



JOURNAL OF THE OIL AND COLOUR CHEMISTS' ASSOCIATION

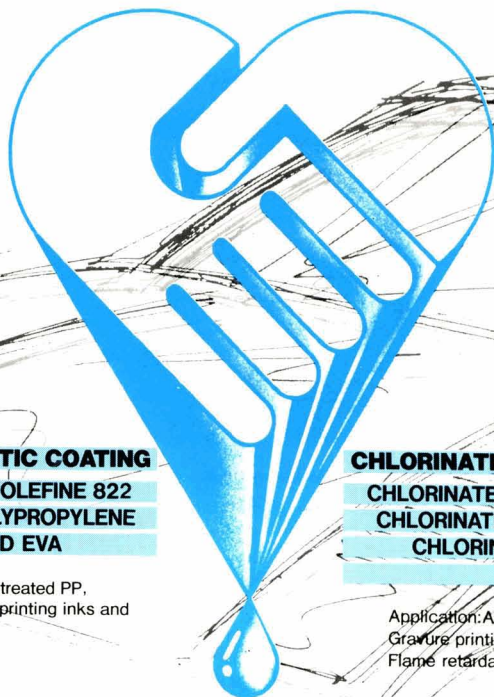
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An official Journal of the Paintmakers Association of Great Britain Ltd.

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Cover: Power station site, structural steel blast-cleaned to SA2½, 2 coats of vinyl/acrylic copolymer waterborne primer and pure acrylic waterborne finish (Photo courtesy of ICI Resins).

Forthcoming Features: January — Corrosion, Process Operation; Dispensing and Mixing, February — Quality Assurance; PO, Computer Controls, March — Adhesion; PO, Filtration.
Contributions are welcomed at least five weeks prior to publication date.

JOCCA is published by the

Oil and Colour Chemists' Association

Priory House, 967 Harrow Road, Wembley HA0 2SF, England

Tel: 01-908 1086 Fax: 01-908 1219 Telex: 922670 (OCCA G)

Annual subscription rate for 1989: £80.00 (\$160), post free by surface mail.

Single copies £8.00 (\$16), post free by surface mail.

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Printed by The Herald Press, 17 Rother Street, Stratford-upon-Avon, Warwickshire CV37 6NB Tel: 0789 66261



The new Jotun-Henry Clark marine and industrial coatings plant at Flixborough.

Jotun – Henry Clark opens new UK factory

Jotun – Henry Clark (part of the Jotun Group), manufacturer of protective coatings since 1832, has commenced production from its new £5 million plant at Flixborough. Built primarily to supply EEC markets, the plant will also export its marine and industrial coatings to Scandinavia, the Middle East, the Falklands, Costa Rica, Panama, Bermuda, Malta, Turkey and Caracao.

Based on the most advanced production equipment, the design of the new facility has benefited from the wide experience of the Jotun group in building such plants throughout the world.

Occupying an 8-acre site and planned staffing of up to 100 the

Part of the finished goods warehouse at Jotun-Henry Clark's Flixborough plant.



new plant includes an administration building, raw materials store, bulk storage area, production area and finished-goods warehouse.

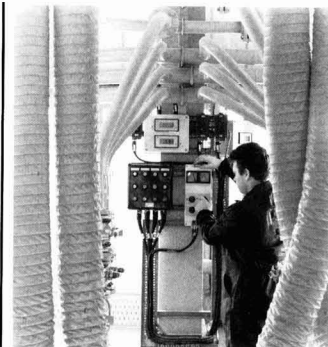
The administration building, covering 500m², accommodates staff responsible for operating the plant, together with local-area sales and technical support teams.

The raw materials store covers 1000m², and houses materials that must be protected from the weather before being moved into the production area. A link building to the production area allows all-weather movement of the materials.

Bulk storage tanks contain the solvents and resins needed in the manufacturing process, and have capacities ranging from 20 to 60m³. Many of the tanks, along with pipelines to the production area, can be heated to optimise flow properties in cold weather.

The production area is on two levels, each of 1800m², to provide gravity feed from mixing tanks to the filling and packing areas. Annual output will be 8 million litres initially, increasing to 10 million.

The production equipment consists of dissolvers, mills, mixing tanks, weighing machines, filling lines and some 70 associated tanks with capacities up to 6000 litres.



Regulating the mixing speed of Diap dissolver at Flixborough. Above the control box are load cell displays of the weight of material in the dissolver, and to the left are push buttons for the control of pumps.

The dissolver supply tanks incorporate load cells for high-accuracy batch weighing, the data being fed to a computer for automatic checking and recording to ensure that each product conforms exactly to its formulation.

Both automatic and semi-automatic filling lines are installed. Currently, two automatic lines allow 5000 litres of paint to be filled and palletised hourly. Later, when automatic palletising has been installed, this output will be doubled.

The quality control team is also accommodated in the production area, and is responsible for the



Nick Scotland, Managing Director of Jotun-Henry Clark welcomes guests to the opening ceremony of the new Flixborough factory. Sharing the platform with him are (l to r) Mr Odd Gleditsch Jr, Chairman of the Board of Jotun A/S and the Mayor of the Borough of Glandford, Councillor Winston Woods. The ball mill behind them is a 1901 porcelain lined pebble grind media ball which was used in the old factories at Millwall and then Brighton.



At the Jotun press conference (l to r): Martin Hersey, Factory Manager; Derek Clark, Director and Board Member Jotun-Henry Clark; Odd Gleditsch Jr, Chairman of the Board of Jotun A/S; Nick Scotland, Managing Director of Jotun-Henry Clark Ltd and Morton Olsen, Managing Director of Jotun A/S.

testing of both incoming raw materials and outgoing products. The plant has been set up to operate in accordance with BS 5750.

The finished goods warehouse, of 1800m², has racking for over 2500 pallets of paint. Depending on product type and can size, this represents some 1.3 million litres. Fire protection for the whole racking system is provided by sprinklers.

New World leaders in colour management

Brauerie Eichof, parent company of Swiss colour instrumentation specialists, Datacolor — whose exclusive UK distributors are Yorkshire Colour Systems Ltd of Bradford — is acquiring US company Applied Colour Systems Inc (ACS), an Armstrong World Industries company. The approved

transaction is expected to be completed by the end of November with both Datacolor and ACS remaining as autonomous companies. ACS's 1989 world-wide sales are £19.2 million and Datacolor has consolidated annual sales of £12 million.

News in brief

Croda Paints has completed Phase 1 of redevelopment of their Hull based manufacturing plant with the installation of bulk resin and storage facilities.

Croda Resins has signed an agreement with Monsanto to market Resimene high solids, cross linking, amino resins in the UK.

Courtaulds Coatings has acquired 85% of the capital of Hoba Industrie-Chemie GmbH, a leading West German manufacturer of specialist coatings for application to metal and plastic packaging.

Thomas Swan & Co Ltd of Durham has registered for BS 5750 Pt 1.

Baxenden Chemicals of Accrington has registered for BS 5750 Pt 2.

Dow Corning Plants — silicone products — in Barry, (UK) and Seneffe (Belgium) has registered for ISO 9002/BS 5750.

ICI Colour & Fine Chemicals phthalocyanine pigments has registered for ISO 9002. The phthalocyanine green pigments are manufactured at Grangemouth, Scotland and phthalocyanine blue pigments are manufactured at Villers St. Paul, France.

Hampstead Colours (Division of Floridienne (U.K.) Limited) have moved into their new laboratory block.

Solrec Ltd, the solvent recovery company, has centralised its chemical recycling to a new £3/4m factory in Morecambe.

Evans Clay Co have purchased the airfloat kaolin clay business of J. M. Huber Corp, Georgia.

NL Chemicals restructures: KRONOS and RHEOX are formed

The incorporation was announced this week of the two new companies KRONOS, INC., and RHEOX, INC., created to operate respectively, the titanium dioxide pigment business and the speciality chemical business formerly known as NL Chemicals, Inc. The two new companies are subsidiaries of Houston, Texas based NL Industries Inc, but will act totally independently on a global basis. NL Industries corporate Vice President, Fred Montanari said that the creation of the two independent worldwide companies would enable them to adopt a more focused management approach that would be of direct benefit to all customers.

Currently the world's fourth largest producer of titanium dioxide pigments, KRONOS, INC.'s production exceeds 320,000 metric tons of pigment per year, with manufacturing facilities located in Canada, West Germany (2), Belgium and Norway. A sixth plant is currently under construction in

Lake Charles, Louisiana, USA, which will increase the company's pigment capacity by 80,000 metric tons.

Titanium dioxide products will now be sold under the trade name KRONOS in North America. The TITANOX brand name will be phased out. "We are completing the globalisation process for our pigments business by bringing the KRONOS name to North America," explained Montanari. "Our large, multi-national pigment customers will immediately recognise benefits such as uniform product nomenclature and specifications." "KRONOS, INC., is headquartered in Houston, Texas.

The restructuring launches RHEOX, INC., as the world's largest producer of rheological additives for solvent-based systems, with products sold under the familiar trade names of BENTONE, THIXATROL, THIXCIN, M-P-A, BARAGEL and RHEOLATE. The company also manufactures NALZIN 2 anti-corrosive pigments for high-

performance coating systems.

RHEOX, INC., currently has sales of well over \$100 million per year worldwide. There are five manufacturing facilities in the USA and Europe. European operations being Abbey Chemicals Ltd., of Livingston, Scotland and Benton-Chemie at Nordenham, West Germany.

RHEOX's global headquarters and research and development laboratories are located in Hightstown, New Jersey, USA. The European headquarters, headed by David Wood, is in Brussels with technical support based in Leverkusen, West Germany.

RHEOX customers will particularly benefit from the restructuring in the areas of new product development, according to RHEOX, INC., President, Michael J Kenny. "In the speciality chemicals business, customer interaction and concentrated technical support is essential to the new product development process. As a separate, independent, specialities company RHEOX can now focus its resources to serving this need."

Orkem acquires Coates

Orkem, the nationalised French chemicals company has acquired Coates Brothers, the inks, synthetic resins and coatings group for £300m.

ICI Resins expands into powder coatings

ICI Resins is expanding its product range through production of speciality polyester resins for a variety of applications — with particular emphasis on the rapidly expanding powder coatings market. A multi-purpose polyester plant is being built at the ICI Resins factory in Waalwijk, Holland and will come on-stream in the second quarter of 1990. At the same time, a new research and applications-support laboratory is being set up at Runcorn in the UK.



SCM ISO 9002: left Dr R. V. Pearse, Quality Assurance Manager at SCM Chemicals Limited and right, Mr Jim Hancock, Director and General Manager of SCM, proudly bearing the certificate for the registration to International Standard for Quality ISO 9002-1987.

SCM gains ISO 9002

SCM Chemicals Limited, based in South Humberside, England, has gained registration to the International Standard for Quality Management Systems ISO 9002 for the production of their TiONA range of Titanium Dioxide pigments, and for their service to the market. The ISO 9002 standard is the international equivalent of BS 5750:Part 2:1987

**JANUARY:
UK CORROSION 89
REVIEW**

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Products

New Degussa binding agents

Degussa have introduced the following new binding agents: Degalex VP-BF 150 is specially intended for formulations of non-blocking high-gloss dispersion paints. Degalex EM 170 is for formulations of façade and concrete protective paints. Degalex VP-EM 210, an acrylate/alkyd emulsion, has been developed specifically for formulas of aqueous wood stains and varnishes. Degalex VP-EM 174 is a particularly abrasion-resistant binding agent for pro-environmental, aqueous parquet varnishes.

