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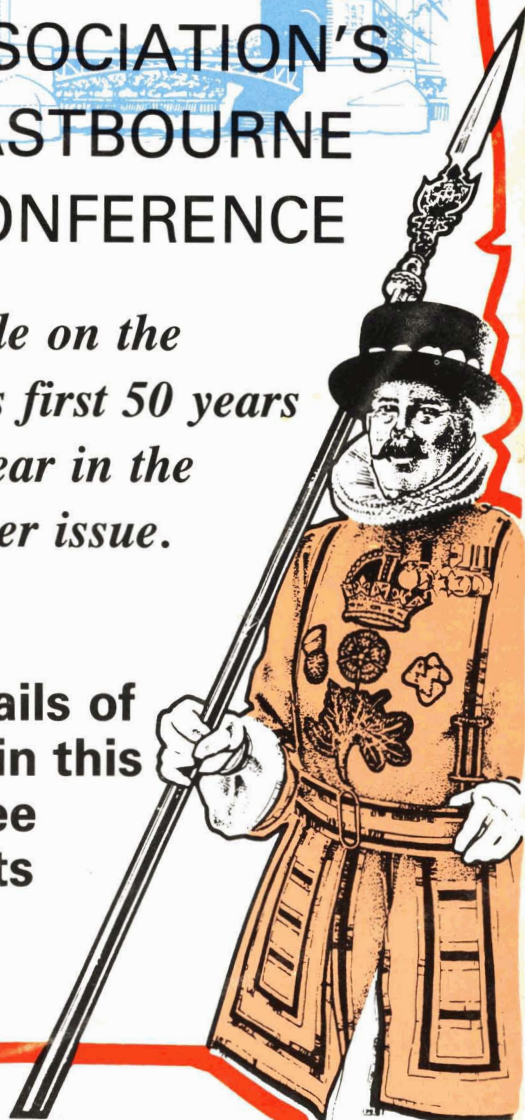
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**CHLORINATED POLYETHYLENE
CHLORINATED POLYPROPYLENE
CHLORINATED RUBBERS
etc.**

APPLICATION

- Anticorrosive coatings
- Road marking paints
- Gravure printing inks
- Adhesives

ADHESIVE GRADES (Low Chlorine Type)

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adhesives for untreated
Polypropylenes
- Primer for plastics



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Editorial correspondence should be
addressed to the Hon. Editor, JOCCA,
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General correspondence should be
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Assistant Editor:

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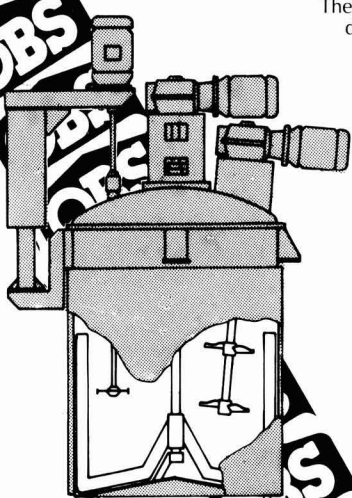


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The versatile paint resin—'Haloflex'*

R. G. Humphries

ICI Chemicals and Polymers Group Resins and Surface Coatings Intermediates Business, PO Box 13, The Heath, Runcorn, Cheshire WA7 4QF, UK

Abstract

This paper describes the development of a unique family of vinyl acrylic water-based latices designed by ICI Mond Division for the coatings industry.

The latices are achieving considerable success as universal primers on blast cleaned and galvanised steel and have already exceeded original expectations in performance.

Progress in anti-corrosion paints are reviewed and a number of newcomers to the Haloflex family will be introduced. These include a specifically modified polymer with very low water vapour transmission rates designed as a replacement for polyethylene laminates in the packaging industry and high molecular weight polymers with very good flexibility at low temperatures, ideal for coatings and contact adhesives.

Introduction

Water borne coatings offer several immediate and tangible benefits over their solvent based counterparts—non-flammability, freedom from toxicity during manufacture and application, easier clean-up of equipment and less problems with waste disposal.

Initial attempts to penetrate the steel structures market were made with acrylic or modified acrylic polymers. Whilst acrylics have many admirable properties their high permeability to water vapour and oxygen makes them unsuitable for use as primers. Furthermore they are prone to early rusting and flash rusting, give poor results in accelerated tests and exhibit variable adhesion on a number of substrates.

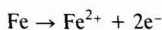
ICI Mond Division introduced a new family of water borne vinyl co-polymer latices to the market in the early 1980's specially designed to cope with the above problems. These polymers gained rapid acceptance in the market and can be used to formulate primers which compare favourably with existing high performance solvent based anti-corrosive paints. Recent developments in these primers are discussed and some of the newer development polymers in the range introduced.

Polymer design – 'Haloflex' 202/'Haloflex' 202S

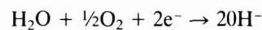
To be effective, primers must have good adhesion, good mechanical properties, good weathering properties and most important of all, they must prevent substrate corrosion. As mentioned previously, this latter aspect is where many of the existing water-based coatings appear to fail.

Corrosion is quite simply the tendency for iron to revert to its original state via the following anodic and cathodic reactions:

Anodic



Cathodic



In principle, therefore, corrosion could be prevented by coating with a film that is virtually impervious to water and oxygen, but which also has a sufficiently high electrical resistance to inhibit the passage of charge across the polymer-metal interface. The resistance and conductivity are in turn dependent on the water content/uptake of the film as this determines the diffusion characteristics and ion permeability within the film. Low permeability to water vapour and hence low water pick-up were therefore extremely important in the design of these new polymers¹.

ICI Chemicals and Polymers Group technologists were already familiar with the fact that polymers with a high chlorine content exhibited low water vapour permeability from ICI's extensive chlorinated rubber technology. Applying the same principles to the development of an air drying water borne copolymer led to the evaluation and acceptance of copolymers of vinyl chloride and vinylidene chloride. They provided the degree of low permeability required and the correct balance of toughness and flexibility as the vinyl chloride is a hard monomer compared to the relatively soft vinylidene chloride. A further refinement was copolymerisation with acrylic ester monomers to give better control over film formation, improve thermal and UV stability, improve specific adhesion and still maintain the excellent barrier properties. The latex is also manufactured by emulsion polymerisation in the virtual absence of colloids.

Primers based on these latices give outstanding results on blast cleaned steel. In addition, the high chlorine content of the polymer (64%) offers possibilities in flame retardant and intumescent paints.

'Haloflex' 202 can therefore be described as an acrylate modified vinyl chloride – vinylidene chloride copolymer. 'Haloflex' 202S on the other hand is a prestabilised grade equivalent in performance to 202, but with the added advantages of better tropical storage and much simplified paint manufacture.

Paint formulation

At this stage it might be useful to discuss in more detail a typical 'Haloflex' based paint in order to outline the principles of formulation, choice of ingredients and review the properties. A general purpose anticorrosive primer has been selected based on 'Haloflex' 202. This section also includes some recent anticorrosive pigment evaluations.

A general purpose anti-corrosive primer is listed in Formulation 1. This was one of the early development formulations selected principally on its outstanding salt spray performance. The method of preparation outlined is particularly suitable for high speed cavitation equipment.

* Presented at the OCCA South African Division 11th National Symposium held on 21 October, 1986 at Durban

Formulation 1

General purpose anticorrosive primer based on 'Haloflex' 202

Premix	% W/W
Water	8.09
Defoamer	0.20
Thickening agent	0.20
Dispersing Aid	0.20
Latex stabiliser (30% w/w solution)	4.23
Coalescing solvent	1.98
Dispersion	
Anticorrosive Pigment	5.73
Extender	15.42
Colouring Pigment	2.57
Let down	
'Haloflex' 202	59.00
Fungicide	0.10
Water to viscosity	2.28
	100.00
PVC	20.4
Volume solids (%)	43
pH	4-5

Most of the generic ingredients above will be familiar to the formulator of latex paints, e.g. defoamers, dispersing aids, coalescing solvents, anticorrosive pigments etc but inclusion of latex stabiliser merits a few words of explanation as does our decision to formulate at acid pH.

Latex stabilisation

The 'Haloflex' emulsification polymerisations are carried out in the virtual absence of surfactants and protective colloids. This procedure has the advantage that protective colloids which can impair latex coalescence are avoided. Furthermore, the low permeability of the latex is preserved enabling it to be used in barrier coating applications. When it comes to paint formulation additional surfactant and dispersing agent can be selected to optimise paint performance and stability. From the latter view point it was found that block copolymers of the ethylene oxide/propylene oxide types (Synperonic PE/F87) were extremely effective. The optimum level was ultimately established to be around 3% by weight on polymer solids and permeability and electron microscopy studies established that the Synperonic stabiliser accelerated the rate and extent of the coalescence and had a role to play as a pigment dispersant and mill base stabiliser².

pH

It will have been noted that the paints are formulated around pH 4-5 whereas most conventional emulsion paints are formulated at pH 7-9. This is because copolymer latices such as 'Haloflex' with a high chlorine content tend to undergo dehydrochlorination at high pH levels. In fact the higher the pH the higher is the rate of dehydrochlorination. The main side effects of this reaction is a progressive drift to the lower pH or a corresponding increase in the chloride ion concentration in the paint or latex aqueous phase. The rate of dehydrochlorination could be reduced by reducing the chlorine content of the latex but this would be to the

detriment of the all important barrier properties of the polymer. A series of experimental paints were stored at 40°C and the pH monitored with time. It was found that the pH ultimately fell to 4 regardless of the initial figure and furthermore at pH4 the increase in chloride ions had virtually ceased indicating that the dehydrochlorination reaction was complete. Although it was possible to buffer the paints at the higher pH the amount of buffer required for the chlorine rich latices was such as to impair the protective properties of the finish paint and for this reason it was decided to formulate at acid pH. Whilst this limits the formulator to a certain extent in the choice of extenders and certain thickening agents there was still plenty of extenders, surfactants and anti-corrosive pigments around to enable the formulation of highly effective anti-corrosive paints. In fact, formulating at a slightly acid pH has given a number of tangible advantages:

- better specific adhesion particularly to galvanized surfaces.
- virtual elimination of flash rusting on blast cleaned surfaces.

Flash rusting is essentially the oxidation of ferrous to ferric irons which under high pH conditions produces the distinctive rust spots of ferric hydroxide by localised precipitation³.

- Zinc phosphate has been widely evaluated in many of our development formulations and outstanding results in accelerated corrosion tests have been obtained in primers containing around 5-6% volume concentration of the pigment. This can partly be attributed to the solubility of the zinc phosphate in the slightly acidic paint.
- The paints are more tolerant to lower standards of surface preparation and perform well on blast cleaned surfaces corresponding to Sa2 of Swedish Standard SIS 055900-1967.

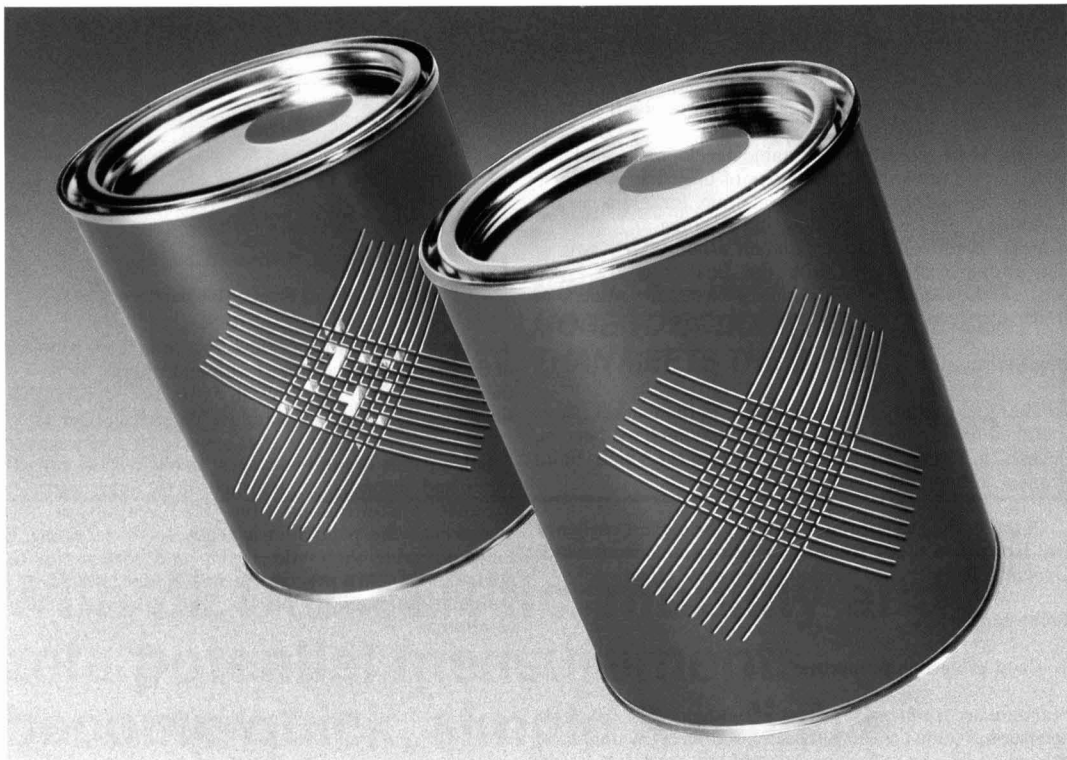
Anti-corrosive pigments

Zinc phosphate has already been mentioned and is very much the standard by which the other anti-corrosive pigments are judged. At levels of around 6% by volume in a paint of total PVC 20-30% service lives in excess of four years have been obtained in our most aggressive industrial exposure sites with dry film builds of 80 microns. In other industrial applications where specifications are usually asking for 250 hours salt spray from a dry film thickness of 25 microns a faster acting pigment may be required. Of the many evaluated by the development teams the modified aluminium triphosphates (K white) have given very good results at equivalent volume concentrations to the zinc phosphate. They are, however, a little expensive and the best compromise may be to include them as a partial volume replacement for the Zinc Phosphate.

