federation in 1981 with special responsibilities for technical and marketing liaison with Croda's printing ink operations worldwide. On reaching retirement age he became a company consultant with continuing responsibilities for technical liaison with UK and overseas printing ink companies.

An active member of OCCA since 1947, he has served on Hull, Scottish and Eastern Branch committees. He is a past member of Council and was chairman of the Eastern Branch 1967-1969. He has contributed to Student section lectures and OCCA exhibition school lectures. He has also published several papers in *JOCCA* and was a one time contributor of "Drying oils, driers and varnishes" section to "Reports on progress in applied chemistry".

Mr Hutchinson is a past member of the Council of PIRA and was chairman of the Technical Committee of the Society of British Printing Ink Manufacturers from 1974 to 1983.



G. H. Hutchinson

Formulation of solvent-based paints pigmented with titanium dioxide

Dr J. E. McNutt

Ternary diagrams are useful for visualizing the relationships among millbase, finished paint, and dry film compositions. Such diagrams will be explained. Guidelines will be given for formulating millbases for various dispersion methods. Examples will

be given of using these diagrams to: develop instructions for millbase preparation and letdown; anticipate potential for shock and seeding problems, and how to avoid these letdown problems.

Dr J. E. McNutt

Biography

Dr J. E. McNutt has worked for Du Pont since 1954, when he received a PhD in Engineering from Carnegie Institute of Technology in Pittsburgh, PA. His early career was devoted to R&D on surface phenomena, including metallic corrosion and deposition of coatings. In recent years he has been assigned to technical service for customers using titanium dioxide in paints and plastics.

New materials for the furniture industry

A. J. Sparkes

Summary

Although solid wood is still an important raw material for the manufacture of chair frames and some types of reproduction furniture, wood based sheet materials such as hardboard, particle board and more recently medium density fibreboard (MDF) have largely replaced wood in the cabinet industry. The introduction and widespread use of these substrates has been paralleled by significant development of a wide range of furniture surfacing materials starting with traditional wood veneers surfaced with lacquer and including plastics laminates, and PVC and paper foils. The successful use of these surfacing materials has been dependent upon the development of sophisticated adhesives systems for use with high speed laminating equipment.

Medium density fibreboard has created new opportunities for designers as its homogenity derived from the close packing of the wood fibres allows intricate machining operations to be carried out on the faces and edges of panels without the need for elaborate treatment of the exposed surfaces. An improved sealer was however required to treat the relatively porous edges of MDF. Many examples of furniture and other items manufactured from MDF finished by stain and lacquer or direct painting are seen in the shops.

Possible health hazards due to formaldehyde emitted from MDF and particle boards manufactured with urea formaldehyde binder have caused some concern in North America and parts of Europe. Control measures have included the use of resins with a lower level of free formaldehyde, neutralising treatments based on ammonia, and the application of surfacing materials which slow down the rate of emission of formaldehyde from the board. The few problems that have occurred in the UK have been associated with the storage or use of products containing formaldehyde in areas of low ventilation. However in order to reduce public concern, further development of low formaldehyde emission boards will be necessary.

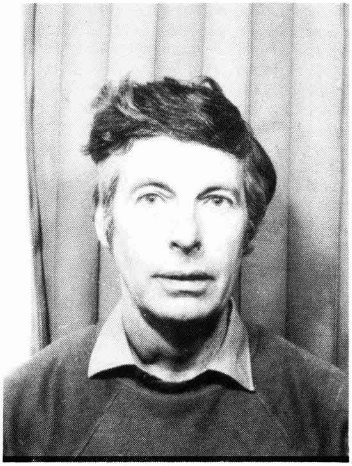
In the area of surfacing materials, wood veneers are still widely used despite their relatively high cost and their exacting production requirements. Bonding conditions have not changed significantly in recent years although the industry is showing an increasing interest in PVAC adhesives as an alternative to the widely used UF adhesives. Most wood based domestic furniture is finished with nitrocellulose or precatalysed lacquer. More resistant chemically converted systems are used for educational, office, hotel and other types of contract furniture. The present role of water borne finishes will also be considered.

Plastics laminates were the first man made surfacing materials to replace wood veneers. Here recent interest has been centred on the development of a continuous manufacturing system as an alternative to the well established high pressure flat press system. Apart from the advantage of the supply of plastics laminate in long length reels, the development has resulted in a reduction in manufacturing costs.

Melamine resin impregnated paper foils are being used for all layers of the first generation of continuous laminates to accommodate the lower laminating pressures and the short time under pressure. However, continuous machines capable of applying higher pressures and developments in resin technology will soon enable laminate manufacturers to use phenolic based backing foils to impart greater flexibility at lower cost.

Although PVC foils had a successful period of use in the furniture industry, increased costs of these oil derived materials has resulted in a decline in use in favour of paper foils. Resin technology has played an important part in the development of paper foils both in respect of pre and post printing impregnating treatments and surface coatings. Electron beam and UV radiation cured coatings are likely to be used increasingly on decorative foils for the furniture industry.