For further information Enter L201

Sandoz dispersions

Sanduvor 3225 Dispersion is an aqueous preparation of a mixture of a UV absorber of the benzotriazole type and a radical scavenger. As both active substances are very finely distributed the product is optimal for use in water-based dispersions and emulsions. Based on its high absorption in the 290-370nm range, the product is ideally suited for stabilising water-based lacquers and coatings against the effect of UV light. Sanduvor 3225 Dispersion reduced loss of gloss and cracking due to the influence of light and weathering and in transparent systems reduces the discoloration of the substrate without any influence on the transparency of the clear lacquer.

For further information Enter L202

Equipment

Winkworth high speed mixer with extrusion discharge

Superior discharging control has now been introduced by Winkworth Machinery Limited for their High Speed RT Refiner Mixer of powders, pastes, slurries, ganules etc. Rapid mixing is achieved by a profiled paddle or plough type blade,



Winkworth High Speed RT Refiner with extruder screw nozzle.

revolving in a cylindrical process chamber with the option of high speed refiners.

A variable speed extrusion screw with interchangeable discharge devices allows doughy materials and pastes to be discharged under tight control directly into containers, onto a conveyor or via a tube filling device. Control of powders or granules can also be achieved whilst discharging.

The removable extruder screw with electronically controlled variable speed drive runs in reverse during mixing drawing material into the process chamber and eliminating both the need for a valve and the 'dead' space associated with slide, butterfly or ball valves. The designs and technology of the extruder screws available are based on the well proven Winkworth 'Z' Blade Extruder Mixers which are used for really tough doughs and pastes.

The photograph shows a 200 litre vacuum model with an extruder nozzle which can be quickly changed for a tube filler or tub filling device so that the mixed de-aerated product can be directly filled from the mixer.

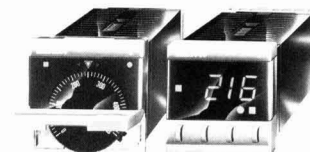
The range is available from 25 to 2500 litres as standard, with all models available in mild or stainless steel, jacketed vacuum, with or without high speed refiners and with profiled paddle or plough blades. Trial facilities are available at the Winkworth Customer Test Centre, where this new concept of mixing and extruding can be tested.

For further information Enter L203

Formulation aid

Ledger Scientific has introduced its computer program ACRYCALC for IBM — compatible computers. ACRYCALC is a formulation aid for acrylic and vinyl copolymerisation. Using the Q and e values of the monomer, it calculates the composition of the polymer produced by copolymerisation of up to 10 monomers. A routine for calculating the estimated Tg from the Tg's, of the homopolymers is also included.

For further information Enter L204



CAL 9900 and 8000

New miniature temperature controllers

CAL have introduced the digital, auto-tuning CAL 9900 series and the analogue CAL 8000 series which uses surface mount technology, to pack the maximum features into the space saving 48mm² formats. Both controllers operate in the range -100° to 1600°C and are ideally suited to multi-zone applications needing precise thermal profiles.

For further information Enter L205

US Focus

by Abel Banov,
Co-publisher of the American
Paint & Coatings Journal

More flexible epoxies for containers, appliances

The need for more flexible and formable coatings for containers and appliances has long been recognized, and now a new epoxy phosphate ester resin has come along to fill the need.

Where the usual solid epoxy resin is relatively non-reactive with the usual curing agents, the phosphate esters behave like telechelic polymers (which means they have reactive end-groups). Exceptional improvements have been reported when the phosphate esters are substituted for the base resins.

Researchers at Dow Chemical Co found by converting 1,2-oxirane end groups in commercial epoxy resins into reactive end groups, the new resins can increase linear molecular weights during curing, thus improving flexibility. In a wedge-bend test, flexibility loss at all percentages of crosslinker was found to be vastly improved when the phosphate ester resin was used.

Although reduced by the change, solvent resistance of the resulting coatings was found to be adequate for many applications. After a 10-minute bake at 400 F, the use of 30 per cent crosslinker,

for instance, resulted in a successful test at 200 double-scrubs with methyl ethyl ketone.

The Dow Products are XP 71739; XU 8096.07; XU 71926; and XU 71899.

Harder, more flexible Exterior Powder coatings

Powder coatings that can take weather and still have sufficient hardness and flexibility have not been commonly available. Now, a new crosslinking agent for polyester and acrylic polymers has been found capable of imparting these desirable characteristics. The material, highly monomeric are mostly tetramethoxymethyl glycoluril, belongs to the family of aminoplast crosslinking agents.

Offered now in semi-works quantities, the product is produced by American Cyanamid Co; a full plant will be completed in the third quarter of 1990. In addition to hardness and flexibility, the product has excellent solvent resistance and low-temperature cure potential. Low weight loss is reported during cure.

Recommended applications include lawn and garden equipment and furniture; automotive trim, and building products, including extrusions, as well as appliances and general industrial equipment.

The company's technical staff has prepared starting formulations and notes that to ensure good flow during bake and to obtain coatings with high gloss, a blocked catalyst such as a sulfonate ester is recommended in some formulations. Highly basic fillers,

such as calcium carbonate, should be avoided because they tend to buffer the acid catalyst and retard cure. Clay extenders are suggested for filled formulations.

The Cyanamid product is Powderlink 1174.

Universal latex for premium architectural

One latex to cover all architectural coating bases has been sought for a long time. Now Reichold Chemicals is marketing a vinyl-acrylic latex product claimed to be capable of providing outstanding scrub resistance and touchup in interior flat wall paints; excellent gloss and water resistance in interior semi-gloss; and good weatherability and water resistance for exterior finishes.

The product can be formulated with either cellulosic and/or associative thickeners, and provides considerable formulating latitude while maintaining consistent viscosity control.

The Reichold product is a Wallpol 40150.

Reactive diluent Reduces volatiles

Reductions of volatile organic compounds by 0.5lb/gal, or more, have been reported when a new reactive diluent is used.

Union Carbide Corp, producers of the material, designed the product so that solids can be increased while reducing viscosity of crosslinking coatings. The diluent can be used in water-dispersible coatings systems based on alkyd, polyester, and

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acrylic polymers, crosslinked with melamine or other amino resins. The materials is a 75 per cent solution of hydroxyethyl ethylene urea in water. It serves as a cosolvent in the liquid coating.

The Union Carbide product is UCAR RD 65-2.

Literature

Surfactant training modules

The British Association for Chemical Surfactants (BACS) are now producing a training programme on the technology and use of surfactants in industry. The intention is to provide a text plus 35mm slide illustrations which can be used to give a lecture. S1 — An Introduction to Surfactants, S2 — Anionic Surfactants, S3 — Nonionic Surfactants, S4 — Cationic Surfactants, S5 — Amphoteric Surfactants. For further information contact: Mr A. W. B. Cowan, BACS, John Marshall Street, 246-254 High Street, Sutton, Surrey, SM1 1PA.

New noise at work regulations

New regulations on noise at work have been welcomed today by Patrick Nicholls, Parliamentary Under-Secretary of State for Employment, as "a firm foundation for a European programme". The regulations which come into force on 1 January 1990 are designed to protect people at work from risk of hearing damage caused by noise. Together with existing health and safety legislation they will enable GB to meet the requirements of European Community Directive 86/188/EEC. "The Noise at Work Regulations 1989", SI 1989 No.1790, ISBN 0 11 097790 4, are available from HMSO and booksellers price £1.65.

Deb, hygiene specialists, has published a free 6pp leaflet on COSHH.

For further information Enter L206

Institute for Scientific Information has published a leaflet on their Index to Scientific and Technical Proceedings.

For further information Enter L207

Meetings

Environment and Safety

A two-day conference and exhibition on 'Environment and Safety' will be held on 10-11 January 1990 at the New Connaught Rooms, London WC2. The conference will have a 2-day COSHH Update symposium and a simultaneous Environment programme. The conference is organised by the International Professional Association for Environmental Affairs. For further information on the: Conference call 01-892 3637, Exhibition call 0727 31337.

BCDTA 'Diamond Awards' Dinner

A new highlight in the business calendar of chemical traders and distributors makes its debut next year when the BCDTA (British Chemical Distributors and Traders Association) stages its inaugural awards dinner to recognise the Industry's outstanding achievers. The 'Diamond Awards Dinner', will be held on Wednesday 21 February 1990 at the Crowne Plaza Hotel, Manchester, when over 400 guests are expected. For tickets (£40 + VAT) contact: Colin Wainwright, The BCDTA, Suffolk House, George Street, Croydon CR0 0YN, UK.

Central European Coatings Show

Polymers, Paint Colour Journal is staging its first Central European Coatings Show from 26-29 March 1990 at the Atrium Hyatt Hotel in Budapest, Hungary. The exhibition will present Paint, Printing Ink and Adhesive manufacturers from the COMECON countries, as well as Austria, Yugoslavia, Northern Italy, Greece, Turkey and the Middle East with the opportunity to meet manufacturers and suppliers of raw materials used in the manufacture of these products. The exhibition will be accompanied by a two and a half day conference, scheduled for the 26-28 March 1990. For further information contact: PPCJ, Queensway House, 2 Queensway, Redhill, Surrey RH11 1QS, UK.

People

BASF Coatings & Inks Ltd

BASF announces the following top management changes at BASF Coatings + Inks Limited, with effect from 1 January 1990:

Geoffrey Watson, Managing Director, becomes Deputy Chairman, with particular responsibility for the company's relationship with large international customers, in close co-operation with other European operations of BASF Lacke + Farben AG, its parent company

Arvid Lillesund, presently Managing Director of BASF Denmark, becomes Managing Director responsible for all UK operations of BASF Coatings + Inks Limited. Mr Lillesund is a Norwegian national, who graduated in Economics from Mannheim University in 1967, and joined the BASF Group in the same year.

Croda Board appointments

Barry Alcock, aged 30, has been appointed Operations Director, Glenn McAuliffe, aged 32, formerly of PPG, has been appointed Technical Director, and replaces John Cornish who recently retired.

Degussa Limited

Ulrich Kunze — formerly head of the business area Precious Metal Products of Degussa AG has been appointed Managing Director of Degussa Limited in Great Britain.

Shackell, Edwards

Derek Markham has joined Shackell, Edwards & Co Ltd, printing ink manufacturers, as Deputy Managing Director.

Hickson

Alan Nelson has been appointed chairman of the Surface Coatings Division of Hickson International PLC and Alan Mellor, formerly Director and General Manager has been appointed Managing Director.

Film formation by emulsion paints

by J. W. Nicholson, Materials Technology Group, Laboratory of the Government Chemist,
Queen's Road, Teddington, Middlesex TW11 0LY, UK

1. Introduction

In order for emulsion paints to form satisfactory coatings having properties of adhesion and integrity, a number of things are necessary. Firstly, the wet formulation has to be capable of being spread evenly across the substrate and of remaining spread without developing faults such as crawling or pinholing. Secondly, the formulation must be able to be converted from the liquid to the solid state, still without introducing film faults or reducing adhesion. And finally, once into the solid state, the film must remain coherent and retain adhesion. The present article examines these topics as they affect the way in which emulsion paints can be applied and used in the modern world.

2. Emulsion paints

Emulsion paints are not formulated from true emulsions, this term strictly referring to a dispersion of one immiscible liquid in another. They are more correctly described by the term "latex", which means a dispersion of polymer, generally a rubbery material, in a liquid phase that is usually water¹.

Latex paints are generally of pale colour², the main pigment being titanium dioxide³. This pigment requires very different dispersing aids from a typical polymer latex. Despite this, there have been very few studies on the combined colloidal behaviour of pigment and latex in the presence of their respective dispersing agents. Indeed, the assumption has generally been made that since pigment dispersants are generally negatively charged, they will have negligible effect on the stability and behaviour of a negatively charged latex⁴. However, a study by Jaycock and Kayem⁴ a few years ago provided evidence that cast considerable doubt on the validity of this assumption.