The new range of calcium silica ion exchange pigments from W. R. Grace sold under the tradename, Shieldex, have also been studied and have given equivalent performance to Zinc Phosphate in anti-corrosive tests.

Earlier work showed Zinc Chromate to be effective in very small levels (around 1% volume concentration in the paint), but problems with localised flash rusting believed to be caused by traces of Sodium Chromate in the pigment forced us to withdraw this option.

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Extenders

Extenders play a big part in ultimate paint performance. Initial recommendations were based on Barytes which gave very good salt spray results on blast cleaned steel. More recently, in the search for a general purpose primer where the substrate choice has been widened to include galvanized steel, talc extenders are preferred on the basis of their better adhesion. The PVC has also been increased from 20 to 30%.

Thickening agents

Initial work was largely carried out with the cellulosic thickeners and whilst their ability to hold the densest pigments in suspension could not be questioned their flow and levelling during brush application left much to be desired. More recently considerable development work has been carried out with a new series of urethane thickeners which, although not as effective gram for gram as the cellulose, give very good flow and levelling.

Most recent formulations contain around 0.8% w/w of the urethane thickener with the final viscosity being controlled by the type and level of coalescing solvent of which the water miscible types are preferred.

Dispersing aids

As the product has gained acceptance in the market place, the need for a wide variety of formulations have become necessary. These include: glossy primers, high build primers, primers for dip applications etc.

Of these, the glossy primers offered the biggest challenge and led to a detailed study of dispersing aids from which Orotan 850 appears to be among the most effective.

Paint properties

1. Good protection properties

Maintenance primers for steel should prevent substrate corrosion. Paints based on these new chloropolymers have proved to be very effective primers. In accelerated tests they exhibit a performance previously unattainable with latex paints due to the unique barrier properties of the fully coalesced film as explained earlier. In salt spray tests they easily outperform existing acrylic rich and acrylic modified latices and compare favourably with the high performance solvent based types such as epoxy, vinyl and chlorinated rubber.

Paint film thickness has a part to play and detailed testing both in accelerated and natural exposure sites has shown that a dry film thickness of 50 microns over the blast profile is required. It is also advisable that excessively thick films be avoided in order to achieve through drying and an upper limit of 80 microns dry film is ideal. A relatively large trial in a section of one of our Runcorn plants has shown that a primer coating of 80µm dry was capable of giving 4-5 years in an industrial atmosphere without overcoating before any repainting was required.

2. Fast drying

The drying rates of water borne paints are of major importance to anyone considering their exterior use. The dry time of the coatings depends on temperature, humidity

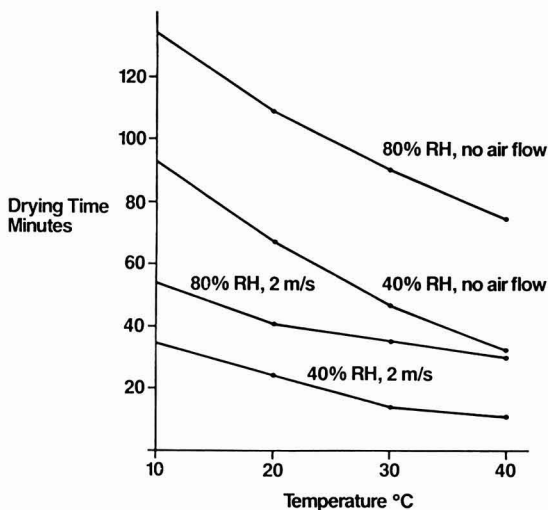


Figure 1. Drying times of 100 micron dry film.

and, most importantly, air flow. Figure 1 gives an indication of the drying times achieved, with and without air flow over the surface. Air flow is thus an extremely important factor since as little as 2 m/s will reduce the drying time by 50%.

Latex paints dry in two stages; firstly evaporation of the water and secondly, coalescence of the latex particles to produce a continuous film. On completion of the coalescence the paint film is ready for overcoating. The following table shows the drying and coalescence times achieved in two practical trials under two different temperature/humidity regimes with relatively static air conditions.

Table 1

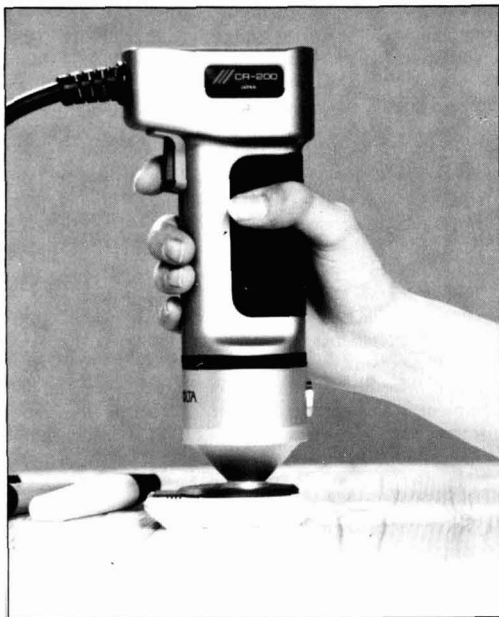
Drying/Coalescence Times

Conditions	Touch Dry	Time to Coalescence
20°C 60% RH	10 minutes	2 hours
10°C 80% RH	2 hours	4 hours

In another practical trial when the air temperature was 15°C and the relative humidity approximately 50%, a three coat 'Haloflex' paint system was successfully applied within one working day.

Drying times are, of course, dependent on film thickness and, to some extent, on formulation. These paints are normally formulated to 40-45% volume solids and are best applied to a maximum one coat dry film thickness of about 80 microns. At higher dry film thickness, particularly under slow drying conditions, e.g. low temperature, high humidity and poor air flow, then the phenomena of flash rusting may occur with primer coats. This is minimised if the applied paint reaches the touch dry stage within one hour.

A frequent criticism of water borne paints is the narrow painting window for their use. Practical experience of these



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new paints has demonstrated that there is more latitude than is commonly envisaged. Obviously the coatings should not be applied to steel at temperatures below the minimum film forming temperature of the paint. Normally the low temperature application limit is of the order of 5°C. The coatings will withstand rain, some 30-45 minutes after application.

3. Good adhesion to steel and aged paints

Suitably formulated vinyl/acrylic paints adhere well to blast cleaned steel, galvanized steel and burnished aluminium as well as to concrete, asbestos cement, wood and many plastic surfaces, making the polymer an ideal candidate for an all purpose primer. They are quite tolerant to poorer surface preparation, e.g. blast cleaning to Sa2 and compatible with modern wet blasting procedures.

They can be applied over most types of shop primer and show good intercoat adhesion allowing trouble free overcoating. The primers can be used in systems with other water or solvent based paints including acrylics, chlorinated rubbers, vinyls, alkyds and epoxies. In maintenance situations where the primer may be required to adhere to aged paints it is advisable to carry out a patch test. Experience has shown there should be no problem over aged alkyds, chlorinated rubber, vinyls or epoxies.

4. Good mechanical and chemical resistance

These coatings are tough, flexible and remarkably impact resistant. They have proved particularly suitable for objects or environments where there is a risk of mechanical damage, e.g. containers and factory applied coatings. Steel can be primed offsite with the knowledge that damage to the paint film will be minimal.

Frequently in industrial maintenance situations a degree of chemical resistance is required. The coatings will resist splash and spillage of many corrosive chemicals including inorganic acids and alkalis. They will also resist gasoline, mineral and diesel oils and aliphatic solvents and alcohols. Prolonged contact with some chemicals may soften the paint which normally recovers on drying.

The coatings can be removed with aromatic solvents, chlorinated solvents or ketones.

5. Safety in use

A particular benefit of water borne coatings; replacing solvent with water increases worker safety, simplifies equipment clean-up and eases compliance with legislation on air and water pollution.

All latex paints offer reduced fire and explosion hazards but additionally 'Haloflex' based paints, having a high chlorine content, will not support combustion.

Maintenance painting

1. Rust converters

In common with all paint systems, 'Haloflex' based coatings perform best on blast cleaned steel, but they do have an advantage over competitive systems that they are more tolerant to less stringently blasted surfaces because of their acid nature.

'Haloflex' based coatings are making their mark in the rust converter markets. Rust converters are usually based on tannic acid which forms a black chelate compound with iron oxide. The reaction takes place under acid conditions and, being acidic, 'Haloflex' latices are an ideal binder for such coatings. Conversion of the rust is combined with the formation of a barrier film which gives some protection from further corrosion and provides a novel treatment for rusty surfaces. Inclusion of low oil absorption extenders further enhances the initial adhesion and improves performance in accelerated tests. These should be selected with care and be free from iron and carbonate. Acid washed barytes is particularly effective.

These vinyl acrylic/tannic acid coatings are self priming but for long term protection it is advisable to overcoat with a suitable solvent or water based topcoat. Surfaces are normally wire-brushed and preferably water washed before treatment and a common system used is one brush coat of rust converter followed by one or two coats of finish paint.

2. 'Haloflex'/Tar emulsion blends

Another approach being studied at present is the development of maintenance primers where the binder is a blend of 'Haloflex' and an aromatic petrol tar emulsion. Initial problems of stability have now been overcome by judicious choice of type and level of surfactant and surprisingly good results have been obtained on wire-brushed steel.

Early formulations evaluated included primers of 20 and 30% PVC extended with talc, pigmented with 2% volume concentration of red iron oxide and containing 3% volume concentration of Zinc Phosphate.

'Haloflex' bitumen emulsion blends have also been studied for this application with disappointing results. However, they do offer possibilities in roof coatings and fence coatings.

Finish paints based on 'Haloflex' 208, 'Haloflex' DP 251

'Haloflex' based paints can be overcoated with other water and solvent based paints including alkyds, chlorinated rubber epoxies and vinyls. Despite this however, there has been a need to develop topcoat polymers in the 'Haloflex' family.

'Haloflex' 208 is the current polymer recommendation. 'Haloflex' 208 is also a vinyl chloride/vinylidene chloride/acrylic copolymer, but in contrast to 'Haloflex' 202, the vinylidene chloride has been reduced to enhance light stability. Thermal stability has also been improved. Paints based on 'Haloflex' 208 have performed well in both accelerated tests and under actual service conditions. One of the best examples of the performance of the 'Haloflex' 202/'Haloflex' 208 system is evidenced on a methylene chloride stock tank in our Castner Kellner Works, alongside the River Mersey. Painted in July 1982, it is still in excellent condition after four years with no signs of film breakdown.

'Haloflex' 208, however, is a rather soft polymer with a slight residual tack which could lead to blocking problems under certain service conditions. This was taken into account in developing a new polymer, currently known as 'Haloflex' DP 251 which has been specially designed for use

over 'Haloflex' primed steel. The polymer has a low minimum film formation temperature, shows very little surface blocking, gives very good intercoat adhesion and of course performs well in accelerated and natural weathering tests.

Some of the key polymer characteristics are:

Type : vinyl acrylic
 Solids content : 54-56%
 MFFT : 10-13°C
 Particle charge : Anionic/Nonionic
 Chlorine content : 38% (on solid latex)

A typical starting formulation is given in Formulation 2, along with a summary in Table 2 of its performance upon physical and accelerated tests. Results with 'Haloflex' 208 and a couple of commercially available acrylic paints are included.

Formulation 2
'Haloflex' DP 251 experimental topcoat

Premix	% w/w
Water	5.50
'Foamaster' H2	0.2
'Synperonic' NP30 (20% w/w soln)	1.48
'Primal' RM8	0.57
'Texanol'	1.69
Bactromet	0.15
Orotan 850	0.31
Pigments	
Tioxide RCR2	18.51
Water	9.96
Let Down	
'Haloflex' DP 251N	61.62
PVC	15
% volume solids	40
Density Kg/l	1.3
Coverage m ² /l 50 µm DFT	8.0

Table 2

Physical tests

Paint	60 degrees	Blocking N/cm ² x 10 ⁻¹		Rocker Hardness
	Gloss	1 Day	1 Week	
'Haloflex' DP 251	64	36	13	10
'Haloflex' 208	81	51	41	6
Acrylic 1	71	not tested	12-15	41
Acrylic 2	86	20	2	30

Accelerated tests *500 HRS ASTM tests*

Paint	Humidity	Distilled Water	
		Soak	Salt Spray
'Haloflex' DP 251	9	7.5	8.5
'Haloflex' 208	6.5	6.5	5.0
Acrylic 1	10506	2.5	3.5

Substrate: Blast cleaned steel Sa 2.5, 75µm profile.

System : 2x'Haloflex'/Zinc Phosphate Primer : 80µm dry
 : 2x'Haloflex' DP 251 Top Coat : 80µm dry

Total DFT 160µm

In summary therefore the new polymer has the following attributes:

1. Low minimums film formation temperature.
2. Levels of coalescing solvents required will also be low.
3. Considerably lower tack than 'Haloflex' 208.
4. Good performance over 'Haloflex' based primers on blast cleaned steel.

The future

This paper has taken us in detail through the development of what we believe to be a unique series of water-based polymers designed for steel protection. Formulation into anti-corrosive paints has also been discussed, and the properties of these paints detailed.

Ideas for formulation of maintenance primers for wire brushed steel are also given.

A specially designed topcoat polymer has been introduced to the market and suggested starting formulae and results are included.

This is by no means the end of this story and further grades are under development. These include:

'Haloflex' DP 260

Specially designed for primers where thermal stability may be a prime requirement. The composition has been optimised to give the best balance of

- thermal stability especially in the presence of zinc phosphate.
- better barrier properties because of its high chlorine content.
- maintain the low film formation temperatures.

Outlets envisaged for this polymer include primers for steel and galvanized steel, particularly at higher working temperatures, coatings for concrete where good carbon dioxide barrier properties and stability on alkaline substrates are important requirements.

'Haloflex' DP 402

Polymer designed for clear barrier films with low blocking characteristics. 402 is in compliance with the US Food and Drug Administration's Regulation 21 CFR 176/170 (direct contact with aqueous and fatty foods) and is ideally suited for the fibreboard coating market.