The final part of the article is concerned with the assessment of the performance characteristics of this wide range of furniture surfacing materials using test methods that have been developed in conjunction with members of the Paintmakers Association and suppliers of plastics laminates and decorative foils. These methods and the complimentary specifications are now incorporated in British Standards and are used widely by manufacturers and purchasing authorities.



A. J. Sparkes

Biography

Mr A. J. Sparkes BSc graduated from Imperial College, London. He spent nine years at the Bowater Development Corporation Research Laboratory at Northfleet, Kent, working on projects concerned with the physical and optical characteristics of paper surfaces and the machining characteristics of hardboard.

He has spent 18 years at the Furniture Industry Research Association, latterly as head of the materials technology section. Mr Sparkes has written papers on the use of hot melt adhesives, bonding wood veneers with UF adhesive, continuous plastics laminates and paper foils. He is FIRA's representative on BSI and ISO Committees concerned with finish test methods and specifications.

Oxo-acetates – new solvents for high solid paint formulations

Dr M. A. Taylor and J. P. de Greef

Summary

The use of high solids coatings, which is already established to some extent in the US, will spread to Europe and the rest of the world, either in response to environmental legislation and concerns, or as these coatings show cost/performance benefits over traditional, lower solids paints.

High solids coatings need powerful, less volatile solvents than those used in traditional paints. Unfortunately, few commercial solvents meet these requirements, and some of those that do are suspect with respect to toxicity.

Oxo-acetates, recently introduced to the US market under the trade name "Exxate", overcome most of these difficulties. The Exxate grades give advantages in the manufacture, formulation and application of coatings based on a range of resins. In addition, preliminary tests show that Exxate grades are comparatively non-toxic.

The benefits resulting from the use of the Exxate grades will be discussed in detail and samples will be available for inspection.

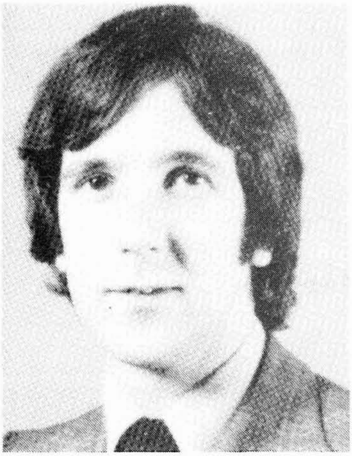
Biographies

Mr J. P. de Greef is a senior staff engineer in Esso Chemical Europe's Solvents Technology Division, Machelen, Belgium. In over 16 years in the petrochemical industry he has served in a wide variety of positions mainly in product applications and marketing technical support groups across Europe. He graduated as chemical engineer from the Catholic University of Leuven (Belgium).

J. P. de Greef

Dr Malcolm A. Taylor is currently development manager, Solvents Division at Esso Chemical Ltd, Southampton.

He joined Esso Chemical immediately upon graduating from Edinburgh University.



Dr M. A. Taylor

In the eight years since then he has worked in the sales and marketing groups of Plastics Division and Solvents Division as well as a period spent as quality control group head in the Elastomers Department.

Parameters controlling colour acceptance, part II

H. G. Stephen

Summary

This paper follows part I which was presented at FATIPEC, Lugano, 1984.

The two papers, taken together, show that improved Universal Colorants may be formulated so that they will produce minimum energy level differentials, leading to accurate colour matches free from film defects, when added to decorative paints. The paints to be tinted must also be formulated for optimum electrostatic and thermodynamic properties, to normalise the effect of the entropy level of the entering colorant.

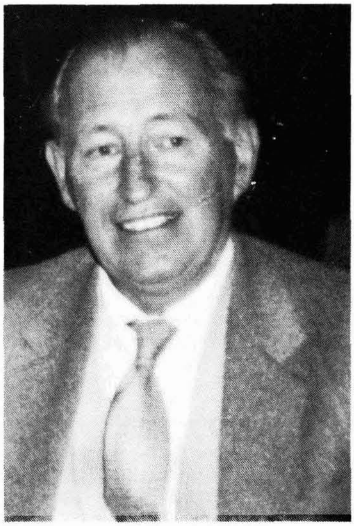
From the foregoing it can be seen that no magic single ingredient can be added to any particular paint formulation to give acceptable appearance, with a range of Universal Colorants. It is possible, with refined formulation techniques, to develop higher standards of performance than is currently available with commercial colorants.

Biography

Before his retirement, Mr H. G. Stephen was technical manager of Kemrez Chemicals, with whom he served 20 of his 45 years in the surface coatings industry in development, sales, and research functions.

Long a leader in coatings education in Australia, he was senior lecturer in C & G Paint Tech. in the Melbourne College of Decoration. A foundation member of the council of this college, he served it for 21 years, for a while as Council President.

He has delivered some 14 papers to OCCAA state branches and conventions, as well as to the FSCT (St. Louis, 1979) and FATIPEC (Lugano, 1984).