They examined a model system that consisted of a vinyl acetate homopolymer latex with sodium dodecylsulphate, SDS, as its dispersant, together with a rutile titanium dioxide pigment surface treated with alumina, whose dispersant was sodium hexametaphosphate, SHMP. Briefly, they found that the effect of the latter dispersant was to flocculate the latex, while the dispersing agent for the latex itself, SDS, tended to solubilise the latex. The overall conclusion from this work was that for good paint storage stability, the SHMP concentration should be kept as low as possible consistent with good pigment dispersion, and the SDS concentration should be kept as low as possible in order to avoid problems associated with the adsorption of surfactant onto the latex⁴. These problems included progressive hydrolysis of ionic groups in the polymer molecules of the latex, leading to instability of the latex and flocculation of the polymer particles. These results show the importance of a proper understanding of the complex interactions that take place in practical emulsion paints, and that care must be exercised in formulating these systems.

3. Film formation by "emulsion" paints

Film formation and drying are more complex for these paints than for paints derived from true solutions, whether or not the solvent is water. The initial stage, once the film has been spread, is evaporation of the water. This early drying is controlled by the vapour phase diffusion of the water⁵, and

when it is complete, polymer and pigment are left behind, together with certain minor constituents of the paint formulation.

In order for the film to give an acceptable level of performance, in the second stage of drying the polymer particles need to flatten and coalesce^{1,6}. This process also needs to occur in such a way that pigment particles become fully incorporated into the final film. The readiness with which the second stage of film formation occurs is dependent on a number of factors, including:

- (i) the natural hardness of the polymer,
- (ii) whether or not appropriate coalescing solvents are present in the formulation, and
- (iii) the ease with which the electrostatic repulsive forces at the surface of the polymeric particles, introduced by the presence of dispersing agents, can be overcome.

All of these factors are themselves dependent on the temperature at which film formation is to take place. Hence it follows that the ease with which this kind of paint will give satisfactory films varies with the prevailing temperature. Moreover, there is a minimum temperature below which films do not form satisfactorily. Under these conditions, the second stage of film formation simply does not happen, and the loss of water by evaporation just results in a powdery finish having no cohesion. Thus, it is important to formulate emulsion paints in such a way that they are known to be capable of forming satisfactory, continuous coatings under the anticipated conditions of use.

3.1 Minimum film-formation temperature

The minimum film formation temperature, MFFT, is influenced by a number of factors, and is not a property of the polymer phase only, but of the entire latex formulation. It is related to the glass transition temperature, T_g , of the polymer component, and is affected by the same molecular features. For example, the inclusion of "soft" monomers is found to lower both T_g and MFFT⁷.

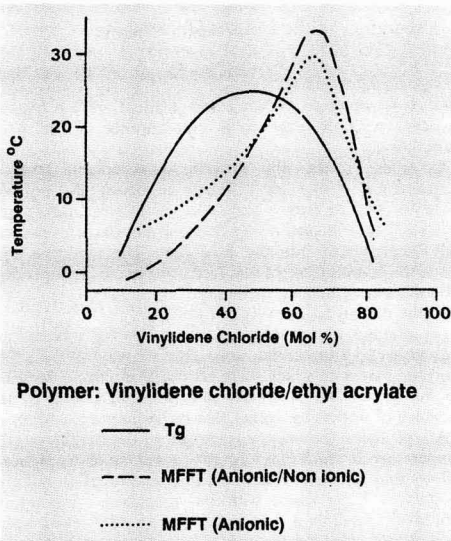
Despite this similarity, the relationship between T_g and MFFT is complex, and varies with copolymer composition. Ellgood⁸ has studied this relationship for various latices prepared from copolymers of vinylidene chloride, VDC. The variation in T_g and MFFT with composition is shown in Figure 1, for example, for a series of latices made from ethyl acrylate with VDC. Two series of latices were prepared, each having a different type of dispersing agent, and this, too, had an influence on the exact value found for MFFT.

Overall, it can be seen that there were considerable differences between T_g and MFFT. The maximum in T_g occurred at 50 mol% VDC, whereas the maximum in MFFT occurred at 65 mol%. Moreover, from about 55 mol% VDC, MFFT was found to be higher than T_g , whereas below that value, T_g had been found to be higher.

3.2 The process of coalescence

Coalescence of the polymeric phase of the latex is the crucial step in the formation of satisfactory films by emulsion paints. Over the years, a number of theoretical mechanisms have been advanced to explain this process, though it is still not completely clear which of them is really important in the practical application of paints made from polymer latices⁹.

Figure 1
Relationship between MFFT and T_g .



These various mechanisms will be briefly reviewed in the sections that follow.

3.2.1 Sintering: The mechanism based on sintering was first proposed by Bradford *et al* in 1951¹⁰, and elaborated in a further paper two years later¹¹. Briefly, this process was held to occur following the complete evaporation of the water. This was assumed to force the polymer particles into intimate contact with each other, after which viscous flow of the polymer molecules, aided by the desire to minimise the surface energy, was assumed to cause sintering to occur. High surface tension of the polymer would be expected to favour this process and thus provide the driving force, but high viscosity and large particle size within the parent latex would oppose it.

There have been criticisms of this mechanism^{9,12}. The main one is that there is not generally any evidence that film formation by coalescence occurs *after* water evaporation has taken place. Instead, before water loss is complete, there comes a stage at which both processes appear to occur simultaneously¹². On the other hand, there is evidence from electron microscopy studies of latex films that polymer particles do establish contact and then fuse together; thus sintering may yet have some importance in the formation of films by practical emulsion paints.

3.2.2. Capillary theory: A mechanism of coalescence based on capillary action was proposed by Brown in 1956¹². It arises from the observation that after a significant amount of the water has evaporated from the film, the polymer particles left behind form first a network structure of essentially undistorted spheres, and then a fused and continuous polymer film.

Brown went on to consider the forces acting on the initial latex particles, and concluded that in film formation, the main forces were:

(i) that of capillary pressure arising from the negatively curved surface of the water in the spaces between the particles. This was designated F_c .

(ii) the resistance of the spherical polymer particles to deformation, designated F_G .

In order for films to be formed, the capillary force must

overcome the resistance to deformation of the particles, hence the condition for film formation is:

$$F_c > F_G$$

Brown considered further the nature of the force resisting the deformation, F_G , and related it to the magnitude of the time-dependent elastic modulus of the polymer, G_t , which itself arises from the viscoelastic nature of the polymers used⁹. Relating G_t to the capillary forces involved enabled Brown to redefine the condition for film formation as

$$G_t < 35\delta/r$$

where δ is the surface tension of the polymer-water interface, and r is the radius of the polymer spheres.

Experimental work has been directed at testing whether Brown's condition, which can be rewritten as $G_t/r/\delta < 35$ applies at film formation, and in a number of cases it has been found to. For example, Brodnyan and Konen¹³ showed that a latex of homopolymer n-butyl methacrylate gave a value of 8.5, and would readily form films. By contrast, however, a latex copolymer consisting of 50% ethyl acrylate/50% methyl methacrylate, also formed films readily, yet had a $G_t/r/\delta$ value of 260, well above Brown's maximum of 35. Overall, then, there does seem to be something in the concept of capillary attraction being a driving force for film formation in these systems, but there are exceptions, and it is again by no means clear precisely how important this mechanism is in film formation by practical emulsion paints.

3.2.3. Mutual diffusion: Neither of the preceding mechanisms takes account of the action of dispersant at the particle surface, or of the observation that actual films are not completely homogeneous. These were taken into account, though, in formulating a mechanism of coalescence based on mutual diffusion, as developed by Voyutskii¹⁴. Briefly, the process was envisaged as comprising three steps:

(a) Initial evaporation of the water leading to close packing of polymer particles, and increasing concentration of dispersant in the remaining aqueous phase.

(b) Increasingly close approach of polymer particles, leading to deformation and loss of spherical morphology. This step may be driven either by the force of surface tension or by capillary pressure, as outlined in the preceding sections, but these are not thought to be important in the final step, which according to Voyutskii is the most important of the three.

(c) With increasing water loss, the dispersant begins to move away from the polymer-liquid interface, and either dissolves in the polymer phase, or forms aggregates between the polymer particles. This latter process is favoured when the dispersant is insoluble in the polymer, and its occurrence has two effects. First, the film would become heterogeneous, as is observed experimentally, and second, as the dispersing agent is removed from the polymer surfaces, so these surfaces cease to repel each other. This enables the particles to come into closer contact with one another, and at the areas of contact, the polymer molecules are able to diffuse across the erstwhile boundary, thus bringing about coalescence.

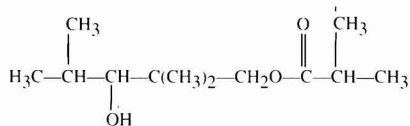
This proposed mechanism would predict that the degree of coalescence should depend not only on the nature of the polymer and the casting conditions, but also on the nature of the surfactant. As was shown earlier for VDC latices, such a result is indeed found experimentally⁸. It has also been confirmed in studies of film formation using model polyvinyl acetate latex¹⁵.

In this study, it was shown that stabilisation of the latex by sodium hexametaphosphate resulted in heterogeneous films exhibiting clear evidence of polymer flocculation when examined by scanning electron microscopy. By contrast, the dispersant sodium dodecyl sulphate was found to allow the formation of smooth homogeneous films, showing no sign of flocculation.

3.2.4 General conclusions on the mechanism of film formation: As has been shown in the foregoing sections, there have been a number of theories put forward to explain the phenomenon of coalescence in latex-based coatings. However, it has not been possible to choose between them. Indeed, it may not be possible to produce a general theory of coalescence that is applicable to all latices; more probably, these various mechanisms contribute to different extents, depending on the precise formulation of the latex. A variety of monomers are used to prepare latices for commercial paints, and each is known to make a specific contribution to such factors as durability, hardness and water resistance¹⁶. It is highly probable that they also alter the relative importance of the various mechanisms of coalescence. Moreover, certain modern latices, such as the so-called "Haloflex" resins developed by ICI, which are based on vinylidene chloride¹⁷ and designed as a vehicle for novel waterborne anticorrosive paints, are formulated to contain a reduced amount of surfactant compared with conventional latices. Colloidal dispersion in these latices is maintained mainly by the charged end groups in the polymer molecules, and this means that coalescence by mutual diffusion is less likely than in latices prepared from uncharged molecules. For latices prepared from charged molecules, the other mechanisms of coalescence might be expected to predominate. Certainly films prepared from them have been found to have low permeability to water and oxygen¹⁷, a fact that confirms that coalescence into coherent films unquestionably does occur for these materials.

3.2.5. Internal stress in latex films: As coalescence and loss of volatiles occurs in a film spread from a latex, so an internal stress develops within the coating¹⁸. This can lead to various problems, including filler/binder dislocations, the growth of microfissures, and the loss of adhesion, all of which affect the film integrity. Internal stress and its effects on film formation in emulsion paints has been studied extensively by Perera^{18,19} who has used a stressmeter to measure the development of this internal stress. The basis for the measurements was the extent of curvature that is found to occur when latex paints are coated onto very thin stainless steel shims. Measurement of the deflection, together with knowledge of the elastic properties of the substrate, enabled the internal stress to be calculated¹⁸.

A number of formulation variables have been found to influence internal stress, including extent of pigment loading and the presence and nature of organic "coalescing" solvents. For pigmented films above the critical pigment volume concentration, Perera found that there was a rapid build up to a maximum internal stress, followed by a sharp decrease. This decrease was assumed to occur by various relief mechanisms operating in the coating, including the formation of microfissures and dislocations at the boundary between pigment and binder. For films prepared from formulations that included coalescing solvents, generally comprising oxygenated molecules of the ether-ester type, the development of internal stress was found to be dependent on the level of solvent within the film. For example, a film with no coalescing solvent was poor, since it underwent mudcracking shortly after being applied to the substrate. By contrast, the presence of 5% of the solvent "Texanol" in the film allowed relaxation and stress relief processes to occur in such a way that film integrity was maintained. Texanol has the structure:



For this system, it was found that internal stress reached a steady state value after a few hours, and did not change much for the following three months over which measurements were made. Similar results were found with 10% Texanol, although the steady state value of internal stress was lower than at 5%, and this proved to be the optimum level of solvent. Raising the concentration to 15% led to an increase in steady state internal stress¹⁸.