'Haloflex' 402 provides a much better water barrier (under both vapour and ponding conditions) to acrylic/wax coatings and offers further advantages in terms of glueability and overprinting with aqueous based inks.

'Haloflex' 312

'Haloflex' 312 combines an unusual combination of high molecular weight and a low glass transition temperature (-12°C) making it ideal for highly pigmented roof membranes which are applied at dft's around 400 microns per coat.

A typical formulation pigmented to a total PVC of 40% has the following properties:

- extensibility of the detailed film 50°C.
- flexibility down to -15°C.
- can be rendered completely inflammable by addition of 10% antimony oxide.
- good adhesion to felt/bituminous surfaces.

Conclusion

It is believed that this expanding range of chlorine

Heat-cured ionomers prepared from poly(acrylic acid): A new class of waterborne coating material

J. W. Nicholson, R. P. Scott and A. D. Wilson

Materials Development Sub-division, Laboratory of the Government Chemist, Department of Trade and Industry, Cornwall House, Waterloo Road, London SE1 8XY, UK

Summary

The chemistry and technology of a new series of water-borne coatings are described. These coatings, which have no volatile organic component in them, are based on poly(acrylic acid) which has been partially neutralized with sodium or potassium ions. The cured coatings belong to the class of materials known as "Ionomers", i.e. polymers containing a minority of ionized functional groups neutralized by a few per cent of ions. A detailed study of the crosslinking of these coatings using reflectance infrared spectroscopy, is reported, and shows that they cure by formation of anhydride groups, followed by rapid decarboxylation. These coatings, which were found to adhere well to metallic substrates, have been evaluated as possible clear lacquers for the interior of food and beverage cans.

Introduction

Water-borne coatings are currently the subject of great interest, and there have been many new developments in recent years^{1, 2}. The reason for this interest has been the desire to restrict the use of organic solvents for a mixture of environmental and economic reasons. The environmental concern has mainly been expressed in the United States, where there is now legislation governing the atmospheric release of photochemically-active organic solvents, particularly olefins, aromatics and branched-chain ketones. As well as reduced atmospheric pollution, water-borne coatings have other environmental advantages. In particular, for personnel working in paint application, there is less exposure to potentially harmful organic vapours², as well as less risk of fire³. Moreover, waste disposal from water-borne paint spray lines is easier than disposal from lines using solvent-based coatings⁴. Economically, too, water-borne coatings are attractive. Water is the cheapest solvent available⁵, and organic solvents, based on crude oil, are becoming increasingly expensive^{2, 6}.

In the current paper a new class of water-borne coatings is reported, which are based on poly(acrylic acid), and as a result, are completely water-soluble as formulated. They cure at elevated temperatures to yield ionomer coatings having excellent flexibility, adhesion to metal

containing vinyl acrylic copolymers will help to answer many of the formulation problems associated with moving no water borne high performance coatings, both for painting of structural steel and for industrial finishes.

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substrates, and good resistance to a wide range of chemicals.

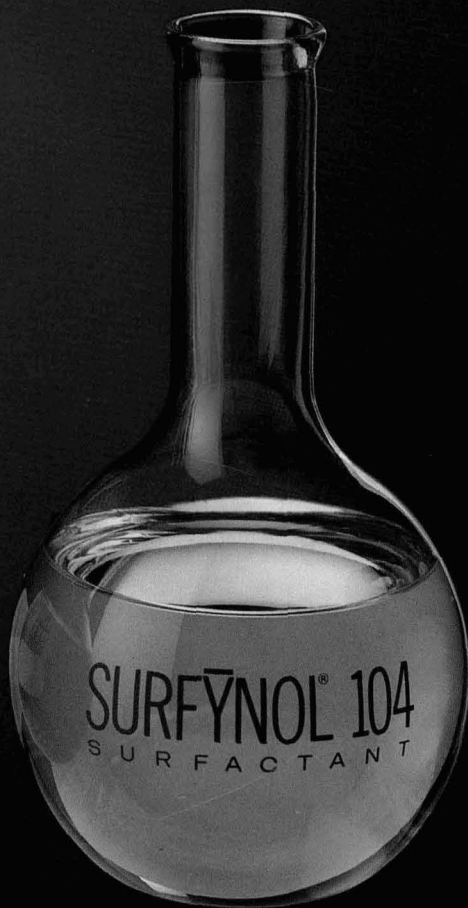
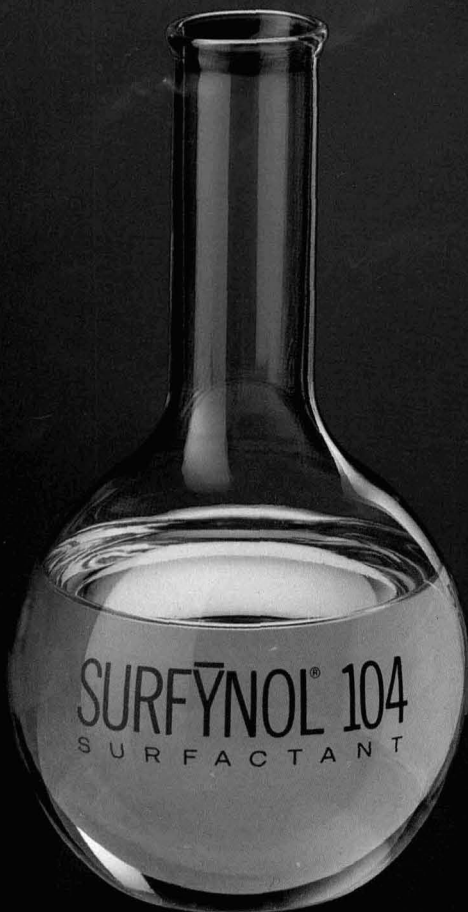
The term "ionomer" for these coatings needs explaining. It was originally coined by the Du Pont company in America in the mid-1960's for their range of hydrocarbon and fluorocarbon polymers⁷. These polymers included a minor proportion of an acid function which was neutralized to varying degrees by a metal or quaternary ammonium ion, the mole percentage of metal ion not exceeding 20, and generally lying in the range 5-10%. In this paper the term "ionomer" has been used for these coatings because, on curing, they become similar to conventional ionomers, in that they contain a minor proportion of ions embedded in a substantially organic matrix.

In the present work, these ionomer coatings have been examined as possible lacquers for coating the interior of metal cans, since this is an area with well defined requirements. In particular, for this purpose, coatings should be thin (2-5 μm), capable of application by rollercoating or spraying, have good adhesion to tinplate, tin-free steel or aluminium, and cure in 8-10 minutes at about 200°C⁸. In addition, they should be resistant to a variety of chemicals and processing conditions, impart no flavour to the contents, and show minimal leaching of organics, none of which may be toxic.

Tests have not yet been carried out on the present ionomer coatings to find out whether they conform to these requirements. However, since the basic materials are widely used in dentistry as filling materials^{9, 10}, where they need to be highly resistant to foodstuffs, and also non-toxic, there seems every prospect of them proving acceptable.

Experimental

1. *Materials*: Poly(acrylic acid), "Versicol E7" (ex. Allied Colloids), molar mass 30,000 was used. It is supplied as a 25% solution, and formulations were prepared from this by diluting to 10% or 15% solids. The acid was partly neutralized (up to 20%) by reaction with sodium or



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potassium hydroxide (both "AnalaR" reagent grade, ex. BDH Ltd). Small amounts (less than 1%) of Triton X-114 (ex. BDH Ltd), a non-ionic surfactant were incorporated into the formulations to improve substrate wetting.

2. *Application and cure*: Coatings were applied by aerosol spray to degreased panels (dimensions 100 mm × 150 mm), after which cure was brought about in a forced air oven at 200 or 250°C.

3. *Test procedures*: The following tests were carried out, the British Standard number of the method is shown where appropriate:

(a) Bend test round a 3 mm mandrel (BS3900, Part E1: 1970).

(b) Cross-cut adhesion, results of which are expressed on a 6-point scale running from 0 (the best) to 5 (the worst), as defined in BS3900, Part E2.

(c) Exposure to the following, for the times and temperatures specified:

(1) Distilled water at room temperature for one hour

(2) 3% NaCl solution at room temperature for 24 hours

(3) 5% acetic acid solution, boiling, for one hour

Results of these tests were expressed on a 5-point scale, running from 0 (the best) to 4 (the worst) defined as follows:

0 = film unaffected

1 = slight misting/loss of gloss

2 = substantial loss of gloss and/or slight swelling

3 = substantial swelling and/or partial film loss

4 = complete loss of film from substrate

(d) Dry film thicknesses were measured using the appropriate Digital Coatings Thickness Gauge, i.e. for either ferrous or non-ferrous substrates, Type SS1200 or SS600 respectively, (ex. Sheen Instruments Ltd). In addition, coating weights were assessed, simply by weighing panels before film application and after curing.

Results and discussion

In this study an attempt was made to use a curing regime similar to that employed in current can-coating technology (typically 10 minutes at 200°C⁸). However, we found that within this limited time of cure, the temperature had to be increased to 250°C in order to produce ionomer coatings with optimum properties. Surprisingly, when tinplate was used as the substrate no flow of tin was observed although the temperature of cure was 18°C above the melting point of tin. This behaviour was attributed to the thinness of the tin layer on modern tinplate¹¹ combined with the fact that much of the tin will be locked up as the alloy FeSn₂¹². Whatever the explanation, the fact was that there was no flow on heating at 250°C, and the ionomer coatings on tinplate cured as well as on any other metallic substrate. Finished coatings were smooth, glossy and either clear or of a pale brown hue.

Infra red spectroscopic study of cure mechanism

The cure reactions were followed by reflectance infra red spectroscopy. Samples were prepared on polished tinplate by heating thin films (approx. 5 μm) at 250°C for various times, after which the reflectance infra red spectra were recorded. There were three absorption bands of interest. A pair at 1,795 cm⁻¹ and at about 1,750 cm⁻¹, are both assigned to anhydride groups, the former being the more distinctive¹³. The latter band was not at all distinct, since it forms part of a broad group of unresolved carbonyl bands in these spectra. The third band of interest, which occurs at about 1,550 cm⁻¹ is assigned to the neutralized carboxylate groups in the film¹³.

Elsewhere¹⁴, two possible cure mechanisms for ionomer coatings have been identified, the exact mechanism depending on the nature of the metal ion. In the present paper, the discussion is limited to the cure reactions of systems partially neutralized with sodium or potassium ions, since these gave coatings with the level of chemical resistance needed in practical applications.

Poly(acrylic acid) itself is known to convert to insoluble films on heating, and this process has formed the basis of a coating proposed for use on tinplate¹⁵. The chemistry of this conversion has been studied by a number of workers¹⁶⁻²⁰, all of whom found that the major reaction that occurs on heating is the formation of anhydrides. However, there were conflicting views on the nature of the anhydrides formed. These differing views were resolved by Eisenberg et al¹⁹, who showed that the structure adopted depended on the temperature, higher temperatures (i.e. above 200°C) favouring the formation of intermolecular anhydride bridges, while lower temperatures (about 150°C) favoured intramolecular anhydride cyclization.

As part of the present study, infra red spectra were recorded of films of poly(acrylic acid) cured at 250°C, a typical example being shown in Figure 1. The presence of anhydride groups is evident from the prominent band at 1,795 cm⁻¹.

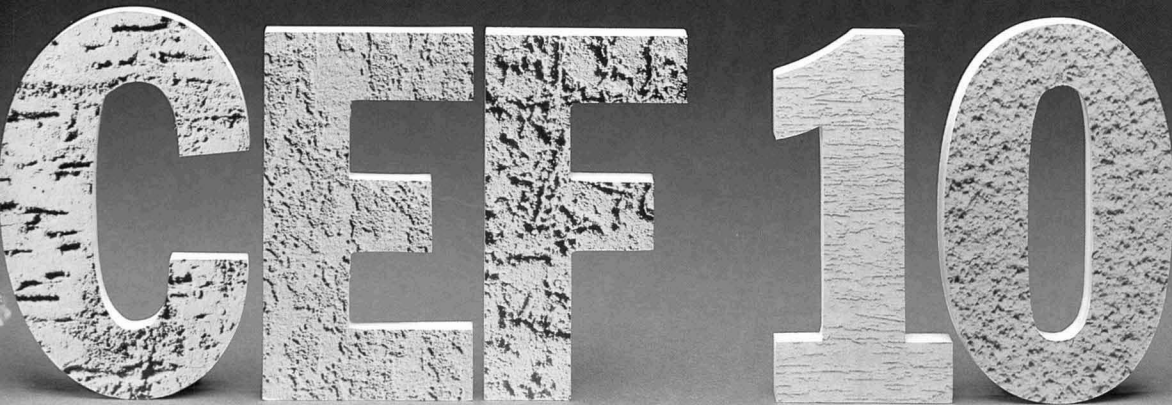
This result confirms the findings of previous workers. However, when the cure of partially neutralized poly(acrylic acid) was studied, additional curing reactions were revealed.

Figure 2 shows spectra of two sodium-containing films (10% neutralized), with cure times of two minutes and 10 minutes respectively at 250°C. It can be seen that the prominent anhydride peak visible at t = 2 minutes has been substantially reduced by t = 10 minutes. In order to follow the course of this change, a series of spectra were recorded, and the ratio of the anhydride band (ca. 1795 cm⁻¹) to carboxylate band (ca. 1550 cm⁻¹) was plotted against time of heating. The carboxylate band was chosen as the base adsorption because, not only is it distinct, but also, since the cations are not volatilized, from considerations of electroneutrality, its content cannot change. A similar series of spectra were also recorded for poly(acrylic acid) 10% neutralized with potassium hydroxide. The results obtained for both series of spectra are shown in Figures 3 and 4, where the ratio of anhydride to carboxylate peaks are plotted against time of heating. Both curves can be seen to be very similar.