H. G. Stephen

Progress in Timber Finishing in Britain

R. J. Woodbridge and E. A. Hilditch

Summary

Within the last few years there has been a considerable growth in sales of specialist coatings for timber, ranging from low build transparent stains to high build 'microporous' opaque coatings. The specifier is often totally confused as to which type of coating to use for a specific purpose or the relative performance of different types.

The paper will review traditional and modern use of timber and the particular problems posed by the British climate.

The nature of timber and problems relating to type and quality will be considered, together with the influences of joinery design and use in practice.

Basic requirements for successful finishing systems will be reviewed, followed by a consideration of the factors which must be taken into account in selecting the right finish for specific circumstances. Both technical and aesthetic considerations apply to this choice.

Biographies

Mr R. J. Woodbridge, CChem, FRSC, FTSC is the technical manager, Berger Decorative Paints, and Vice President of the Paint Research Association. Educated at St. Brendans College, Bristol, he continued his higher education at Merchant Venturer's Technical College Bristol.

In 1941 he joined John Hall & Sons, which subsequently became part of the Berger, Jenson & Nicholson Group. In the 43 years he has been with Berger, he has worked almost entirely in the field of decorative paints.

In the early 50's he specialised in emulsion paint technology, and was associated with the development of a number of major innovations in this field. Previous papers presented include "Evaluation of emulsion paints" *(JOCCA*, June 1955), which discussed effect of paint formulation on permeability, and more recently at PRA Symposia on "Air and polymer extended decorative paints" and "The role of aqueous coatings in wood finishing".

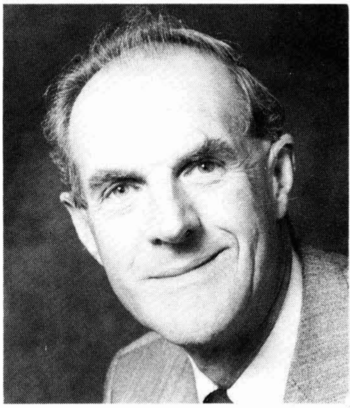
R. J. Woodbridge

E. A. Hilditch, BSc, CChem, FIWSC, FRSC, was educated at De Aston School, Market Rasen and City of Gloucester Technical College.

Married with one son, he did his National Service in the RAF, and joined Cuprinol Ltd in 1954, where he became technical director in 1962.

Mr Hilditch is widely involved in all aspects of research, development and application of wood preservatives, wood stains (including some of the earliest commercial products to be placed on the UK market) other wood finishes, damp proofers and paint driers.

Currently Vice President of the British Wood Preserving Association and one time Chairman of the technical committee, he is also a member of a number of British Standard committees dealing with wood preservatives.



E. A. Hilditch

occa news

Obituary

S. Leonard Davidson

Frank J. Borrelle writes:

It is with much sorrow that I advise you of the passing of S. Leonard Davidson, a Past-President and Honorary Member of the Federation. He was 66.

S. Leonard Davidson

Leonard, a graduate of the University of California, began his 40 year career in the coatings industry with National Lead Co., San Francisco, in 1943. He served as chief chemist from 1944 until 1951, when he was transferred to Perth Amboy, NJ, as chief, paint development.

In 1967, he moved to NL's Hightstown, NJ laboratories as a senior chemist and colour consultant. He was a regulatory affairs specialist with NL Industries at the time of his retirement on 1 March 1983.

Leonard was President of the Golden Gate Society in 1950, and President of the New York Society in 1965. He received the latter's Kienle and PaVaC Awards in 1960 and 1968, respectively.

In 1954, he was Chairman of the New York Society's Technical Subcommittee on "Colour Matching in Production" and presented that group's still well known and in-use "Colour Matching Box" at the Federation's Annual Meeting in 1955.

He initiated the evening colour courses sponsored by the New York Society and served as the intructor for several years. He wrote several papers on coatings and pigments technology and lectured in this country and abroad on these subjects as well as "Colour Control in the Coatings Industry". He received the Federation's Bruning Award in 1969 for his contributions to the science of colour in the coatings industry.

He served as President of the Federation in 1970-71 and was elected to Federation Honorary Membership in 1981.

He was a member of ACS, ASTM, and Treasurer of both the Inter-Society Colour Council and the Munsell Foundation. He was also a long-time member of the Oil and Colour Chemists' Association and earlier this year was elected a Vice-President and was admitted as an FTSC.

He is survived by his wife, Edythe; two sons, Charles and John; a daughter, Susan; and three grandchildren.

new members

Ordinary members

Boaler, G. G. *(Manchester)*
Butterworth, J. S., BSc *(London)*
Clelford, P., BSc *(London)*
Garton, K. J. *(Thames Valley)*
Gee, P. *(Midlands – Trent Valley)*
Haythornthwaite, D., BSc *(Manchester)*
Metcalf, P. V. *(London)*
Miron, J., PhD *(General Overseas – USA)*
Nelson, P. C., BSc *(Manchester)*
Pilot, H. *(General Overseas – Mauritius)*

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