Overall, Perera's work has demonstrated a further aspect which needs to be understood and controlled in order for satisfactory film formation to occur in emulsion paints, namely development of internal stress. Care is clearly needed in the formulation of the wet paint in order to minimise the magnitude of the steady state internal stress which is able to develop in the dry film.

4. Conclusion

In this article, important aspects of the formation of acceptable films from emulsion paints have been discussed. As a class, waterborne coatings are continuing to grow in commercial importance, and this growth seems likely to be sustained in view of the environmental acceptability and cost-effectiveness of using water as a paint solvent. Consequently, growing understanding of the topic of film formation by emulsion paints will be necessary in order that these coatings may be employed with maximum effectiveness.

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Third generation waterborne urethanes

by P. L. Jansse, Product Development Laboratory, ICI Resins bv, Sluisweg 12, 5140 AC Waalwijk, Holland

During the last decade, ICI Resins has played an important role in the area of aqueous urethane dispersions. Environmental and governmental pressure have caused an important shift from solvent based systems to waterdispersed systems.

New product types, with novel properties, have been developed within ICI Resins in the last two years.

This paper describes three new binder systems which will find use in existing and new areas of application.

"NeoRez" Precrosslinked waterdispersed urethanes

Summary

Incorporation of multifunctional isocyanates in order to precrosslink waterdispersed urethanes, offers new or improved properties with respect to solvent resistance and metal protection. A defined balance of hard and soft segments in the polymer backbone is needed in order not to increase the minimum film-forming temperature.

Introduction

During the last decade, waterdispersed urethanes have found application in a wide variety of industries. They have been shown to be very suitable for coatings on wood, plastic, metal and mineral substrates, either alone or combined with acrylic. In many cases, however, the use of an aziridine-based multifunctional crosslinker is necessary in order to obtain properties that can compete with two component systems. If one, however, incorporates up to 20% of a multifunctional isocyanate into the polymer backbone, waterborne polyurethanes can be synthesized that can rival two-pack urethanes in chemical resistance without crosslinking.

There are problems, however, with precrosslinked dispersions in maintaining good coalescence. This paper describes approaches to overcome these problems.

An important parameter for viscosity control of the dispersion is the molecular weight per crosslink which is defined as the ratio:

$$\frac{\text{total weight of solids} \times \text{functionality of crosslinker}}{\text{the number of isocyanate group equivalents}}$$

Values of 4000 or more will result in urethane dispersions that show good oven stability (52°C). In this paper, results of products developed by this new technology are described.

Results

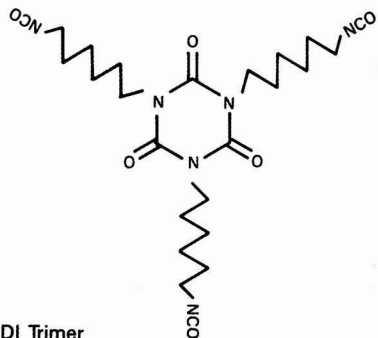
Precrosslinked urethane dispersions exhibit their excellent properties when the ratio hard and soft segments allows easy coalescence of the multifunctional isocyanate modified urethane dispersion particles. The technology only applies to urethane compositions that exhibit a low (4°C) minimum filmforming temperature. A high content of hard segments mainly determined by the number of short diol moieties in the molecule, will not allow the urethane film to properly coalesce. A hard segment content of less than 50% by weight will not influence the self-coalescence. Various types of these isocyanates are possible (Scheme 1), of which their choice depends on price, ease of handling, toxicity and reactivity of their isocyanate groups. Traditionally, when designing a polyurethane in water, branching can either be obtained by introducing triols (Scheme 2) directly into the backbone during prepolymer synthesis, or one can use multifunctional amines as chain extenders. Both methods have the disadvantage of obtaining high viscosities or even the chance of gellation of the final dispersion. An efficient way to overcome this problem but still have crosslinked polyurethane films after coalescence is the addition of multifunctional isocyanate after the preparation of the

Scheme 2

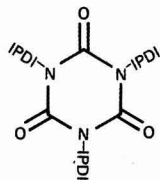
Types of one component crosslinkers.

	Advantage	Disadvantage
<p>Triols in Prepolymers</p> $\begin{array}{c} \text{CH}_2\text{OH} \\ \\ \text{CH}_3\text{CH}_2 - \text{C} - \text{CH}_2\text{OH} \\ \\ \text{CH}_2\text{OH} \end{array}$	Cheap Controlled Crosslinking	Thick Polymers 20,000 mw/cr
<p>Triamines as Extenders</p> $\begin{array}{c} \text{H} \\ \\ \text{H}_2\text{NCH}_2\text{CH}_2\text{NCH}_2\text{CH}_2\text{NH}_2 \end{array}$	Low Viscosity Prepolymer Cheap	Inefficient Crosslinking

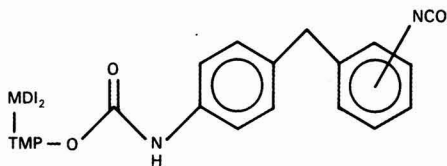
Scheme 1
Triisocyanate crosslinkers.



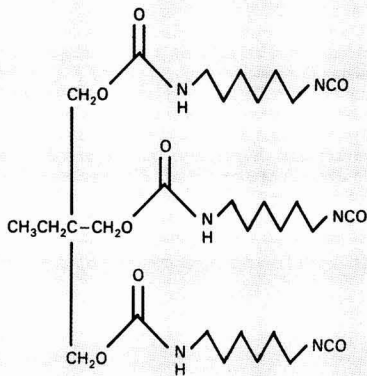
HDI Trimer



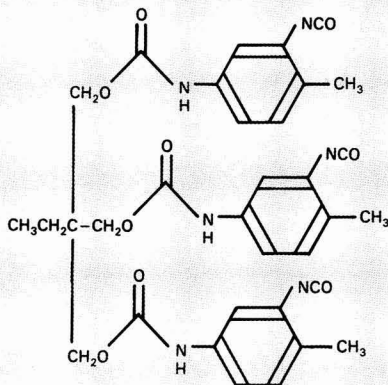
IPDI Trimer



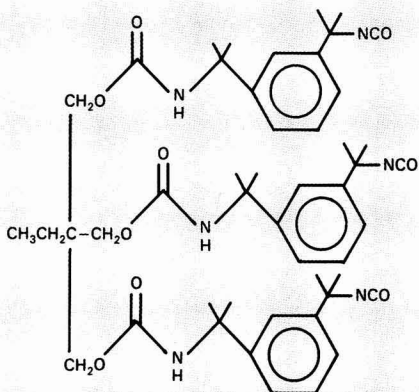
2,4':4,4'-MDI Adduct



HDI Adduct



TDI-TMP Adduct



TMXDI TMP Adduct

isocyanate terminated prepolymer. The second step in the process is essentially the same as for conventional polymers, i.e. neutralisation followed by dispersion and chain extension, either afterwards or simultaneously. Figure 1 gives the effect of the presence of crosslinker on König Hardness and % elongation at break for an aromatic diisocyanate based polyurethane dispersion.

The use of 5% multifunctional isocyanate also results in a great improvement in corrosion resistance after 28 days of humid ageing, when no whitening of the film or rust occurs.

For urethane dispersions based on an aliphatic

Figure 1
The effect of precrosslinking on hardness and elongation at break for an aromatic urethane.

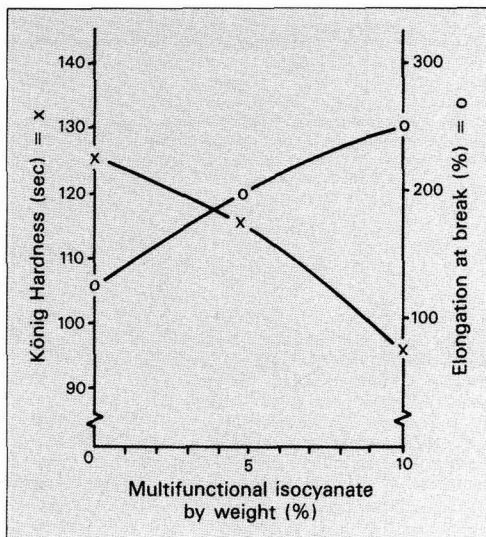
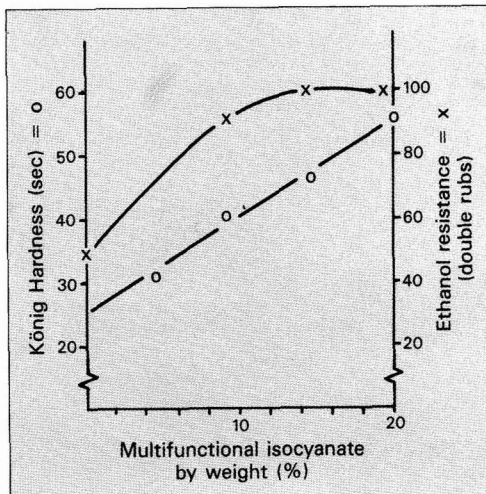


Figure 2
The effect of precrosslinking on hardness and ethanol resistance for an aliphatic system.



diisocyanate, the effect of precrosslinking on alcohol resistance is significant. Figure 2 shows the change in alcohol resistance when precrosslinking is applied. Also the increase in König Hardness is shown.

The data given in Figures 1 and 2 represent typical properties for urethanes which form films at low temperatures (4°C) and are precrosslinked up to 20% by weight.

Conclusions

The use of precrosslinking technology to improve the chemical resistances of aqueous urethane dispersions is a very useful tool. Various polyisocyanates can be used for this purpose.

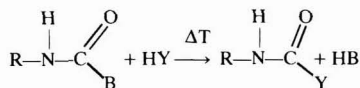
"NeoRez" Blocked waterdispersed urethanes

Summary

Waterdispersed urethanes of different molecular weight, having terminal or pendant blocked isocyanate groups find their way into the marketplace where the use of solvent based systems has become unacceptable. A range of isocyanates and blocking agent was investigated and the deblocking temperature and hydrolytic stability studied.

Introduction

Wicks^{1,2} has extensively reviewed blocked isocyanates. These materials can be regarded as isocyanates that have been reacted with a compound that traditionally reacts with isocyanates, but which will prevent the reaction of the isocyanate group at room temperature. Elevated temperatures between 120 and 240°C will make the bond between the isocyanate group and the blocking agent labile, allowing the liberated isocyanate group to react with a second component that carries active hydrogens. This overall reaction can be expressed as follows:



and can proceed through two possible mechanisms: elimination-addition or substitution. Most of the reactions as described in earlier work took place in a low VOC or solvent free reaction environment. The reaction mechanism of the curing reaction of a waterborne blocked polyurethane is, in essence, not much different from a solventborne system. However, other factors come into play such as dispersion stability, film formation, pigmentability and compatibility with water dilutable second components.

In particular the stability of the dispersion is critical because it depends on the molecular weight and the sensitivity of the isocyanate-blocking agent bond towards hydrolysis. Here lies a dilemma for low temperature curing waterborne systems. Aromatic blocked polyurethane dispersions in water tend to hydrolyse quickly whereas at the same time, low deblocking temperatures are observed for such systems. Our present work is directed towards the development of hydrolytically stable waterdispersed blocked polyurethane systems which cure upon heating at temperatures of 150°C or less.