In each case, there was a rapid build up to a maximum

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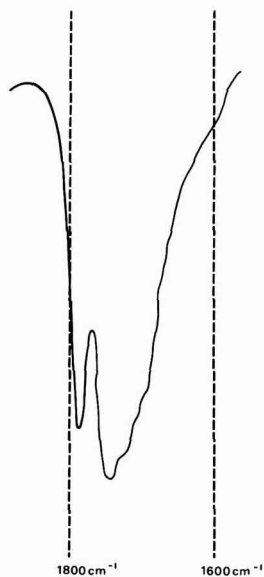


Figure 1. Infrared spectrum of poly(acrylic acid) heated at 250°C for ten minutes.

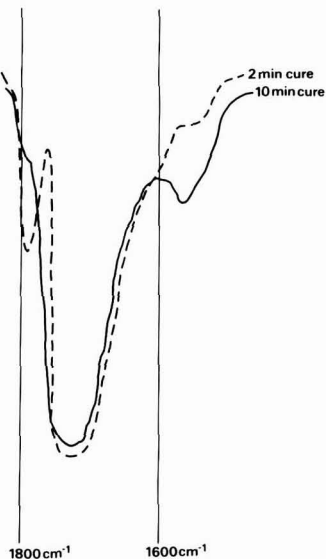


Figure 2. Infrared spectrum of 10% neutralized sodium poly(acrylate) heated at 250°C.

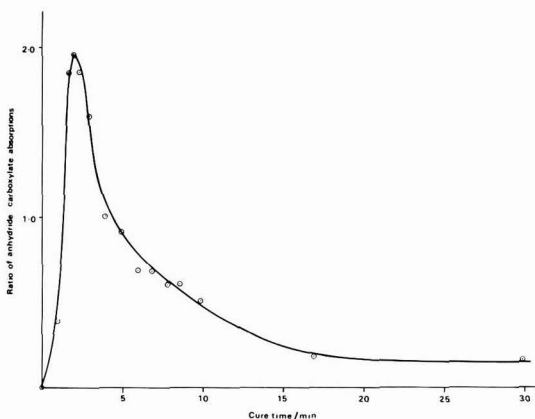


Figure 3. Variation in amount of anhydride with time of heating for 10% neutralized sodium poly(acrylate) at 250°C.

anhydride content at $t = 2-3$ minutes, followed by decay of anhydride to a final value of about 10-15% of the maximum. This decay shows that decarboxylation occurs; anhydrides are known to undergo thermal decarboxylation to ketones²¹, especially in the presence of nucleophiles²². In partially neutralized poly(acrylic acid), the nucleophilic component which catalyses this reaction is the carboxylate group.

The non-coordinating nature of the metal ions means that the actual site of any given carboxylate group may shift within the polymer chain as the reaction proceeds, and this almost certainly aids the reaction. It is significant that coatings which contain coordinating ions, such as Cu^{2+} or Co^{2+} , do not lose anhydride peaks on prolonged heating, and also do not develop particularly good chemical resistance¹⁴. This shows that the resistance to chemical

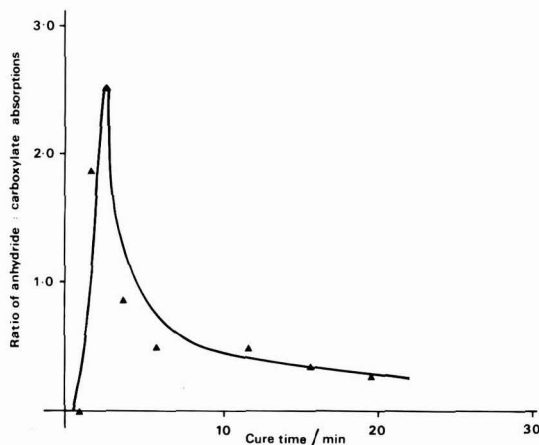
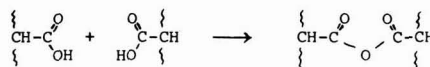


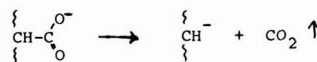
Figure 4. Variation in amount of anhydride with time of heating for 10% neutralized potassium poly(acrylate) at 250°C.

attack is improved by the conversion of anhydride groups, which are readily hydrolysed, to keto groups, which are hydrolytically stable.

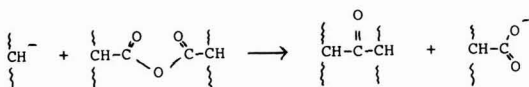
The overall cure process, then, may be summarised in the following sequence:



and:



then:



Performance of coatings

Test results for typical ionomer coatings on aluminium are given in Table 1.

Table 1

Properties of Ionomer coatings on aluminium (films approximately 5 µm thick)

Coating†	A	B	A	B
Cure temperature (°C)	200	200	250	250
Bend test	Pass	Pass	Pass	Pass
Cross cut adhesion	0	0	0	0
Distilled water, room temperature, one hour	3	4	0	0
NaCl (3%), room temperature, 24 hours	3	4	3	0
Acetic acid (5%), boiling, one hour	3	4	3	1

†A = poly(acrylic acid), B = 10% Na⁺ neutralized PAA

From Table 1, it can be seen that of the coatings cured at 200°C, the ion-free formulation, A, showed superior chemical resistance to its equivalent ionomer formulation, B. These results highlight an important feature of the ionomer system: partial neutralization, though essential for the decarboxylation step of the cross-linking, does increase the water solubility of the parent polymer, since the hydrophilic nature of some of the polymer segments is increased². This effect is well-known, and has been used to solubilize otherwise insoluble carboxylic acid polymers²³. It is therefore essential to allow the cure reactions, especially the irreversible decarboxylation step, to proceed as far as possible in order to insolubilize the films completely. The results in Table 1 demonstrate that this objective is achieved by curing at 250°C (10 minutes), since after such processing, the ion-containing coating, B, showed superior chemical resistance, to the unneutralized one, A.

Finally, in addition to the tests described above, rub tests were carried out using tissues soaked in the organic solvents methyl isobutyl ketone or acetone. In all cases, the ionomer films showed complete resistance to these solvents.

Degree of neutralization

In order to examine the effect of sodium ion content on the resulting coatings, a series of partly neutralized sodium poly(acrylates) was prepared, and films were prepared with curing at 250°C for 10 minutes. The dried coatings were subjected to the room temperature, distilled water test, and the results, which were assessed as before, are shown in Table 2.

It can be seen from Table 2 that as expected, solubility in water increased steadily as sodium content rose. The other feature of note, which was not necessarily expected in advance, was that at sodium levels above 20%, film formation was not satisfactory, showing that the ability to form films, as well as water resistance, is highly dependent on the degree of neutralization.

Table 2

Effect of altering sodium ion content in ionomer coatings

Extent of neutralization (%)	Appearance of film	Distilled water resistance
5	Pale bronze hue	0
10	Pale bronze hue	0
15	Almost colourless	2
20	Colourless	3
25	Badly reticulated	4
50	Does not form a film	0

Conclusion

The ionomer coatings described in this paper are readily prepared from inexpensive components. As formulated, they are completely water-soluble, and they are capable of undergoing irreversible cross-linking on stoving, the mechanism of this process having been shown in the current work to consist of anhydride formation followed by decarboxylation. The resulting films had good gloss and flexibility, and were resistant to various chemicals. They show some promise as interior lacquers for metal cans, and further developments in this and other applications may be anticipated.

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An evaluation of 'Silane' treated concrete

H. L. Robinson

Taywood Engineering Ltd, Design and Research Laboratory, 345 Ruislip Road, Southall, Middlesex UB1 2QX, UK

1. Introduction

The use of Silicone based water repellent treatments for concrete has resulted in recent widespread interest and concern within the construction industry, regarding the performance and specifications of these materials.

There exists a wide range of potentially different silicon based water repellent treatments and to a first approximation their respective performance depends on several synergistic factors:

1. The nature of the material i.e. whether it be the parent silane, partially hydrolysed silane/siloxane or silicon resin, is critical for optimum performance. Each type will eventually produce a hydrophobic product, but they are all at different stages of conversion (Hydrolysis).
2. The nature of the alkyl group on the silicon based material, whether it be the relatively small methyl or larger iso-Octyl, has a dramatic effect upon the materials volatility, hence evaporation rate which also greatly affects performance. Generally the larger the alkyl group the lower the volatility¹.
3. By adding a catalyst, usually based on Titanium or Tin, the reactivity of these materials can be controlled.
4. Dilution by different solvents, usually ethanol or white spirit, can also affect performance¹, especially the degree of dilution.
5. The chemical characteristics of the mineral substrate onto which these materials are applied, are also critical to their performance¹⁻³.

Other important factors affecting performance include: water content in the pores of the substrate i.e. too much water will restrict penetration. Environmental conditions at time of application can also prove to be critical. Ideal conditions would include a relatively high humidity i.e. 80% RH, relatively low ambient temperature i.e. 10°C and preferably calm conditions i.e. no wind to reduce loss of material due to evaporation.

These materials are generally applied by spray on vertical surfaces and ponded on horizontal surfaces, i.e. bridge decks.

A great deal of research into the performance of these materials has been carried out in West Germany namely by: Wacker Chemie GmbH¹, Th. Goldschmidt Ltd², Dynamit Nobel Ltd³, and in Hungary by Seidl et al⁵. Such materials have been in use in West Germany for up to 17 years and case histories proving their effectiveness at reducing water/chloride ingress and freeze thaw action can be cited.

Taywood Engineering Ltd., acting as an independent test house, is currently engaged in a research programme designed to evaluate the performance of protective surface treatments for concrete including silicon based treatments. Tests include: Chloride diffusion, carbon dioxide diffusion, accelerated carbonation testing, penetration depth, effect on substrate pore structure, effect on moisture loss from

concrete and evaluating the effect of varying the pH of simulated pore water upon the rate of conversion of the water repellent material to the silicon resin product.

For the purpose of this study four different water repellent materials were evaluated:

- System I Methyl Oligomeric Siloxane + Tin Catalyst, diluted 1:11 with white spirit.
- System II 80% iso-Octyl Oligomeric Siloxane + 20% Methyl Methoxy Silane, diluted 1:11 with white spirit.
- System III 75% iso-Octyl Tri-methoxy monomeric silane + 25% iso-Octyl oligomeric siloxane, diluted 1:4 with white spirit.
- System IV iso-Butyl Tri-Methoxy monomeric silane.

2. Experimental

2(a) Standard concrete

For the purpose of the aforementioned tests, two different concrete mixes were designed:

		Kg/M ³
Mix A:	Cement	200
	20ml Gravel	840
	10ml Gravel	420
	M-Grade sand	775
	Water	150
	Density	2385
W/C =		0.75

The above mix design was used to cast standard concrete cubes, 100mm x 100mm x 100mm, for accelerated carbonation testing. The concrete cubes were cured for at least 28 days in a polythene bag and conditioned at 60%RH, 24°C to constant weight before treating with water repellent.

		Kg/M ³
Mix B:	Cement	311
	10ml Gravel	1030
	M-Grade sand	874
	Water	212
	Density	2340
W/C =		0.68

Cores were taken from the above concrete mix and used for chloride and CO₂/O₂ diffusion testing. The concrete cores were cured for 28 days in a polythene bag followed by conditioning at 80% RH, 24°C for two weeks prior to treating with water repellent.

2(b) Chloride diffusion

Figure 1 shows the diffusion cell apparatus designed to measure ionic diffusion coefficients through treated

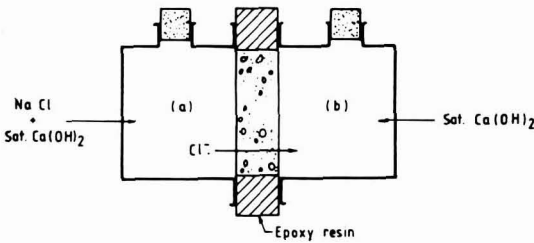


Figure 1. Diffusion cell.

concrete specimens. The sample is a concrete slice, 20mm deep, 100mm in diameter. The slice is cut to a uniform thickness, as thin as possible, allowing for aggregate size, to reduce test time. The water repellents were applied by a dip method onto the top face of each core and conditioned at 80% RH, 24°C for two weeks prior to testing.

The test slice was cast with an epoxy resin ring around the outside and the finished test piece assembled as shown. The apparatus was tested for leaks with distilled water. The test solutions were poured into each compartment and the date and time noted, t_0 .

The time intervals between chloride testing depend on the rate of diffusion through the slice. The first analysis was made after 2-3 days by withdrawing a 10ml aliquot from compartment (b) and the volume made up with saturated lime solution. The diffused chloride concentration was plotted against time. Sufficient measurements were made to ensure that steady state diffusion had been achieved. From the steady state flux of chloride ions through the treated concrete, an effective diffusivity was calculated, using Fick's Law.

Diffusion coefficients were compared against simultaneous measurements on an untreated control specimen. Normally results are expected in 70-90 days, depending on the materials permeability.