Results

Water-dispersed systems which contain blocked isocyanate

functionality with or without the presence of a compound that is reactive towards the isocyanate group, can be synthesized in several ways. One method is to obtain an isocyanate terminated prepolymer containing acid functionality (anionic), which then is blocked, followed by dispersion in the water of the neutralized blocked prepolymer. These waterdispersed oligomers can also be combined with hydroxy functional high molecular weight acrylic or urethane aqueous dispersions which form a "self-reactive" system on their own. While the first and second system remain difunctional blocked urethane molecules, a third approach results in pendant blocked isocyanate groups. The latter is achieved by incorporating a short diol containing a blocked isocyanate functionality. Agnostou and Jaul¹ have reported that the use of differential scanning calorimetry (DSC) is a very useful tool to study the temperatures at which deblocking of the masked isocyanate group occurs. The method provides the opportunity to select a diisocyanate-blocking agent combination that has favourable deblocking characteristics. Various adducts of commercially available diisocyanates and blocking agents were prepared in ethylacetate or in methoxypropanolacetate from which the adducts are precipitated directly after preparation or upon cooling to -5°C in an ice/salt bath. Evaluation of the various adducts by means of DSC confirmed earlier findings¹ that aromatic systems tend to deblock at a lower temperature than their aliphatic analogues. This is especially the case with adducts of toluene diisocyanate. This effect is assumed to be caused by the reduced basicity of the urethane group for such aromatic systems, caused by a charge delocalisation effect.

Table 1 summarizes the most important DSC-results of the various adducts.

Table 1

Deblocking temperatures of diisocyanate/diadducts

Diisocyanate	Blocking agent	Deblocking temperature (°C)
Toluene diisocyanate (TDI,80/20 2,4'/2,6'-isomer)	2-pyrrolidone	221
	imidazole	155
	acetoneoxim	120
	methylethylketoxim	118
4,4'-diphenylmethanediisocyanate (MDI)	E-caprolactam	182
	methylethylketoxim	145
	acetoneoxim	130
4,4'-dicyclohexylmethane diisocyanate	methylethylketoxim	140
	acetoneoxim	136
isophorone diisocyanate (IPDI)	acetoneoxim	143

From Table 1 it can be especially seen that oxim blocked aromatic diisocyanates would offer a very attractive route to the synthesis of aqueous coating systems that cure at temperatures of 130°C or less. Unfortunately we have found that these systems do not exhibit sufficient hydrolytic stability in the pH range of 7.5-9.0.

The use of blocked isocyanates derived from 4,4'-dicyclohexylmethanediisocyanates offers the compromise of hydrolytic stability and reasonable reactivity.

At ICI Resins, two systems blocked with 4,4'-dicyclohexylmethanediisocyanate containing mekoxim blocked isocyanate functionality have been developed. They both have found their way in waterborne stoving finishes.

Conclusions

Blocked urethanes can be easily obtained in water. The type of blocked isocyanate functionality and blocking agent determines the lability of the bond between the isocyanate

and blocking agent. Hydrolytic instability was observed for aromatic systems.

"NeoPac" urethane acrylates

Summary

Intimate combinations of urethane and acrylate were obtained by means of emulsion polymerization of acrylic monomers in a urethane environment. It was found that the urethane component dominated the final dispersion particle, therefore most of the urethane properties such as resistance to solvents and selfcoalescence were retained. A competitive price is achievable because of the presence of a significant amount of acrylic polymer.

The work in this manuscript builds on earlier technology of the synthesis of urethane-acrylate graft copolymers.

Introduction

The combination of a waterborne urethane dispersion and an acrylic emulsion has always been a suitable method of obtaining some urethane properties at reduced raw material cost, though compromises have to be made. Even today, well defined customer needs are translated into urethane-acrylic blends, based on the separate components, which have very acceptable properties at a competitive price. Our approach to the development of urethane-acrylates as a single system provides a number of great advantages in terms of: a) less water sensitivity because the emulsifier is not needed; b) improved stability because the ionic urethane is a powerful stabilizing agent; c) chemical resistance because the morphology of the particle is such that the urethane properties dominate and d) environmental acceptability because only a little cosolvent is needed. Apart from these physical features, there is a practical advantage in having a one-pot system that is directly ready for use. The work described in this paper provides information on properties and polymer design characteristics.

Results

The synthesis of urethane-acrylate polymers having improved properties with respect to blends begins with the preparation of a stable aqueous urethane dispersion in which the cosolvent is replaced by acrylate monomers. Because all acrylate monomers are essentially insoluble in water, the bulk of the material will be located inside the urethane particle, providing anionic stabilization through the neutralized carboxyl group of the polyurethane chain. A subsequent emulsion polymerization using this urethane dispersion as the stabilizing system for additional monomer which is gradually fed, yields an intimate combination of an acrylic particle that, we speculate, is entirely surrounded by the anionic urethane outer shell.

Water sensitivity of such systems is very low because of the absence of hydrophylic emulsifier. Self-coalescence and urethane-like properties are only achieved when there is a correct balance between the minimum film forming temperature of the urethane and T_g of the chosen acrylic monomer composition. Urethane backbones having too much hard segments, like short diols or cyclohexyl rings do not offer the desired film formation. Examples of selfcoalescing systems are given in Figures 3 and 4. The compositions are based on IPDI-polypropyleneglycol-styrene-MMA and BA. They can easily withstand a hundred double MEK-rubs and remain filmforming at low temperature with increasing T_g of the acrylic part.

Continued on p.484

New proposed environmental legislation for UK ink manufacturers

by M. G. Bigg, Principal Inspector, HMIP/Local Authority Unit, Her Majesty's Inspectorate of Pollution, Romney House, 43, Marsham Street, London SW1P 3PY, UK

Introduction

'Stable prosperity can be achieved throughout the world provided the environment is nurtured and safeguarded', Margaret Thatcher, speech to the Royal Society, September 1988.

Safeguarding the environment is critical, not only for our sake but also for our children and the future.

The Bruntland Report 'Our Common Future' published in 1987, when discussing strategies for sustainable industrial development stated 'resources and environmental considerations must be integrated into the industrial, planning and decision making process of government and industry'.

Recent legislation and proposals for further legislation have repercussions for both printing ink manufacture and much of British industry. A systematic procedure has been put forward for the protection of the environment across land, water and air.

In this paper I will try to explain the current situation and the effects of proposals for the future as far as they could affect printing ink manufacture.

As Prince Charles said at a recent environmental conference in London 'by now the man in the street has begun to get the message . . . We now know that we are all in this together, unlike anything else before'.

Setting the scene

With particular reference to air pollution control there has been a series of laws and regulations, starting with the original Alkali Act in 1863, followed by further Alkali and Clean Air Acts, through to the Health and Safety at Work etc Act 1974, and the Control of Pollution Act, 1974. These two acts are the basis of much of our current national pollution control legislation. In the local authority sector pollution control is also based on the Public Health Act 1936 and the 1897 act for Scotland.

Generally much of the legislation has been reactive, either to control specific new processes or emissions as problems have arisen, or, in the case of the Clean Air Acts, as a direct consequence of the major smogs of the 1950s.

Similarly, those responsible for implementing the legislation have changed their titles and responsibilities. The Alkali Inspectorate gave way to the Alkali and Clean Air Inspectorate followed by the Industrial Air Pollution Inspectorate, which was part of the Health and Safety Executive. Eventually on 1 April 1987, Her Majesty's Inspectorate of Pollution was established within the Department of the Environment. It brought together:

- (i) Her Majesty's Industrial Air Pollution Inspectorate
- (ii) Her Majesty's Radiochemical Inspectorate
- (iii) The Hazardous Waste Inspectorate
- (iv) A new Water Pollution Inspectorate

Current developments

On 28 June 1984 the EC Council Directive on 'The Combating of Air Pollution from Industrial Plants' was adopted. The broad requirements of the directive are met by the current practices of HMIP and the processes identified in the directive are already included in the schedules of registrable works.

The Directive required implementation by 30 June 1987 and enactment was anticipated in the first consultation published by the Department of the Environment in

December 1986 entitled 'Air Pollution Control in Great Britain: Review and Proposals'. Due, however, to the desire to consult further, two pieces of legislation have been enacted. A further regulation had been introduced to implement formally European Air Quality Standards.

- (i) The Health and Safety (Emissions into the Atmosphere) (Amendment) Regulations 1989.

These Regulations amended the categories of works under the control of the air pollution provisions of the Health and Safety at Work etc Act 1974 and the substances that are to be treated as noxious or offensive.

- (ii) The Control of Industrial Air Pollution (Registration of Works) Regulations 1989.

These Regulations will implement the requirement of the Directive for providing a more open and visible system of control. Whenever a new scheduled works or substantial modification to any existing scheduled works is proposed, the operator has to make a formal application which is also advertised in at least one local newspaper in each of two successive weeks. Opportunity is provided for the public to comment. The system is similar to that pertaining to the discharge of liquid effluents to surface water. Registrations are recorded in the register held and available for public inspection and copying at the offices of the local inspectorate and local authority.

Existing scheduled works will continue under the current legislation. It is intended that the terms of the registrations will themselves become legally enforceable conditions within the next 12 months or so.

In addition to the requirement to be registered there remains a general duty on the plant operator to use the 'Best Practical Means (BPM)' in respect of any other aspect of his operations not specified in the conditions of the registration insofar as they are relevant to the control of emissions to air. The measures include provision for aspects of any registration to be kept in confidence for commercial or national defence reasons.

- (iii) The Air Quality Standards Regulations 1989

These regulations set the EC limits and placed a duty on the Secretary of State to monitor air quality and take action to remedy any breaches.

Future developments

Following the feedback on the consultation paper in 1986 a second consultation paper on 'Air Pollution Control in Great Britain, Works Proposed to be Scheduled for Prior Authorisation' was published in December 1988.

Both consultation papers recognised that local authorities needed new powers to control a middle range of industrial processes which do not warrant central control because they are neither potentially gross polluters nor technically difficult to control, but which can nevertheless give rise to significant local pollution. The consultation papers proposed a better and more cost effective control system for these industries than is available under the current mix of Clean Air Acts and the Public Health Acts, which normally force the local authority to wait for a nuisance to develop before it can act to remedy it. The Government also wishes to provide an open and more publicly visible system of control.

As everyone is probably aware the list of scheduled processes is proposed to be split into a Part A and a Part B. The larger, technically more complex processes are in Part A for control by the National Inspectorate. The smaller, more numerous, less complex processes are in Part B. They will be regulated by local authorities exercising the same prior approval and enforcement roles as currently exercised by HMIP Inspectors.

IPLA

In the 1986 consultation paper it was recognised that with the addition of Local Authorities as enforcing authorities, positive co-ordination between the enforcing authorities was required. Regular liaison would be important to facilitate co-operation in ironing out local problems, to exchange experiences, to help develop an overview of air quality in each area and to ensure consistency in enforcement policy and practice. This is particularly important considering that there are theoretically about 44 air field inspectors compared to 457 local authorities in England, Wales and Scotland.

As a result the HMIP/Local Authority Liaison Committee (IPLA) was set up along the lines of the Health and Safety Executives HELA Committee. The committee is not a statutory body but does have an influential steering role. It aims to promote dialogue in exchange of information between HMIP and the local authorities. It also advises the Secretary of State on the need for and content of national notes on the Best Practicable Means and other guidance.

The Committee meets quarterly and includes representatives of the local authority associations, the professional environmental health institutions, Her Majesty's Inspectorate of Pollution, Her Majesty's Industrial Pollution Inspectorate (Scotland) and as observers the appropriate Government departments.

Local Authority Unit

This unit provides the secretariat for IPLA. The unit is responsible for the more detailed monitoring of the new arrangements and the production of central guidance for the local authority sector. It also provides appropriate input for the training of environmental health officers. The main aim of the unit is the production of notes on Best Practicable Means for the Part B processes.

The unit consists of a Principal HMIP Inspector, a seconded Principal Environmental Health Officer and appropriate support staff including a full-time secretary and an executive officer. The unit is co-located with the HMIP District Air Office in Birmingham.

Method of operation

The aim is to produce notes on the Best Practicable Means for all the Part B processes which will probably number about 40, by statute date for the new legislation. We are aiming to achieve a workable agreement between the local authority sector, industry and HMIP. It is planned that as the BPM notes are agreed and published so the local authorities should take over responsibility for each process.

From discussions with the industries proposed to come into Part B and the local authorities it is clear that there is a very strong driving force to obtain an agreement on the Best Practicable Means.

For the Printing Ink Manufacturing Industry we have followed the same procedure as with many other processes:

- (i) Exploratory discussions with industry, feedback on the consultation paper. In these discussions with the industry it was proposed that a category of works will be scheduled covering 'printing ink works': that is

plants in which organic solvents and/or pigments are used in the manufacture of printing inks.

It was agreed that there should be no bottom cut off figure for the scale of industry to be covered.

- (ii) Establish working party. The Printing Ink Works Working Party includes four representatives of the industry, a representative of a local authority and a representative of HMIP. One of the first actions of the working party was to agree which processes the notes actually referred to and it is proposed that they were identified as printing ink works in which:
Pigments, resins, oils and solvents are mixed and blended and subject to subsequent operations such as milling and dispersion to produce a final product.
After several meetings a first draft is agreed.
- (iii) First draft circulated to HMIP Inspectors and the Chief Inspector. First draft amended and confirmed.
- (iv) First approved draft formally sent to industry for discussion, and to IPLA members for comment.
- (v) Working party reconvened. Responses assessed, wider consultation and final draft produced.
- (vi) Final draft circulated to industry and IPLA members for comment and then to Chief Inspector for approval.
- (vii) Printing and issue.

This approach has been endorsed by the IPLA Committee as a means of ensuring the involvement and agreement of the local authorities who will be responsible for the implementation of this part of the legislation.

Criteria

In drafting an agreement on the Best Practicable Means the following issues need to be addressed and agreed.

- (i) The exact nature and scale of processes to be covered including where appropriate raw materials and products, eg. the handling of dusty pigments and the finished ink.
- (ii) The timescale to bring existing plants up to current and/or intermediate standards. Requirements are agreed for implementation on new plant but so as not to discourage investment in new plant a timescale has to be agreed for existing plants to reach those requirements as well.
- (iii) The frequency of view of the Part B Notes on BPM and the position at existing works.
- (iv) The frequency and scope of testing and monitoring. The practicability of testing has to be reviewed and the need for testing particularly on small plants agreed.
- (v) Emission limits and standard conditions for testing. The availability of technology to meet the arrestment requirements or the need to adopt alternative technologies must be assessed.
- (vi) Avoidance of emissions from ancillary operations. Practicability of controls and limits.
- (vii) Design and height of vents and/or exhausts.
- (viii) Maintenance, training, action in case of malfunction, housekeeping. Often good environmental control comes down to good housekeeping and although the central registrable process may be well controlled so often it is ancillary operations which let a company down.

Integrated pollution control

In July 1988 the Government issued a consultation paper which outlined proposals for integrated pollution control. The objectives of the proposals were:

- (i) To reflect the environment as it is—integrated, not separated;

- (ii) To produce a flexible system transparent in its operation;
- (iii) To streamline the regulatory system and to reduce the burden on industry;
- (iv) To improve the efficiency and effectiveness of HMIP, resulting in more efficient use of pollution control resources.

The proposals would give HMIP integrated pollution control over discharges of specified wastes to air, water and land for:

- (i) Processes in Part A of the December 1988 consultation paper;
- (ii) Processes discharging red list substances to water in significant quantities;
- (iii) Processes generating large amounts of special wastes.

These proposals will not generally affect Part B processes, the category under which it is proposed Printing Ink Works are likely to come, which would remain under the control of local authorities in respect of atmospheric emissions only.

Terminology

Throughout this paper I have referred to the approach of Best Practicable Means (BPM) as is currently required under the 1974 Health and Safety at Work etc Act which states that the operator of a registered works shall 'use the Best Practicable Means for preventing the emission into the atmosphere from the premises of noxious or offensive substances and for rendering harmless and inoffensive such substances as may be so emitted'.

In the 1984 EC Directive there is a requirement that 'all appropriate preventive measures against air pollution have been taken, including the application of the best available technology, provided that the application of such measures does not entail excessive costs'. This is usually abbreviated to 'Best Available Technology Not Entailing Excessive Cost (BATNEEC)'.

Conclusion

A whole new area is proposed to be covered by Air Pollution Control Legislation. Measures are expected shortly to set up the new two tiered system of control by the National Inspectorates and local authorities. By means of the establishment of IPLA and the LAU the introduction should be smoother and the consistency of enforcement ensured.

Much work has and is being done on the production of Part B Notes on BPM. Due to the breadth of processes to be covered, the contributions and support of the rest of HMIP, industry and local authorities are fully appreciated. I must express my thanks to the Society of British Printing Ink Manufacturers for their full collaboration. This is the first major review of Air Pollution Control Legislation in thirty years and it is only with the full commitment and agreement of industry that we are going to achieve lasting workable agreements. ■

Based on a presentation to the Society of British Printing Ink Manufacturers, 19 May 1989.

Jansse, Continued from p.481

Conclusions

Urethane-acrylates prepared via emulsion polymerization in a urethane environment provide a new route to stable cost-effective products in which the urethane properties dominate the final film. □ □

Figure 3
Effect of acrylic Tg on hardness for IPDI-polypropyleneglycol based urethane.

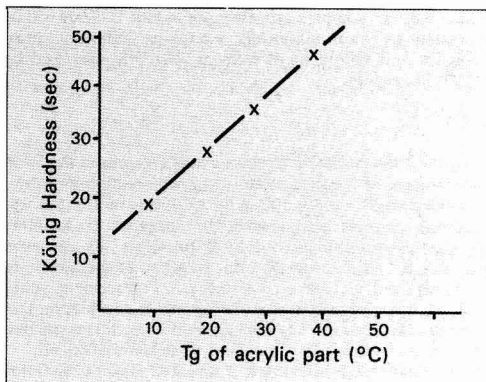
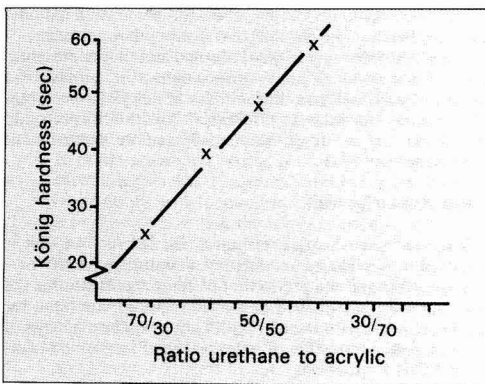


Figure 4
Effect of urethane to acrylic ratio on hardness; Tg of the acrylic part is 50°C.



Acknowledgement

Much of the work on precrosslinked waterdispersed urethanes described in this paper has been conducted by Dr R. G. Coogan at ICI Resins US, Wilmington, Massachusetts. A large contribution to the development of urethane-acrylate dispersions was undertaken by Dr G. C. Overbeek at ICI Resins bv, Waalwijk, Holland. I wish to express my thanks to these two colleagues whose kind cooperation played an important role in compiling this paper.

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The formulation of fluorescent coatings

by M. G. Martindill, Glowbug Ltd, Wilbet Cottage, Witham Road, Black Notley, Essex CM7 8NG, UK

Without doubt, the effect of a well formulated luminescent coating can be striking enough to make heads turn, whether this be on an advertising poster or a rescue vehicle. Of the luminescent colourants available to the coatings chemist, fluorescent are currently the most popular, and are likely to remain so for the foreseeable future. The worldwide demand for fluorescent colours over the last five years has increased exponentially (quite literally) so that most suppliers have found it difficult to keep pace. Confidence that this trend will continue upwards is demonstrated by the magnitude of investment in plant and machinery the manufacturers of fluorescent are committing themselves to.

The larger paint and ink manufacturers already include fluorescent in their shadecards and no longer consider them as specialist colours, but many others still consider fluorescent difficult to process and use, and are reticent to instigate development work. Often this is because of a less than complete understanding of the physical process of fluorescence, which is essential to any attempt at formulating effective fluorescent coatings.

Physics of fluorescence

Fluorescence involves the absorption and re-emission of light by photoactive atoms or molecules (Figure 1). Incident light of a frequency to which the receiving atomic systems are sensitive, is absorbed causing a redistribution of electrons into higher energy levels. This high energy state is intrinsically unstable and the electrons soon fall back to their original lower energy configuration emitting photons as they go. In practice a small amount of energy is lost internally before the emission, and the emitted light has lower energy, therefore lower frequency, than the incident light. For daylight fluorescence to occur, that is to obtain emitted frequencies in the visible spectrum, the absorption of higher frequency incident light is necessary and this may be long wave ultraviolet or daylight at the blue end of the visible spectrum. The time taken to complete the absorption/

emission cycle for daylight frequencies is around one hundred millionth of a second so, in effect, no afterglow is apparent.

By contrast, luminescence involving phosphorescent process depends on absorption/emission cycles of seconds or minutes giving rise to the familiar glow after the incident light is shut off.

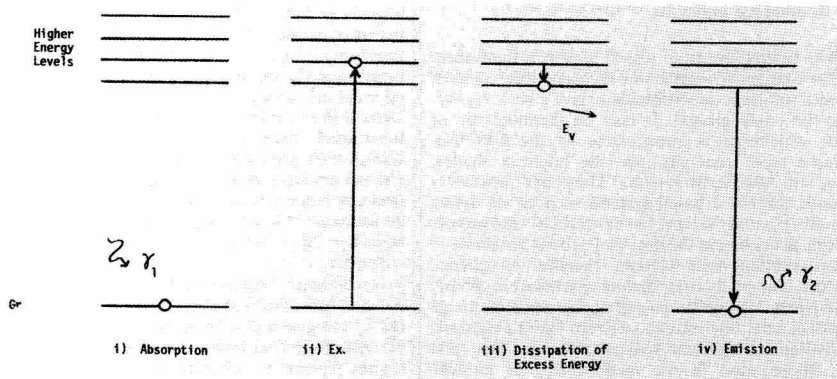
In conventional fluorescent coatings, chemically engineered heterocyclic dye molecules in solution act as fluorescing agents. So as to provide the very best conditions for efficient absorption of daylight or ultraviolet frequencies, and minimum quenching of the re-emitted daylight frequencies, the fluorescer should be uniformly dispersed at very low concentration in a highly transparent medium. Fortunately, the fluorescent pigment manufacturer has done most of the hard work for the coatings formulator by offering a solid solution of a blend of fluorescent dyes in a transparent resin matrix at the optimum concentration for efficient fluorescence to occur. In effect, the physical process of fluorescence is taken care of. The paint or ink formulator can treat the dyed resin, supplied as a fine powder ground to a particle size taking best advantage of the optical properties of the matrix, as a 'pigment' and disperse it into any medium or binder by high speed dispersion. The only care to be taken is not to undo the work of the fluorescent pigment manufacturer by impeding the absorption/emission cycle: overgrinding or overmilling the pigment will reduce fluorescence, as will a coating made virtually opaque by the addition of solid fillers, colourants or toners restricting penetration of incident light as well as the escape of emitted fluorescent light.

The initial steps overcome, several inherent advantages should become apparent to the formulator:

(1) The chemistry of the fluorescent pigment used is entirely dependent upon the resin matrix which, of course, is common to all shades. The formulator can rest assured that if a practical formulation is developed incorporating one fluorescent pigment, all other fluorescent pigments with the same resin matrix should behave similarly.

Figure 1

The process of fluorescence.



(2) The specific gravity of daylight fluorescent pigments is low compared to many ordinary pigments and is similar to that of most mediums in use. Therefore layering on storage is minimised.