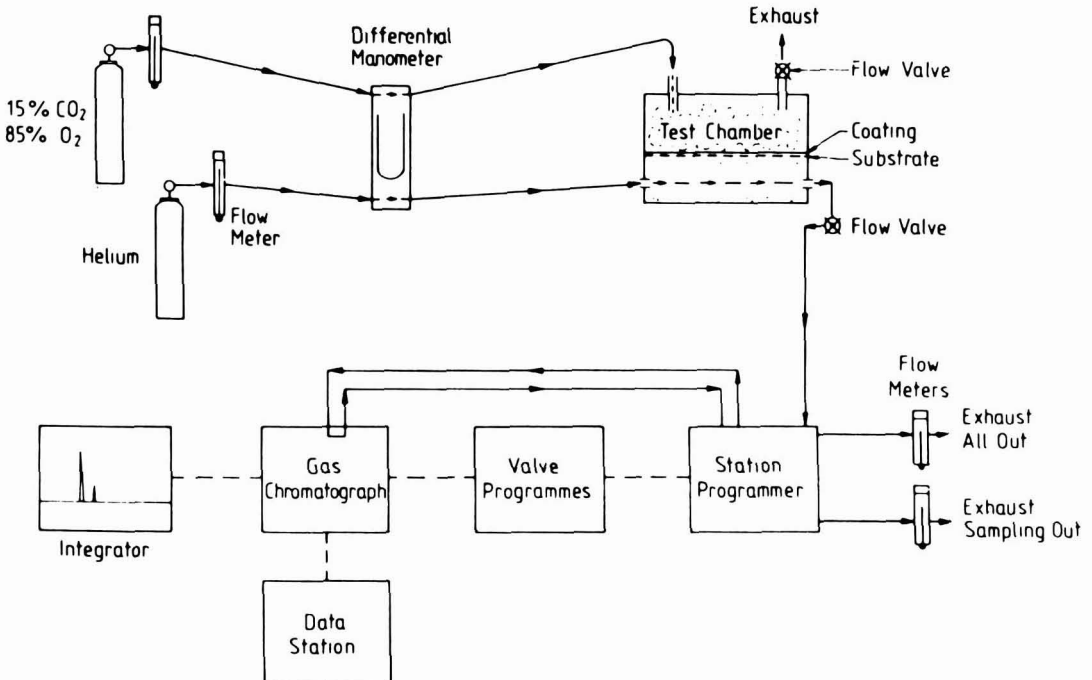
2(c) Carbon dioxide diffusion

The purpose of this test is to assess whether silicon based water repellent treatments affect the CO_2 diffusion rate through treated concrete.

Figure 2 shows the test apparatus, developed to enable the gas barrier properties of both Organic and Inorganic coatings to be measured⁶. The silicon based materials were applied to concrete cores, 20mm x 100mm, by a dip method and sealed around their edge with epoxy. The treated cores were conditioned separately to prevent cross contamination, at 80% RH, 24°C for four weeks, followed by conditioning at 0% RH, 24°C for a further four weeks prior to testing. Conditioning at 0% RH reduces the moisture present in the concrete cores in order to minimise erroneous results since gas diffusion through concrete is affected by the moisture level in the concrete. Also by lowering the relative humidity within the concrete, the risk of carbon dioxide reacting with $\text{Ca}(\text{OH})_2$ i.e. carbonation occurring, is reduced since carbonation occurs at an enhanced rate within the humidity range 50-75% RH at 24°C. If the CO_2 did react with $\text{Ca}(\text{OH})_2$ present in the cores, this would also produce erroneous results.

After conditioning, the cores were sealed in a circular steel rig with the treated and untreated faces exposed. Carbon dioxide (100%) at a known pressure and flow rate was passed over the treated face of the core and helium gas passed over the untreated face at the same pressure and

Figure 2. Gas chromatography apparatus.



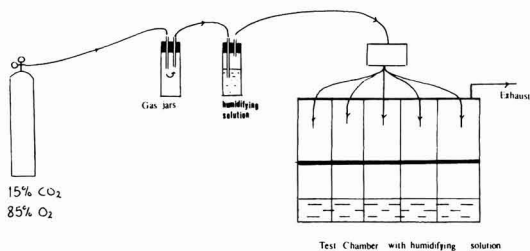


Figure 3. Accelerated carbonation test.

flow rate. The helium gas stream was continuously monitored by gas chromatography and analysed for carbon dioxide. Equilibrium conditions were achieved after approximately 24 hours, upon which the steady state flow of carbon dioxide was calculated from the percentage of carbon dioxide detected in the helium stream and flow rate of the gas.

The diffusion coefficient (DL) for carbon dioxide was calculated using Fick's 1st Law of diffusion⁶. The diffusion resistance coefficient (μ) was calculated accordingly, by dividing the diffusion coefficient for CO₂ in air at 25°C by DL.

2(d) Accelerated carbonation testing

Figure 3, shows the experimental apparatus designed for evaluating the effect of silicon based water repellent treatments on the rate of carbonation through treated concrete.

Standard concrete cubes, 100mm x 100mm x 100mm were sealed on five faces with epoxy and overcoated with a chlorinated rubber paint leaving the top face exposed. Each of the respective water repellent treatments were applied to the top face of three cubes by a dip method, making a total of 12 treated cubes. Three untreated cubes were evaluated simultaneously as control specimens.

The cubes were conditioned in separate chambers to prevent cross-contamination from solvent vapours, at 60% RH, 24°C for three weeks before being subjected to an atmosphere of 15% CO₂/85% O₂.

At periodic intervals one of each treated cube and one control cube was removed from the tank. The approximate depth of carbonation was measured by the standard phenolphthalein test and in the case of System IV determined accurately by derivative thermogravimetry (DTG).

2(e) Influence of pH on water repellent performance

The effectiveness of a water repellent treatment applied to concrete, depends to a first approximation on the rate of conversion (hydrolysis) of the silicon based material into the hydrophobic resin by reaction with the pore walls. The pH of the pore water contributes to the catalysis of this reaction. The higher the pH i.e. 12.5 to 10, the faster the conversion rate. In practice, for optimum performance, the water repellent must react as quickly as possible with the concrete pore walls to offset loss of material due to evaporation. The pH of carbonated concrete is approximately 8.5 and in practice it is this level of alkalinity on which one must depend, for an adequate conversion rate of

the applied treatment.

A simple bench top analysis was conducted on two of the aforementioned repellents, to evaluate the effect of pH on their respective performances. The two materials evaluated were systems III and IV. Both materials were tested at 100% concentration and were not diluted further by solvent.

For the purpose of this test, a standard simulated pore water solution was made up consisting of NaHCO₃/NaOH/H₂O, at pH = 12.5. Both materials were mixed individually with the pore water at four different pH values (12.5, 10.5, 8.5 and neutral 6) in the ratio 1:1 by volume. Nitric acid was used to control the pH.

Visual analysis was used to determine qualitatively the effect, if any, of the level of pH upon the rate of conversion (hydrolysis) of the water repellent material into the hydrophobic resin.

2(f) Depth of penetration

The depth of penetration of the water repellents into the standard concrete, referring to 2(a), was determined simply by breaking open the cubes used in test 2(d) and wetting the concrete cross-section with water. The depth of penetration was defined by a dry zone close to the surface of the treated face.

2(g) Effect on substrate pore structure

A porosimetry technique was used to evaluate the extent to which silicon based water repellent treatments can modify a pore structure compared to an unpigmented acrylic impregnation treatment.

The substrate used in this study was an unglazed porous ceramic tile which provides a reasonably uniform pore size distribution between 10³-10⁴ angstroms. The tile was soaked in alkali solution for two days and allowed to dry out to constant weight at 60% RH, 24°C prior to treating with the two materials under test.

The acrylic resin used was based on a methyl methacrylate/siloxane blend of very low viscosity and compared against system IV.

2(h) Effect on moisture loss from concrete

Two standard concrete cubes (100mm x 100mm x 100mm³) were submerged under water for 48 hours, then allowed to stand for four hours at 24°C, 50% RH until surface dry. One of the cubes was then treated on all six faces by System IV using a dip technique. Both the treated and untreated cubes were subsequently allowed to dry out in an atmosphere of 50% RH, 24°C. The relative rates of moisture loss were determined gravimetrically.

3. Results/discussion

3(a) Carbon dioxide diffusion

Results in Table Z indicate that systems I, II, and III have a significant effect on the rate of CO₂ diffusion through the treated concrete cores. These materials reduce the passage of CO₂ by approximately two orders of magnitude compared to the control concrete. These materials do not however compare with organic polymer

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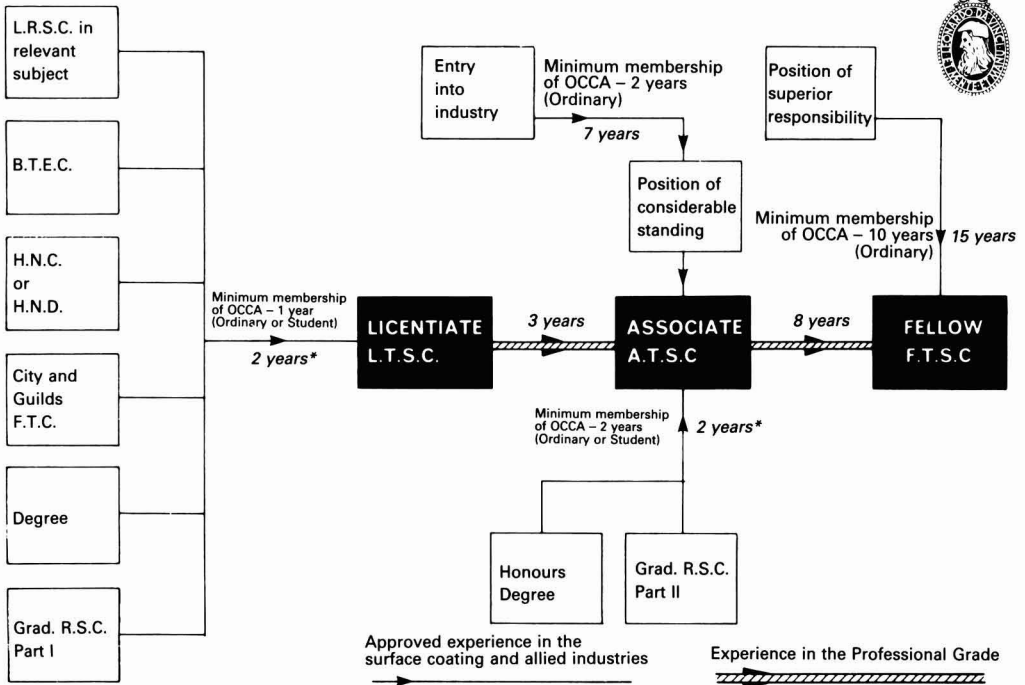
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Table Z

System	D cm ² s ⁻¹	Diffusion resistance Coefficient (μ)
Control concrete	5.3 x 10 ⁻⁴	281
I - Methyl Siloxane	2.2 x 10 ⁻⁶	67,727
II - iso Octyl Siloxane	7.9 x 10 ⁻⁶	18,861
III - iso Octyl Silane	1.03 x 10 ⁻⁵	14,466
IV - iso Butyl Silane	4.88 x 10 ⁻⁴	305

coatings⁶ in terms of CO₂ resistance, the latter generally offering diffusion coefficients of the order 1 x 10⁻⁷ cm²s⁻¹. System IV has no significant effect on the diffusion of CO₂ through treated concrete compared to the untreated control.

The order for these systems in terms of CO₂ diffusion resistance is: I > II > III > IV ≈ Control. This can be explained in view of the fact that systems I and II are basically oligomeric siloxanes, Whereas III and IV are primarily monomeric silanes. Siloxanes have a greater pore blocking effect compared to silanes which only line capillary pores in the concrete. Siloxanes should not, however, be regarded as pore blockers in the same context as polymer organic coatings which form continuous films. Silicon based water repellents do not form continuous films.

Although they are both essentially monomeric silanes, system III has a greater resistance to CO₂ than IV. This can be explained by the fact that system III contains 25% iso-Octyl Oligomeric Siloxane which probably has a minor pore-blocking effect.

3(b) Chloride ion diffusion (Table A)

μ_c = Diffusion Resistance Coefficient for Composite System (i.e. Concrete + Treatment) relative to control standard concrete.

$$\text{where } \mu_c = \frac{D \text{ concrete}}{DT}$$

D concrete = Diffusion Coefficient for Chloride ions through concrete (cm²s⁻¹).

DT = Diffusion Coefficient for Chloride ions through treated concrete (cm²s⁻¹).

Figure 4 indicates the relative performances of the four systems as barriers against chloride diffusion.

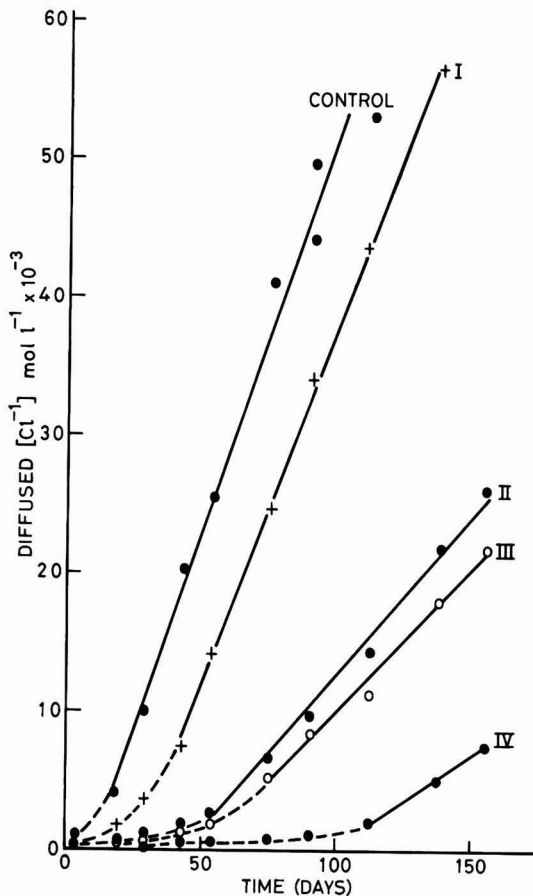


Figure 4. Chloride diffusion of Systems 1-4 and control.

System I appears to have only a delaying effect before the onset of Steady State diffusion after 30 days, at which point the chloride ions diffuse through the standard concrete at approximately the same rate as the untreated control concrete. This result is perhaps not surprising in view of the fact that this system is based on Methyl Oligomeric Siloxane which is known to be susceptible to alkali degradation.

Systems II and III delay the onset of Steady State

Table A

System	Estimated Penetration Depth (μm)	Composite Thickness (cm)	Approximate Time (days) to onset of steady state Diffusion	Composite System	
				D cm ² s ⁻¹	μ _c
Control Concrete		2	10	1.54 x 10 ⁻⁸	—
I — Methyl Siloxane	1,000	2	30	1.2 x 10 ⁻⁸	1.28
II — iso Octyl Siloxane	1,000	2	65	6.16 x 10 ⁻⁹	2.5
III — iso Octyl Silane	1,000	2	74	6.16 x 10 ⁻⁹	2.5
IV — iso Butyl Silane	2,500	2	112	2.85 x 10 ⁻⁹	5.4

diffusion by approximately 65 and 74 days respectively, although thereafter they do appear to retard the diffusion rate rather than only delaying the time taken to achieve steady state diffusion.

System IV performed by far the best, resisting chloride penetration completely for 112 days. Thereafter this system still significantly retarded the rate of chloride ion diffusion through the concrete.

By estimating the penetration depth of each treatment into the concrete, and assuming these depths to be separate individual layers in the same way a polymer coating would be treated, it is possible to determine an approximate diffusion coefficient for each Silicon based treatment, separate from the composite system. This is an assumption but at least it gives a useful indication of the materials performance. Then using the diffusion coefficients for the actual treatments it is possible to calculate an equivalent thickness of concrete, Sc . This term relates the performance of each treatment to an equivalent thickness of concrete in terms of added protection against chloride ion penetration.

System	DT cm^2S^{-1}	μ_w	Sc(cm)
I — Methyl Siloxane	2.72×10^{-9}	5.7	0.57
II — iso Octyl Siloxane	5.13×10^{-10}	30.02	3.0
III — iso Octyl Silane	5.13×10^{-10}	30.02	3.0
IV — iso Butyl Silane	4.37×10^{-10}	35.24	8.81

where: DT = Diffusion Coefficient for chloride ions through the water repellent material.

where: μ_w = Diffusion Resistance Coefficient for the water repellent relative to concrete.

where: Sc = Equivalent thickness of concrete (cm).

$$\text{where: } \mu_w = \frac{D \text{ concrete}}{DT}$$

where: Sc = $\mu_w \cdot S$.

A typical concrete as used in building construction would generally have a chloride ion diffusion coefficient of the order $10^{-9} \text{cm}^2\text{S}^{-1}$. Hence results indicate that System II, III and IV would all offer protection against chloride ingress, although System IV would provide the best long term protection.

3(c) Accelerated carbonation testing

After removing one of each of the treated cubes and one control cube from the 15% CO_2 atmosphere, the specimens were then sliced into two halves. One half was sprayed with phenolphthalein to determine an approximate carbonation depth. The carbonated zone is indicated by a characteristic colour change i.e. pink colouration in the uncarbonated concrete region, differentiating between the carbonated concrete (no colour change). A carbonation depth of 15-17mm was measured in each case using this method of analysis.

No significant difference was observed between the

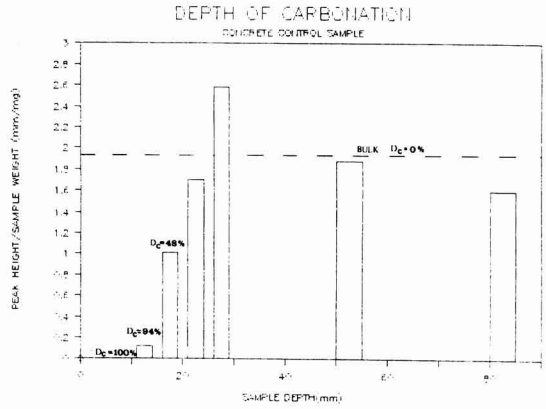


Figure 5. Depth of carbonation of control concrete.

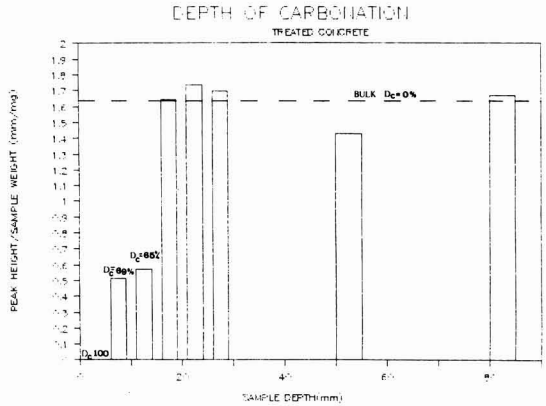


Figure 6. Depth of carbonation of concrete treated with System IV.

treated and untreated concrete.

The concrete treated with System IV was also assessed for carbonation depth and degree of carbonation using derivative thermogravimetry (DTG). Having sliced the treated cube in half, one half was sliced at 5mm intervals and from each slice the degree of carbonation was calculated from the calcium hydroxide decomposition peak at approximately 500°C.

The degree of carbonation, (D_c), was assessed as a percentage of the amount of calcium hydroxide present in each slice relative to an average bulk level, (Table B). An effective carbonation depth, ($D_c \geq 90\%$), for the control concrete, (Figure 5), would be approximately 14mm; this compares with that of 15mm measured by the phenolphthalein test. Similarly for the concrete treated with System IV, an effective carbonation depth of 14mm was determined, (Figure 6), although the degree of carbonation in the range 0-14mm is less relative to the control concrete.

3(d) Depth of penetration (Table C)

The monomeric silane, System IV, penetrates concrete to a greater degree than the other systems. This effect can be related to molecular size, the relatively smaller silane monomers have a lower relative viscosity and hence can penetrate more freely compared with the other materials.

Table B

(a) Control concrete

Sample Depth (mm)	Ca(OH) ₂ Peak Height/ Sample Weight (mm/mg)	D _c Degree of Carbonation
0 - 4	—	100
6 - 9	—	100
11 - 14	0.1218	93.7
16 - 19	1.0161	47.65
21 - 24	1.7044	
26 - 29	2.5800	Bulk
50 - 55	1.8750	Concrete
80 - 85	1.6050	

Average Bulk Value = 1.9411

(b) Concrete treated

Sample Depth (mm)	Ca(OH) ₂ Peak Height/ Sample Weight (mm/mg)	D _c Degree of Carbonation
0 - 4	—	100
6 - 9	0.5122	68.7
11 - 14	0.5683	65.2
16 - 19	1.6408	
21 - 24	1.7380	Bulk
26 - 29	1.6977	Concrete
50 - 55	1.4260	
80 - 85	1.6722	

Average Bulk Value = 1.635

Table C

System	Depth (mm)
I	1
II	1
III	1
IV	2-3

Another possibility may be that penetration is related to dilution in solvent. Systems I, II and III were all diluted with white spirit whereas System IV remained undiluted. If this were the case, then these results suggest that dilution in solvent reduces depth of penetration. System III, surprisingly showed a poor penetration depth in view of the fact that it consists of 75% monomeric silane. This relatively low penetration may be attributed to several synergistic factors i.e. the effect of the siloxane, dilution by solvent or steric effects caused by the relatively larger iso-Octyl groups compared to the iso-Butyl groups present in IV.

3(e) Effect on substrate pore structure

Figure 7 shows how the Acrylic/Siloxane material, (Solids Weight = 10%), appears to block pores in the range 3×10^3 to 40 Angstroms, compared to the untreated substrate. The Silane also occupies pores in the same range, however, the Acrylic/Siloxane has a greater intruded pore

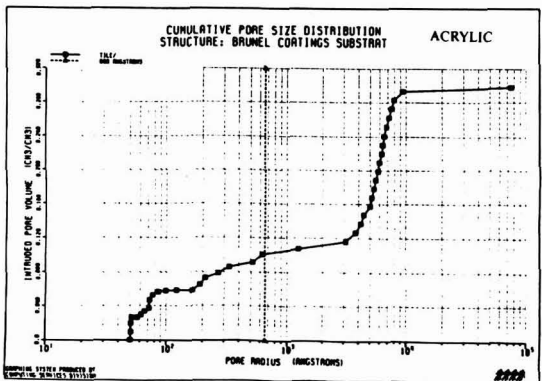
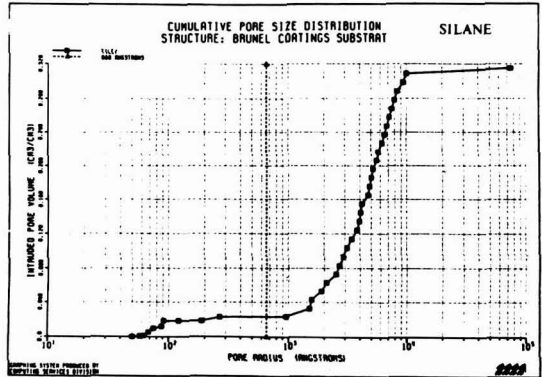
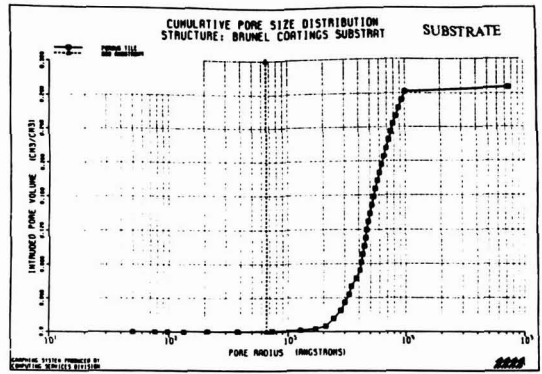
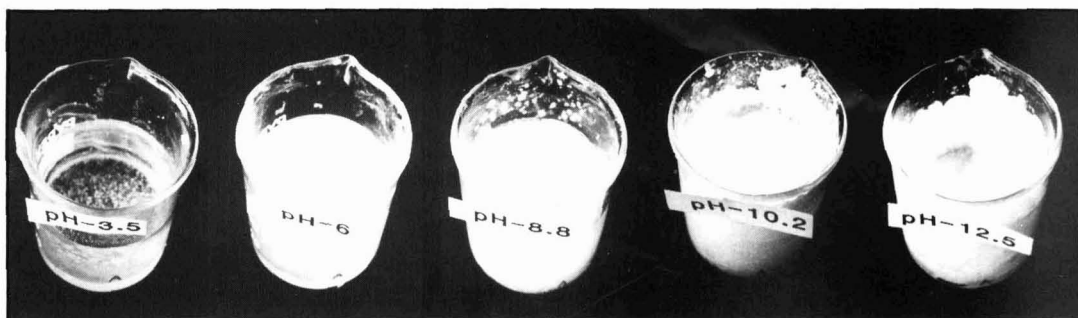


Figure 7. Cumulative pore size distribution of a) unglazed porous ceramic tile b) silane and c) acrylic.

volume. This result infers that the latter material has a greater pore blocking effect compared to the Silane (Weight Solids = 1%) which has mainly a pore lining effect.

3(f) Influence of pH on performance (Table D)

Results tend to indicate that for System III the rate of conversion of the silane into the resin product is related to the pore water pH. The hydrolysis reaction occurs more quickly at higher pH values i.e. complete conversion within three days at pH 10.2 and 12.5. The conversion took longer at pH 8.5 i.e. seven days, whilst at lower pH values i.e. six, no solid resin product was observed. At pH 3.5 a small amount of solid resin product was observed, indicating hydrolysis of silanes to be acid catalysed as well as base



Figures 8-12. Influence of pH on the rate of conversion of the silane into the resin product, photographs taken after seven days.

Table D

		Visual Appearance				
System III		1 Day	3 Days	7 Days	14 Days	21 Days
pH	12.5	Some reaction product	Completely Solidified	Same	Same	Same
pH	10.2	Milky/Opaque	Completely Solidified	Same	Same	Same
pH	8.8	Slightly Milky/Opaque	Milky/Opaque	Completely Solidified	Same	Same
pH	6.0	Clear	Clear	Clear	Milky/Opaque	Milky/Opaque
pH	3.5	Clear	Clear	Slightly Milky	Slight Reaction Product	Slight Reaction Product

catalysed. Similar trends were observed for System IV. Figures 8-12 show the degree of product formation for System III, taken after seven days.

Both silanes analysed, achieved complete conversion at pH 8.8 within seven days. This result is important since in practice these materials may well be applied to carbonated concrete whose pH is approximately 8.8. Hence these results indicate that these two materials in particular will react with pore water in carbonated concrete converting to the silicon resin product.

In practice the relative quantities of concrete pore water and water repellent will be much lower than those used in these tests.

3(g) Effect on moisture loss from concrete

This simple experiment was designed to evaluate the effect, if any, that a silane treatment might have on the rate of moisture loss from concrete. Figure 13 would tend to indicate that the silane treatment has a negligible effect, i.e. after 30 days the treated cube had lost approximately 2.7% of the initial total weight as opposed to a 2.4% weight loss shown by the untreated cube.

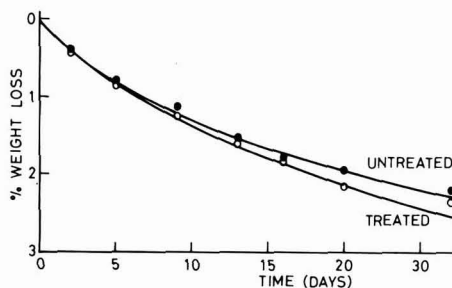


Figure 13. Effect of silane on the loss of moisture from concrete, 50% external humidity, 24°C.

Concluding remarks

The series of tests undertaken were designed to answer particular key questions currently being asked within the construction industry regarding the performance of silicon based water-repellent treatments for exterior concrete application.

Hopefully the test results answer questions regarding chloride ion protection, effect on Carbonation and one or

two other key areas for the four systems submitted for evaluation. It should be noted that the four water-repellents are all commercially available proprietary treatments, although for reasons of confidentiality their trade names have not been revealed.

The main conclusions are as follows:

1. CO₂ diffusion through Silane treated concrete did not differ significantly from the control, however, Siloxane treatments were found to offer increased resistance to CO₂. These results however are not comparable to the resistance offered by polymeric coatings.
2. The carbonation rate through control concrete was found to be similar to treated concrete for each system studied, although no allowance was made for cyclic drying/wetting regimes.
3. A pure monomeric silane outperformed the other siloxanes regarding long term resistance to chloride diffusion through concrete.
4. The relative penetration depths, (1-2mm), for each system were not found to be as high as previously reported for such impregnation treatments.
5. A silane treatment did not significantly modify the

occa meetings

London Section

The confessions of a small paintmaker

The fifth 'technical meeting' of the 1986/87 session was held on 19 March at the "Pearly King", Bow, E3, when some thirty members and guests were entertained to an amusing and witty talk by David Penrice of Newtown Industrial Paints.