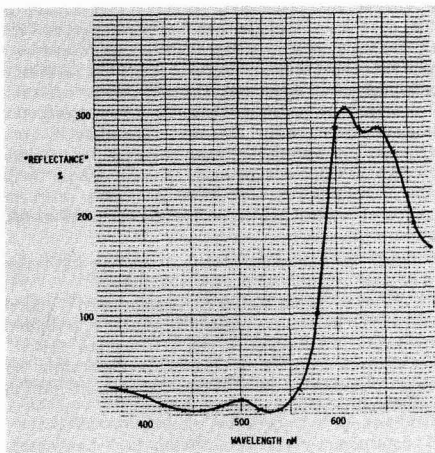
(3) The consistency of dyed resin types ensures good wetting characteristics and ease of dispersion in most systems.

(4) Because the fluorescent dyes are in solid solution within the resin matrix of the 'pigment,' bleed is reduced substantially with the associated advantages of convenience of use and considerations of health and safety. The matrix also offers a degree of protection to otherwise sensitive organic dyes enabling them to be used in hostile environments and with improved lightfastness.

A well formulated daylight fluorescent coating will give up to four times the colour intensity of non-fluorescent coatings of a similar shade or, put another way, they will be seen up to four times further away. Measured on typical laboratory colour reflectance equipment, good daylight fluorescent coatings should give the apparently anomalous result of peaking at well over 100% within a narrow band of frequencies (Figure 2). This is because reflected light is being reinforced by fluorescent emissions.

Figure 2

Typical well formulated daylight Fluorescent Orange* Paint peaking at over 300% *810 Orange 5 made by Sterling Industrial Colours, UK agent Capricorn Chemicals.



Types of fluorescent pigments

The resin matrix of most typical daylight fluorescent pigments for coatings is a melamine formaldehyde complex modified with sufficient sulphonamide to give a brittle carrier, which may be easily ground. It may be thermoplastic or thermoset, whichever is appropriate to the end use. Thermoplastic types generally give the brightest shades, depending on the application. They are normally recommended for water based systems such as air drying paper coatings and water based paints. However, thermoplastic matrices and the dyes they protect are prone to degradation resulting from solvent, chemical or thermal attack. Most commonly, the matrix particles in poorly formulated paints and inks soften and agglomerate on storage in the presence of strong solvents. Solvent based paints and inks usually demand the extra resistance of thermoset resin matrices. Those used in the manufacture of modern

thermoset daylight fluorescent pigments are largely insoluble in all commonly used strong solvents such as esters, glycol ethers, alcohols and ketones. The long term stability of water based formulations is also improved by the use of thermoset types. Increasingly, modern thermoset fluorescent pigments are the first choice for all coatings.

Application

Remembering that fluorescent coatings must be translucent in order to fluoresce efficiently, opacity will always be very low. It follows that the nature and colour of the substrate chosen will be of critical importance. Where fluorescent inks are applied on clean white paper or board this is not likely to be a problem. Pigmentation levels tend to be highest in screen inks – perhaps as high as 30% – and this further reduces the effect of the substrate. Paints usually have less pigment incorporated in them permitting greater control over application characteristics, mechanical performance and appearance. In this case the value of a very clean, uniform, dense blue/white undercoat cannot be understated. It should be remembered that fluorescent coatings must be considered as two coat systems. The real function of a white base coat is to capture more incident light and to reflect inward bound fluorescent emissions. Naturally any attempt to opacify the fluorescent coating with, say, titanium dioxide, will only result in a quenched pastel shade.

Lightfastness

The performance and stability of daylight fluorescent pigments has been improved so much in recent years that modern types are almost impervious to most artificial interior lighting. But fluorescent dyes, like most organic colourants, will be degraded by incident high energy ultraviolet light such as that emitted from ultraviolet discharge tubes or, more commonly, by the ultraviolet content of sunlight. The effect on fluorescent pigments is usually an initial darkening followed by a loss of fluorescence and eventual fading as the dyes are broken down. The apparent lightfastness of fluorescent coatings as measured on the International Blue Wool Scale depends on a number of factors including the degree of dispersion, pigment concentration, type and quality of binder/medium, filmweight, etc. It is always misleading to quote a Blue Wool figure for a colourant alone.

It has been clearly demonstrated that the most effective way of improving apparent lightfastness is the application of a clear overlacquer containing a well dispersed ultraviolet absorbing agent designed to prevent the transmission of higher energy light to the fluorescing film, while allowing the frequencies required for fluorescence to penetrate. Such ultraviolet absorbers can be incorporated into the resin matrix of daylight fluorescent pigments, however this usually results in an increased life of around 20% whereas a carefully formulated UV absorbing top coat has been shown to double effective life, giving an increase of 1 on the Blue Wool Scale. Ultraviolet absorbers commonly available are of the substituted benzophenone and benzotriazole class, such as Uvinol D49 from BASF, absorbing at wavelengths around 350 nanometers. Note that the combination of these with sterically hindered amines and amides in stoving lacquers may be proscribed by certain patents. The Tinuvin range of UV absorbers from Ciba-Geigy is recommended for most coatings.

In addition to the use of a high quality UV overlacquer, improved resistance to fading can be achieved:

- (1) by using as high a pigmentation as possible, provided,
- (2) dispersion is as uniform as possible and,
- (3) the pigment is well wetted,

Continued on p.497

Precis Papers: In this issue of JOCCA there are published in the Horizons Section of the Journal the final set of precis of a number of papers which were received for publication by the late Mr D. S. Newton. The Honorary Editor, Mr J. R. Taylor has refereed these papers and notified Council of their planned publication in precis format. Should readers require further information on these subjects please contact the author(s) whose address for communication is indicated below the title of each paper.

Products of potential industrial value from safflower oil.

by R. S. Balakrishna, G. S. R. Sastry and B. G. K. Murphy, Regional Research Laboratory, Hyderabad 500 007, India.

Summary

The isomerization of safflower oil was carried out using caustic soda with ethyl alcohol (commercial) at 200°C for 6 hours. The isomerised safflower oil/acid/esters were elaidinized using sulphur as a catalyst. Diels-Alder adducts were prepared by reacting isomerized and elaidinized safflower oil/acids/methyl esters with various dienophiles and then characterized. Some of the adducts were epoxidized. The adducts of isomerized safflower oil fatty acids with maleic anhydride and acrylic acid dienophiles were used as part replacement of the fatty acids or phthalic anhydride in alkyd to get alkali resistant and quick drying resins with improved hardness.

Use of castor oil-based products in formulating UV curable coatings.

by B. S. Sitaramam, P. C. Chatterjee and M. A. Sivasamban, Regional Research Laboratory, Hyderabad 500 007, India.

Summary

Two of the castor oil-based products developed by the authors were found to be useful in formulating UV curable coatings. They can be used to the extent of 30-40 per cent by weight of the total binder. They are non-toxic, have little odour and impart good flexibility to the cured film without adversely affecting the cure speed. They were totally compatible with the polyfunctional acrylates now in use. The films also exhibited good film characteristics.

Influence of cosolvents on the solvency power of solvents for resins.

by G. Narender and M. Yaseen, Regional Research Laboratory, Hyderabad 500 007, India.

Summary

Most of the polymeric materials used in the surface coatings industry are soluble in a limited number of primary solvents but they could be dissolved in solvent-cosolvent blends. The cosolvents of proper choice enhance the solvency power of the solvents and ease the dissolution of the polymer in the blend. The data on intrinsic viscosities of linseed oil alkyd and polyamide solutions in various blends of solvent-cosolvent have been used for assessing the influence of cosolvents on the solvency power of the solvents for these resins. The tolerance limit of the resin solution for the cosolvent in the blend has also been estimated from these data. The solubility parameter of the solvent blend in which the resin has the maximum compatibility and optimum intrinsic viscosity has been equated to that of the resin. It is found that knowledge about the solubility parameters of solvents and that of the resins is essential for deciding the composition of solvent blends as the resin has good compatibility with the blend when the solubility parameters of solute and solvent match each other.

Studies on water-borne wire enamels.

by R. Panda* and H. Panda†, Research & Development Section, The Indian Turpentine & Rosin Co Ltd, P.O. Clutterbuckganj - 243 502, Bareilly (U.P.), India.

* To whom all correspondence should be addressed.

† 1, Station Road, P.O. Clutterbuckganj - 243 502 Bareilly (U.P.), India.

Abstract

Water borne wire enamels are currently the subject of widespread interest, yet it is not a simple matter to substitute water for organic solvents. In this paper rosin has been used for the preparation of solvent-based as well as water-borne polyester wire enamels. The enamels have excellent thermomechanical and electrical properties which nearly satisfy the international specification.

Influence of Calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) Ore on the aqueous polymerization of methyl methacrylate.

by A. B. Moustafa, A. A. Abd El-Hakim and M. Abd El-Maboud Saar, Laboratory of Polymers & Pigments, National Research Centre, Dokki, Cairo, Egypt.

Synopsis

The aqueous polymerization of methyl methacrylate initiated by sodium bisulfite was studied at different temperatures 40, 45 and 40°C in absence and presence of $\text{Ca}_3(\text{PO}_4)_2$.

It was also found that the rate of polymerization increased with increase of the $\text{Ca}_3(\text{PO}_4)_2$ amount up to a certain value (6 g/100 ml of the reaction mixture), it also increased with an increase of temperature, the obtained average molecular weights were also found to increase with an increase of the calcium phosphate concentration and the rate of polymerization was found to increase with a decrease in the particle size, this holds good with its catalytic effect. The apparent activation energy was found to decrease with an increase of the surface area of the calcium phosphate.

Oil-modified polyesteramide resins: synthesis, characterization and film properties.

by T. K. Praveenkumar, P. S. Sampathkumaran and P. H. Gedam, Regional Research Laboratory, Hyderabad 500 007, India.

Summary

The polyesteramide resins of 65, 60 and 55% oil lengths from linseed oil, diethanolamine and phthalic anhydride were prepared, using the concept of oil length commonly employed in the manufacture of alkyd resins. The resins were characterized and their clear coatings, both as supported and free films, were evaluated. The coatings are in general, quick drying, possess resistance to water, acid and white spirit. They have good adhesion to the mild steel substrate. The resin of 55% oil length was found superior to the other two resins in many properties.

Water soluble paint vehicles for electrodeposition.

by G. S. Srinivasa Rao and M. M. Shirsalkar, Regional Research Laboratory, Hyderabad 500 007, India.

Summary

Water soluble maleinised cardanol-formaldehyde-drying oil vehicles have been prepared and paints were formulated from them at different pigment volume concentrations using natural iron oxide. Their hydrolytic and storage stability was studied. Electrodeposition of these paints particularly the relationship between time of deposition, voltage and dry film thickness of the coating at constant pH, temperature and bath solids have been studied. The properties of electrodeposited films such as adhesion, scratch hardness, gloss, impact resistance, flexibility, resistance to corrosion, chemicals/solvents were compared with those obtained by either the brushing or spin coating technique.

A semi-pilot plant study on utilisation of waste mica.

by P. C. Gupta, R. Singh, J. N. Chatterjee and P. K. Ghosh, Indian Lac Research Institute, Technology Division, Namkum, Ranchi - 834 010, India.

Abstract

A process for making flexible insulating big sheets (mica folium) from waste mica using shellac as a binder has been developed.

New organo-phosphorus metal complex pigments

by I. M. Abd-Ellah* and R. S. Farag, Chemistry Department, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.

* To whom all correspondence should be addressed.

Summary

Organo-phosphorus Schiff bases were recently prepared. These compounds are capable of forming complexes with various metal salts. The coloured complexes so formed are evaluated as pigments.

Stabilization of calcium carbonate by adsorbed methacrylic acid monomer and its polymer.

by A. A. Abd El-Hakim, Laboratory of Polymers & Pigments, National Research Centre, Dokki, Cairo, Egypt.