David Penrice who is well-known throughout the Paint Industry used as the title for his talk "The Confessions of a Small Paintmaker". Despite the obvious attractions of some of the slides which were shown there was a serious theme running through the talk which largely centred on the setting up and the equipping of a small paint company. The pitfalls and problems associated with such an operation were highlighted and some amusing anecdotes recounted.

Following a question time when it was obvious that some members viewed quite seriously the above mentioned obvious attractions of having your own paint company, a vote of thanks to the speaker was proposed by Ken Arbuckle, Section Chairman.

D. Bannington

Natal Section

AGM

The Natal Section held its AGM on 27 March at the Westville Hotel, Durban, and office bearers and committee members were elected for the 1987/88 year. The speaker of the year trophy was awarded by Dick Philbrick to Mr W. N. Cawood and Prof. D. Williams-Wyn made mention of the fact that council was to honour Ms Ilona Davies with the Jordan Award. The meeting closed at 7.00 p.m. and was followed by a buffet supper for members and their guests.

B. P. Rijkmans

moisture evaporation rate from a treated concrete compared to an untreated control specimen.

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company visits

Macpherson PLC

Mr Charles Wenham, Chief Executive, introduced the 1986 Annual Report and announced the acquisition in April 1987 of Langston Jones and Samuel Smith Ltd, trading as Signpost Paints.

Losses of about £2 million in each of the last three years, following the take-over of Macpherson by the Finnish paint group Tikkurila, have now been eliminated and the 1986 figures show a profit before tax of £2.2 million on a turnover of £101 million. This has been achieved by a massive reorganisation involving redundancies, reduction in overheads, sale of some peripheral parts of the business and a new management structure. They are concentrating on growth of their core business, and have been considerably helped by the Tikkurila expertise in dispersion technology. Their new procedure is to manufacture base colours in bulk using Tikkurila's Omnicolour dispersal system and then make smaller quantities of any colour by tinting, using Tikkurila's Monicolor system. By this means stocks at depots—now called "Paint Centres"—may be reduced but a more rapid service of all colours provided for customers.

The acquisition of Signpost Paints adds a new dimension to Macpherson's position in the retail market. Signpost is the largest independent supplier to the retail trade, being well established in the own-brand and do-it-yourself fields. It is anticipated that with this acquisition the Macpherson group could take third or perhaps second place in the retail market. It is recognised that present margins in this field are too low, often only about 4% on turnover, and Macpherson aim to increase this to 10%.

At present Macpherson is not quoted on the stock market, being wholly owned by Tikkurila which in turn is a subsidiary of the Finnish chemical group Kemira. At some time in the next year or so it is planned to float shares representing up to 49% of the company, with Tikkurila retaining a controlling interest.

T. A. Banfield

Newcastle Section

Water-dispersible epoxy curing agents

The sixth meeting of the 1986-87 Session was held at St Mary's College, Durham, on 5 March when Mr F. B. Richardson of Thomas Swan & Co gave a lecture on "Water-dispersible Epoxy Curing Agents". The meeting was sponsored by Thomas Swan & Co.

Mr Richardson reviewed the development of water-dispersible curing agents by his company since the mid-1960's, when partially acid-neutralized polyamides were first examined. The first marketable product evolved after 2-3 years: it contained organic co-solvent but was low-flam, low-colour and low cost. A completely water-based successor to this came within a further two years and this had significant sales, in Scandinavia particularly, with the UK showing least acceptance. Later developments have since shown higher-solids, paler colour and, most recently, longer pot life. The current market for both paint and non-paint uses is 900-1000 tpa in Europe and Scandinavia with the more recently developed USA market already taking 500 tpa.

The advantages of water-dispersible epoxies in paints are both environmental (non-flam, low toxicity, low odour) and practical (improved adhesion/abrasion-resistance and applicability to new/old concrete and paint). A large non-paint usage is in concrete adhesives and in concrete strength/flexibility improvers.

Mr Richardson reviewed the methods of making water-dispersible epoxy paints using the range of curing agents made by Thomas Swan & Co: these methods involve pigmentation of either the epoxy resin/resin emulsion/curing agent or both resin and curing agent. Manufacture is normally by high speed dispersion or ball milling. A three-pack formula for a primer was illustrated, which could be made two-pack if the resin component was emulsified. Recommendations for gloss paints were discussed, as were anti-graffiti finishes. Specific problem areas with water-dispersible epoxies have been acceptance of universal stainers and performance over solvent-based zinc primers; performance over water-based zinc silicate and acrylic emulsion primers has been good.

The excellent chemical resistance of these products was illustrated, including mineral/vegetable oils, aromatic/aliphatic hydrocarbons and dilute acids/alkalis. These, together with the other advantages already noted, have resulted in the successful use in a very wide range of applications: Mr Richardson listed breweries, hospitals, food storage/processing plants, power stations, concrete car parks, abattoirs, sewage/water-treatment plants, swimming pools and storage silos. He concluded with the most famous structure to benefit, namely the Statue of Liberty during the recent repair programme.

Questions afterwards covered surface preparation requirements, reasons for high usage in Scandinavia and low usage in the UK and ventilation of water vapour during and after application, all of which Mr Richardson answered thoroughly.

The Chairman, Mr R. G. Carr gave the vote of thanks to Mr Richardson and Thomas Swan & Co., after which a splendid buffet with refreshments was enjoyed by all.

J. Bravey

Thames Valley Section

Non-automotive applications of paint electrodeposition

The fifth technical meeting of the 1986/87 session was held at "The Crown", Amersham, on 19 March where the speaker Mr P. I. Hope of LVH Coatings, Birmingham, gave a talk entitled "Non-automotive applications of paint electrodeposition".

Mr Hope gave a brief outline history of electrodeposition which originated with the initial studies by Cross and Blackwell in 1934 using a bees-wax formulation. During the 1940's, the first application of this technology may have been in the wire coating industry. Electrodeposition truly came of age in the 1950's as a method of application for complete coating of interior surfaces of box sections necessary for the construction of the modern motor car.

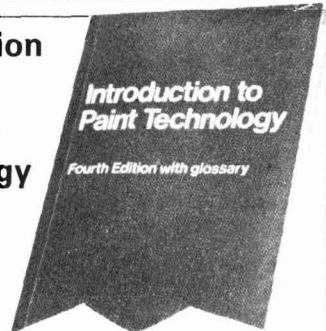
The basis of the talk was to describe: the variety of resin systems currently available to the formulator; techniques employed to ensure adequate film thicknesses which are essential to achieve the benefits of this new technology.

The talk concluded with the speaker recounting some amusing anecdotes from the industry.

The vote of thanks was proposed by Brian Gregory.

J. A. Gant

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The Introduction to Paint Technology, of which over 30,000 copies have been sold, forms an excellent introduction to the whole field of surface coatings and related technologies.

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Oulu Oy merged

Oulu Oy, the well-known Finnish manufacturer of tackifiers for the adhesives industry, has recently merged with one of its parent companies, Veitsiluoto Oy – a leading Finnish paper group. In future the Chemical Division of Oulu Oy will be known as the Chemical Division of Veitsiluoto Oy. The Chemical Division, based in Oulu in Northern Finland, will continue to market its Oulutac range of tackifiers, as well as tall oil and turpentine derivatives.

Hunting acquires EHB

England Hughes Bell & Co Ltd of Manchester has been acquired by Hunting Lubricants and Specialised Products Ltd. The total operation and administration of the business will remain at its Valley Works.

BP Chemicals and Valspar form speciality coatings joint venture in USA

BP Chemicals and The Valspar Corporation of Minneapolis, USA have formed a joint venture partnership to manufacture and market the unique range of oil-absorbent EPOK coatings in the USA and Canada. To be known as Valspar Mebon, the partnership will market the full range of EPOK speciality coatings and sealants based on BP Chemicals' technology in surface adhesion to oil-contaminated substrates.

Polymer Laboratories wins Queen's Award for export

Polymer Laboratories Ltd of Loughborough has received the 1987 Queen's Award for export. In 1986 Polymer Laboratories won the Queen's Award for Technological Achievement. The award is made as a result of overseas sales trebling between 1983 and 1986. Last year's award was for the development of a specialised instrument (the Dynamic Mechanical Thermal Analyser), which rapidly assesses the physical and engineering properties of polymers and composites.

W. R. Grace & Co markets BP's Shieldex

W. R. Grace has signed an exclusive agreement with BP International to market worldwide Shieldex®, a new non-toxic anti-corrosive pigment which works by ion exchange.

1,3 – Butadiene new limits

A Control Limit for 1,3 butadiene has been adopted by the Health and Safety Commission. With effect from 1 January 1988, occupational exposure to 1,3 butadiene air should be controlled so as not to exceed 10 parts per million (ppm) eight-hour time weighted average (TWA).

Crown Paints up for sale

Reed International, the magazine and packaging group, has announced that it hopes to raise £200 million from the sale of Crown Paints and its Polycell business in Europe and N. America. Leading those interested in buying the business is a Crown Management buyout consortium. In charge of the consortium is Peter Burns (Group Chief Executive of the Paint and DIY Group, Reed International PLC) which consists of Paul Lever (Managing Director of Crown Paints), Ray Freshfield (Group Finance Director), Mel Keeler (Director of N. America paint businesses) plus over twenty senior managers.

The Paint and DIY Group employs 5000 people and had a turnover of £288m and a trading profit of £18.9m in the year to 31 March, 1986. The UK accounted for £133m of this turnover, with the overseas turnover amounting to £155m. Trading profit was £8.1m and £10.8m respectively.

Charterhouse Development Capital is supporting the management team's approach for the Paint and DIY Group.



Peter Burns

Commenting on the consortium's approach, Paul Lever said "This is a challenging opportunity for us. The mix of businesses in the Paint and DIY Group, and its geographical spread in the UK, Europe and the USA offers exciting prospects". Charterhouse Development Capital has financed 54 buyouts worth almost £1 billion over the last five years.

products

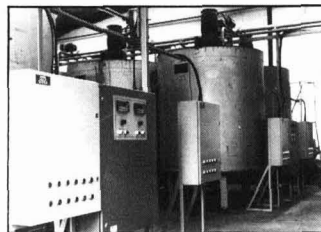
Automated weighing for paint blending

Paint blending in 20 new vessels at Finnigans Speciality Paints Ltd is automatically weight-controlled and monitored using highly accurate load cell based systems supplied by Defiant Weighing Ltd, the Sevenoaks company.

Process operators can now bulk fill each tank with materials to an accuracy of one part in a thousand and then dose out exact quantities to filling machines, thus avoiding wastage. The Defiant load cell system is more flexible than flowmetering and provides a constant record of tank contents plus a loss-in-weight system to monitor the actual amount of paint manufactured before can filling.

Finnigans, part of the Hunting Group, is expanding its production facilities at Prudhoe, Northumberland, to meet export demands for Hammerite paints and Waxoyl rustproofing compound. As the firm is automating certain processes, it needed a weight control system which would interface with a programmable logic controller (PLC) and give comparable accuracy.

The company has installed 19 new paint blending tanks of 5.5 cubic metres and 13.5 cubic metres capacity. The smaller units each have a 3000 kilograms shear-beam load cell on one of their three legs with pivots on the other two, whereas the larger four-legged tanks use 5000 kilograms cells on two legs. Waxoyl processing, however, needs high-tolerance blending, so this particular vessel has load cells on all four legs.



Defiant load cell system

Each shear-beam load cell is machined from a solid billet of stainless steel. One end is firmly bolted to the floor and the other has a mechanical mounting to take the tank leg. As the weight of a tank's contents alters, the load cell deflects minutely. This is accurately sensed by built-in transducers which are linked to a Defiant FAM function module system at the process operator's station.

Reader Enquiry Service No. 20

New high thermal conductivity resin

Robnorganic Systems of Swindon, has available a new high thermal conductivity encapsulating and casting resin. Principal advantages of the new resin PX666C are very high thermal conductivity, low viscosity, flame retardancy and minimal sedimentation in store. The thermal conductivity of the resin has been measured at 1.4 watts/m.k. This high value is achieved through the combination of a non-abrasive filler with a high purity resin system.

Reader Enquiry Service No. 21

Low colour resin

Industrial Copolymers Ltd (specialist resin manufacturers) of Preston announces an addition to its product range *Incozecz 145A*. This compound is a low colour, reactive, polyamide resin which can be used to emulsify and harden epoxy resins for water based surface coatings. The low colour enables the production of white and pastel shades coatings. This product has a pot life extending up to six hours at 20°C.

Reader Enquiry Service No. 22

Cold bend test cabinet

Sheen Instruments Ltd of Teddington has available the *Cold Bend Test Cabinet* developed for testing paint coatings to BS 3900 El. This Spec calls for the paint to be applied to a sample of metal which after a suitable drying time is subjected to a cold bend test at temperatures down to and below freezing point. The coating should be able to withstand cracking or flaking under the test conditions so that the metal substrate is still protected from weathering. The test is carried out on a piece of metal approximately 0.3mm thick and 2½ by 4in which, after the paint has been applied and cured, is mounted in a device which can be made to bend the metal over a mandrel. Various mandrels from 3 to 25mm diameter are available. The bending machine being mounted inside a refrigerator.

Reader Enquiry Service No. 23

meetings**CVP arranges tour to the American Paint Show**

The 52nd Annual Meeting and Paint Industries Show of the Federation of Societies for Coatings Technology will be held in the Dallas Convention Centre, Dallas, Texas on 5-7 October 1987. CVP have planned two trips, a short trip which includes sightseeing of Dallas (including the "Southfork Ranch") and three days at the Paint Show. A longer tour has also been planned, which includes additional visits to major paint companies in the Dallas/Fort Worth area, and taking in a long weekend in New Orleans. The theme for this year's show is "People and Technology, Cornerstones of Progress" and workshops will feature papers on the training of vital people, as well as technological developments in such areas as coating of plastics, manufacturing, and corrosion control. The President Designate of the Oil & Colour Chemists' Association (Mr J. R. Bourne) will be presenting a paper on behalf of the Association. For tour details contact: Tracey Moss, Cray Valley Products Ltd, Farnborough, Kent BR6 7EA.

literature**Bright picture for the Paint Industry**

A new Report from ICC states that the paint and printing ink industry has benefited from the recovery in the Printing industry and from the consumer boom. Trading conditions have steadily improved since the low point in 1980/81, and the Report goes on to show that the industry's aggregate performance indicators are good and getting better. The Business Ratio Report from ICC, 'Print and Printing Ink Manufacturers - 15th Edition' analyses the financial performances of 105 leading companies in the industry over a three-year accounting period to March 1986. The companies are divided into three sub-sectors: paint and varnish manufacturers, printing ink manufacturers and dye stuffs and pigment manufacturers.

Within the industry, the dyestuffs manufacturing sector appears to have been enjoying a small boom, with an overall sales increase of 46.5%. But this is due mainly to the huge growth of the Tioxide Group which is attributed in part to acquisitions. Printing ink manufacturers, on the other hand, suffered near-static sales over the latter half of the period. After a growth rate of 23% over the first 18 months, sales increased by only 0.7% over the following equivalent period.

The largest company by third year sales was Tioxide Group Plc with £426m sales in 1985/86. Next largest was International Paint Plc (£382m in the same year), and then Coates Brothers Plc, who had 1985/86 sales of £189m.

Profitability for the industry is on the whole good. Profits for 82 companies rose from £81m to £150m over three years, while the average profit margin also increased over the same period from 4.5% to 6.8%. Dyestuffs and Pigments Manufacturers managed to raise their margins from 7.4% to 15.6%, an impressive performance alongside the sector's sales growth, but both the Paint & Varnish and Printing ink sectors suffered final year slips in their profit margins, to 3% and 4.9% respectively. Other measures of performance show improvements. Return on capital employed has risen from 8.7 to 14.7%, asset utilisation is up from 1.2 to 1.4, stock turnover has increased to 5.6 times a year, and the average debt collection period has been reduced by 4 days to 76 days.

Apart from a few problem companies, conclude ICC, the Paint and Printing ink sector looks in very healthy shape.

The Report - Painting & Printing Ink Manufacturers, 15th Edition priced at £155.00 is available from Simon Ingman, ICC Business Ratios, 28/42 Banner Street, London EC1Y 8QE.

company reports**PPG Industries Inc**

PPG, the world manufacturer of glass, coatings, resins, and chemicals, reported that net sales in 1986 were up at \$4.7b (+8% on 1985) with net earnings of \$316b (+4% on 1985). Coatings and resins sales of \$1.6 billion were up 12% over the \$1.4 billion recorded in 1984. Coatings sales to the North American original equipment automotive market continued to be strong in 1985 as a result of increased industry volume and improved market penetration. North American automotive refinish sales also registered a strong year, as did industrial finishes. European sales increased as a result of acquisitions in Germany late in 1984 and in England in March 1985. Operating earnings (coat & res) of \$250m were 3% higher than the previous year. Coatings and resins accounted for 37% of company sales and 40% of operating earnings.

Laporte Industries PLC

Laporte Industries, manufacturers of absorbents, building and timber products, and inorganic and organic specialities reported that pre-tax profits in 1986 increased to £64.2m (+15.1% on 1985) on increased sales of £422m (+13.5%).

people

Robert F. Ziegler has been appointed to the position of Executive Vice-President, of the FSCT effective October 31, 1987. He will succeed current retiring Executive Vice-President **Frank J. Borelle**.

Dr Elmar Frommer, Director responsible for BASF Group polyolefin and PVC activities worldwide is appointed to the Board of BASF UK Ltd.

John Fraser, Group Managing Director of CIBA-GEIGY PLC, has been appointed Chief Executive of Ciba-Geigy UK.

K. F. Plant, Managing Director (Operations) will become Chief Executive of the Tioxide Group PLC from October.

I. G. Butler, a non-executive Director of Tioxide Group PLC and Chairman of Cookson Group PLC, will become Chairman of Tioxide Group at the same time.

OCCA ties at £4.25 each are now available from the Association's offices, with a single Association Insignia on either a blue or maroon background.

Report of Council Meeting

A meeting of the Council of the Association took place at 1.30 pm at the Great Northern Hotel, King's Cross, London N1 9AN on Wednesday 15 April 1987. The President (Mr F. B. Redman) was in the chair and 24 members were present.

It was reported that Dr R. A. Ellis had agreed to act as the Association's nominee on the BSI Committee C/17 (now entitled PTC/14).

The President informed the Council of the present position with regard to the appointment of a General Secretary Designate. The agenda for the AGM was confirmed, it being noted that Mr A. J. Newbould would be unable, because of business commitments, to continue as Hon. Technical Education Officer. Section Chairmen were asked to ensure that their Sections' Annual Reports were sent to Priory House by the end of the month and that details of the Committee for the forthcoming sessions were also submitted as quickly as possible. The United Kingdom and Irish Sections were urged to send their programmes for the 1987/88 Joint Programme to Priory House by early June so that adequate time was given for the preparation and checking of proofs, the joint programme would be circulated as usual with the August issue.

The Annual Accounts for 1986 as presented by the Finance Committee were adopted by the Council for publication in the Annual Report. The Finance Committee's estimates for 1987 were adopted by the Council. It was agreed that the names of members whose 1987 subscriptions had not been received by the end of June should be removed from the Register.

The Hon. Editor reported on papers available for publication in JOCCA and the necessity for all members to influence suppliers to use its pages for advertising and paint manufacturers to use the classified columns for situations vacant.

There was a possibility that a fourth Monograph might be published later in the year. The Hon. Editor reminded Council that he would always consider, for publication in JOCCA, letters concerning the Association's activities since JOCCA is the official publication of the Association and he might wish to exercise his right to reply. The same condition would not, however, apply to letters or comments concerning the Association in other publications since it had not been the practice in the past to enter into correspondence about the Association in other journals.

Council endorsed this view.

All but two papers, one being a Keynote Address, for the Eastbourne Conference had now been set in proof form. The President Designate offered to present a paper on behalf of the Association at the FSCT Convention at Dallas 5-7 October 1987 and papers were now being sought for presentation on behalf of OCCA from amongst members attending the SLF Conference at Helsinki 9-11 May 1988 and the FATIPEC Conference at Aachen 18-24 September 1988. Members willing to help the Association in this way were asked to advise the Director and Secretary.

News concerning the progress of the Open Tech Project was received by Council with considerable interest and it was hoped that a number of those who completed Module 8 in 1988 would apply for admission as LTSC.

Applications for the Eastbourne Conference were proceeding at the same level as for previous Conferences and the attention of the Chairmen of Bristol, London and Thames Valley Sections was drawn to the special one day visits which could be arranged by those Sections.

The Hon. Overseas Officer reported on his visit to the Far East and the interest shown in OCCA activities there, which it was his intention to foster.

It was reported that at the Professional Grade Committee held earlier in the day, two Fellows and four Associates had been admitted, one application for Associateship was deferred until the required period of OCCA membership, arrangements had been made for a viva for one LTSC candidate and for the assessment of the written evidence supplied by another LTSC candidate.

It was reported that with the additional space made available by the use of the auditorium at the Harrogate Conference Centre 114 stands could be occupied; already 104 had been reserved.

Mr D. S. Newton, as the senior serving Hon. Officer, proposed a Vote of Thanks to the retiring President for all the effort which he had shown in carrying out the many tasks which fell to the President during his term of office. He had served the Association with distinction and Council carried the Vote of Thanks with acclamation.

A Vote of Thanks was also passed to all those members retiring from Council for their support during their period of service on Council.

It was reported that in the view of the imminent retirement of the Director and

Secretary a special meeting of the Executive Committee had been held, at which each officer outlined the duties and responsibilities which would be assumed by the office holder concerned and the support which would be expected from the General Secretary Designate.

This being the final Council Meeting before the AGM and Conference after which he would be going on leave, the Director and Secretary expressed his thanks to all Council members, past and present, who had supported him over his 36 years as chief executive officer. He particularly wished Council to consider for the future the recruitment to the professional grade which should arise from the completion of the Open Tech project and the publication of more monographs particularly in non-Exhibition years. The President, on behalf of the Council thanked the Director and Secretary for his untiring work and enthusiasm for the welfare of the Association and Council wished him a long and happy retirement.

There being no other business, the President declared the meeting closed at 3.15 pm.

professional grade

At the meeting of the Professional Grade Committee held on 15 April 1987 the following admissions were made:

Transferred from Associate to Fellowship

Boxall, John (*Thames Valley*)

Admitted to Fellowship

Barrett, Ronald Leon (*Scottish*)

Admitted to Associateship

Gallon, Mark Robert (*Manchester*)
Horn, David Michael William (*Thames Valley*)
Nicholson, John William (*London*)
Saville, Rowland Whincup (*West Riding*)

new members

The sections to which new members are attached are shown in italics together with the country, where applicable.

Ordinary members

Arstall, M. J., BSc (*Midlands*)
Cruger, S. L. (*General Overseas—Zimbabwe*)
Houlihan, B. (*Irish*)
Lee, M. W. (*Midlands*)
Quintal, E. (*General Overseas—Kenya*)

Registered Student

Burnside, G. S. (*Scottish*)

Newcastle Section

Ladies Night 1987

The annual Ladies Night dinner dance of the Newcastle Section was held on Friday 20 February 1987 at the Five Bridges Hotel, Gateshead. An excellent dinner was followed by first rate dance music to which members and their wives trod a fair measure. During the evening the chairman of the section Mr Gordon Carr presented Mr Robert Hamblin Director and Secretary of OCCA with an inscribed "Miner's Lamp" to commemorate Robert's many years of service to the Association and to wish him well for a long and happy retirement.

L. Morpeth



Newcastle Section Ladies Night. Standing (left to right): Mr F. B. Redman, Mr D. McKay, Mr B. Windsor, Mrs D. McKay, Mr G. Carr, Mrs G. Lewis, Mr R. H. Hamblin, Mr M. Prigmore and Dr G. Lewis. Seated (left to right): Mrs B. Windsor, Mrs R. G. Carr, Mrs F. B. Redman and Mrs H. Prigmore.

News of Members

M. H. Gamon (Hull Section Chairman) has been appointed a Director of Pearson Panke Equipment Ltd.



Thames Valley Dinner Dance. Mr J. L. Inshaw (Chairman) presenting the retiring Director and Secretary with a whisky decanter and glasses on behalf of the Section.



Thames Valley Dinner Dance. Back row (left to right): Mr K. H. Arbuckle (Chairman, London Section), Mr M. Prigmore (Chairman, Bristol Section), Mr J. Inshaw (Chairman, Thames Valley Section), Mr F. Morpeth (Vice-Chairman, Manchester Section), Mr F. B. Redman (President) and Mr R. H. Hamblin (Director and Secretary). Front row (left to right): Mrs Prigmore, Mrs Arbuckle, Mrs Redman, Mrs Inshaw, Mrs Morpeth.

Bristol Section Dinner Dance (Photo on the right). Back row: Mr T. Wright (Chairman, West Riding Section), Mr R. G. Carr (Chairman, Newcastle Section), Mr F. B. Redman (President), Mr M. Prigmore (Chairman, Bristol Section), Dr G. Lewis (Chairman, Midlands Section), Mr K. H. Arbuckle (Chairman, London Section), Mr G. Fowkes (President, BPVL Club) and Mr R. H. Hamblin (Director and Secretary). Front row: Mrs Wright, Mrs Carr, Mrs Redman, Mrs Prigmore, Mrs Lewis, Mrs Arbuckle and Mrs Fowkes.



SITUATIONS VACANT

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7. **Adhesives Chemist** for development, product presentation plus trials particularly for automotive applications. MIDLANDS **Up to £15,000 p.a.**
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10. **Young Oil Inks Graduate Chemist** as project leader for development product presentation and trials. SOUTH **£12,000 p.a.**
11. **Decorative Paints Graduate Chemist** to lead development, services and QA for major company. SOUTH **£18,000 p.a. and car**
12. **Graduate Packaging Inks Technical Manager** to lead development team for major liquid inks company. SOUTH **£18,000 p.a. and car**
13. **Graduate Oil Inks Senior Technical Manager**, 30-40, to report to chief chemist and responsible for development, servicing and QA teams. SOUTH **£18,000 p.a. and car**
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Oil & Colour Chemists' Association

ANNUAL GENERAL MEETING—19th JUNE, 1987

IMPORTANT NOTICE TO MEMBERS

The combined notice convening the Annual General Meeting and the nomination form for the three elective places on Council was enclosed in the *Journal* sent to all members in the General Overseas Section (December 1986) and U.K. and Irish Sections (January 1987). In addition, in accordance with previous practice and Article 83, a copy was sent by airmail to the Chairman of each of the overseas Sections.

By the closing date for nominations (24 April, 1987) the number received did not exceed three and, under Article 43 (ii) where the number of persons nominated is no greater than the vacancies, the persons so nominated shall be declared elected. Accordingly, no postal ballot is necessary.

Copies of the Annual Report of the Council and the Agenda are being circulated with the May issue of the *Journal* to all Members. In accordance with Article 83, copies of the Agenda and Annual Report have been despatched by airmail to the Chairmen of the four overseas Sections, together with a copy of this notice.

The May issue of the *Journal* in which this notice is enclosed is being sent by AIRMAIL to members of the General Overseas Section who are resident outside Europe and accordingly may reach some destinations before the April issue, which was despatched by surface mail as usual.

PRIORY HOUSE,
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Robert Hamblin,
Director and Secretary.
27 April, 1987

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Brightness	Approx 98	(DIN 53163)
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Specific surface area	3 m ² /g	(DIN 66132)
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