Summary

The dispersion of calcium carbonate in non-polar medium containing small molecules of surface active agent such as methacrylic acid and long chain molecules as polymethacrylic acid was studied. The isotherm of adsorption of methacrylic acid from cyclohexane consists of two steps, while that of polymethacrylic acid from ethanol is one step. The adsorption processes have a marked influence on sedimentation properties of calcium carbonate in a non-polar medium and shows a difference in the rate of sedimentation of the calcium carbonate treated with methacrylic or polymethacrylic acid. The effect of the modification of calcium carbonate with methacrylic acid was found to result in a more stable suspension in non-polar media than when the modification is carried out with its polymer.

Carbon-13 NMR spectroscopic determination of the fatty acid composition of a few edible oils.

by S. Husain*, R. Nageswara Rao, Mohd. Kifayatullah, G. V. R. Sarma and G. S. R. Sastry, Regional Research Laboratory, Hyderabad-7, India.

* To whom all correspondence should be addressed.

Abstract

The chemical characteristics of a few edible oils in terms of saturates, monoenes and dienes have been derived using ^{13}C NMR spectroscopy. Quantitative ^{13}C NMR spectra of 8 edible oils have been recorded in CDCl_3 by suppressing Nuclear Overhauser Effect (NOE) using the gated decoupling technique. The ^{13}C NMR carbon resonances utilized for analytical purpose are at δ 24.8, 25.7 and 27.3 ppm. The results were found in good agreement with those obtained by GLC method within 5% variation.

Effect of calcium carbonate modified surface with stearic acid on its stability and on the aqueous polymerization of methyl methacrylate.

by A. A. Abd El-Hakim, B.M.A. Abd-El-Hady and A. B. Moustafa, Laboratory of Polymers & Pigments, National Research Centre, Dokki, Cairo, Egypt.

Summary

The modification of calcium carbonate powder was carried out using stearic acid from cyclohexane, and the adsorption isotherm of stearic acid from cyclohexane on the surface of calcium carbonate was determined. The Langmuir isotherm for the monolayer surface covering at 10×10^{-5} mol stearic acid /g calcium carbonate was detected. Most stable suspensions were obtained by monolayer modification.

The modification of the calcium carbonate surface with stearic acid decreased the polymerization rate of methyl methacrylate in aqueous medium. The amount of unmodified calcium carbonate had no effect on the determined average molecular weights, while the stearic acid modified carbonate resulted in an increase of the average molecular weight.

Separation of alpha-methyl styrene-styrene-acrylonitrile terpolymer by thin layer chromatography.

by S. M. Sayyah and M. A. Abd El-Ghaffar*, Chemistry Department, Faculty of Science, Al-Azhar University, Nasr City, Cairo, Egypt.

* Laboratory of Polymers and Pigments, National Research Centre, Dokki, Cairo.

Abstract

An easy and rapid tlc technique for separation of α -Methyl Styrene-styrene-acrylonitrile terpolymer has been made in a single and binary eluent system using silica gel as a stationary phase. The separation of the terpolymer according to the chemical composition in both cases has been found to depend upon the adsorption mechanism. The phase profile for Acetone-Toluene system (1:4 by volume) on precoated plates with silica gel was investigated by Gas-Liquid chromatography. The polarity of the eluent has decreased from the start line to the solvent front at the end line (solvent gradient).

New corrosion inhibitors for surface coatings based on fatty materials. Part II: Field testing.

by S. M. El-Sawy and B. M. Badran, Laboratory of Polymers and Pigments, National Research Centre, Dokki, Cairo, Egypt.

Summary

This work describes an atmospheric exposure test of fifty two anticorrosive varnishes. Two testing stations were built specially for this work in two different atmospheres. Reaction products of epoxidised linseed oil reacted with aliphatic amines (methylamine, ethylamine, propylamine and butylamine) and aromatic amines (aniline, *o*-, *m*-, *p*-chloroaniline, *m*-, *p*-toluidine and *o*-, *m*-, *p*-anizidine) were used as effective corrosion inhibitors for varnishes. It was found that aliphatic amine derivatives can be used for preparation of anticorrosive varnishes, they have good corrosion protective properties and their efficiency increases with the decrease in chain length of the alkyl group. Aromatic amine derivatives have less efficiency for corrosion protection under such severe conditions.

Studies on the solubility parameters of alkyd and amino resins used in surface coatings

by D Bagchi* and N. C. Maity Research & Development Division, Berger Paints India Limited, Howrah 711103, India.

* To whom all correspondence should be addressed. Present address: Chemistry Department, Bowling Green State University, Bowling Green, Ohio 43403, USA.

Summary

Polymeric resins used in surface coating paint formulation should be properly soluble in solvent medium which is very important in terms of paint applications and paint film properties. Properly chosen co-solvent enhances the solvency power of the primary solvent and ease dissolution of the polymers in the blend. The maximal solubility of three alkyd resins and one amino resin (used as a crosslinker) in the solvent blend of xylol and/or butanol have been observed studying intrinsic viscosity $[\eta]$ as a function of composition of the binary mixture. $[\eta]$ is also experimentally observed as a function of solubility parameter(δ) computed from the composition of the solvent blend. The δ value of the solvent blend corresponding to the maximum point (maximal solubility) in $[\eta]$ vs δ has been assigned the solubility parameter of the respective resin.

Identification of dicarboxylic acids used in alkyd/polyester resins by PMR and CMR spectroscopic techniques.

by V. S. Kishanprasad, P. H. Gedam and P. S. Sampathkumaran, Regional Research Laboratory, Hyderabad 500 007, India.

Abstract

A method is described for the identification and determination of dibasic acids used in alkyd/polyester resins, involving the use of nuclear magnetic resonance spectroscopy. The resins were saponified with alcoholic potassium hydroxide and the potassium salts obtained were directly analysed by PMR and CMR in D_2O medium. The method can be used in identification of other polycarboxylic acids also.

Anomalous viscosity behaviour of fatty acid esters in solution.

by J. A. Ibemesi† and I. O. Igwe, Department of Pure & Industrial Chemistry, University of Nigeria, Nsukka Anambra State, Nigeria.

† To whom correspondence should be addressed.

Summary

Solution viscosity of oils of melonseed [*Colocynthis vulgaris* Schrad], soyabean [*Glycine max* (L) Merr], and rubberseed [*Hevea brasiliensis* (Kunth) Muell] were determined in different solvents at various concentrations. Reduced viscosity (η_{red}) versus concentration (C) plots showed three types of behaviour: η_{red} increased linearly with C(2-30 g/dl), η_{red} levelled off with C(2-1.2 g/dl) and η_{red} rose steeply with decrease in C (below 1.2 g/dl); the latter behaviour is anomalous. Similar viscosity behaviour were observed for the methyl ester of melonseed oil, methyl palmitate and n-butyl acetate. Molecular cluster formation was believed to cause the observed anomalous viscosity behaviour, the extent of which varied, depending on the nature of the fatty acid ester and the solvent.

Maleopimaric acid and its derivatives

by R Panda* and H Panda, Research & Development Section, The Indian Turpentine & Rosin Co. Ltd, P.O. Clutterbuckganj — 243 502, Bareilly (U.P.), India.

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Summary

Maleopimaric acid was prepared from the oleoresin and wood rosin of *Pinus roxburghii*. Its different derivatives were prepared and properties were evaluated. Also maleic anhydride unreactive type resin acids were analysed and its different derivatives were prepared.

Insulating varnishes from shellac.

by D. N. Goswami and S. Kumar, Division of Chemistry, Indian Lac Research Institute, Namkum, Ranchi 834 010, Bihar, India.

Abstract

Insulating varnishes are widely used for coating of armatures/coils of electric motors and transformers and also for finishing electrical components. Natural resin shellac finds considerable use in the manufacture of insulating varnishes. The present article provides information about the researches so far done on both air-drying and baking type of insulating varnishes based on shellac.

Solution properties of shellac. 3. : "Solubility parameter of shellac"—Part I.

by M. K. Mishra, Chemistry Division, Indian Lac Research Institute, Namkum, Ranchi - 834 010, Bihar, India.

Summary

The solubility parameter, δ , of shellac has been estimated by use of Van Der Waals constants and found to be $2.05 \times 10^{-4} N^{1/2} m^{-4} (10.01 \text{ cal/cc})^{1/2}$.

Determination of the molecular weight of shellac and its fractions.

by P. G. Gupta*, P. R. Bhattacharya† and G. S. Mishra‡, Technology Division, Indian Lac Research Institute, Namkum, Ranchi 834 010, India.

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‡ Director of Indian Lac Research Institute, Namkum, Ranchi, and at present Professor and Head of Dept. of Chemistry, University of Jammu, Jammu, India.

Summary

Dewaxed decolourised shellac has been fractionated by solvent non-solvent technique into several fractions. The molecular weight and intrinsic viscosity of these fractions have been determined and a simple Mark Houwink equation $[\eta]=7.018 \times 10^4 M^{0.67}$ (30°C, dioxane) has been proposed. The integral and differential curves have been drawn.

Modification of shellac — Part II: with melamine.

by S. C. Sengupta*, Indian Lac Research Institute (Indian Council of Agricultural Research, New Delhi), Namkum, Ranchi-834010, India.

* 5, Vivekananda Place, P.O. Konnagar, Hooghly-712235, West Bengal.

Summary

Shellac has been modified with melamine and melamine-formaldehyde under various conditions to give compositions which will thermoset after application and/or baking. Their properties and those of their spirit and water soluble varnishes were studied. A few compositions have been found to give the desired results. The smooth and high gloss films from these have shown good resistance to hot water, heat and spirit. Films on wooden surfaces are also smooth, highly glossy and resistant to hot water, heat and spirit. Their possible uses either as spirit or water soluble varnishes for French polishing, metal enamelling and the electrical industry have been discussed.

Modification of shellac — Part III: with polyisocyanate.

by S. C. Sengupta*, Indian Lac Research Institute (Indian Council of Agricultural Research, New Delhi), Namkum, Ranchi-834010, India.

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Summary

Shellac has been suitably reacted at room temperature with tolylene diisocyanate in a mixed solvent comprising dry methyl ethyl ketone and methanol or ethanol (9:1). The polyurethane coating cured at room temperature and the films were hard, glossy, flexible, uniform and resistant to water, heat, acid, alkali and solvents. Best performances were obtained with 35 to 40 per cent of the diisocyanate. With a higher amount the films became brittle which could be made flexible by incorporation of a small amount of castor oil. The pot lives of the best compositions were ten days and above. Their properties suggest their use in the field of metal lacquering, surface coatings, wood polishing and impregnating agents for water repellancy.

Modification of shellac Part IV: — polyurethanes from shellac ethers and ether esters.

by S. C. Sengupta*, R. K. Das and A. R. Sinha, Indian Lac Research Institute (Indian Council of Agricultural Research), Namkum, Ranchi 834010, India.

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Summary

Shellac-ethylene glycol ethers and their dibasic acid esters have been reacted in methyl ethyl ketone solution with diphenyl methyl and tolylene diisocyanate at room temperature. Compositions containing 30-40% of diphenyl methyl diisocyanate and 20-25% of tolylene diisocyanate respectively have been found to give clear, high gloss highly flexible, uniform non tacky and hard polyurethane coatings on metal, glass and wood. The coatings are also resistant to water, 5% alkali, acid and organic solvents. The ethers behave better than the esters. The pot lives are quite sufficient for application, and conditions for a one-pack system have also been worked out. The properties suggest their use in the field of metal enamelling, surface coatings, wood polishing and water repellancy.

Mechanism of the reaction of lac with sulphur.

by A. Kumar* and B. B. Khanna, Indian Lac Research Institute, Namkum, Ranchi - 834010, India.

* Present address: Dept. of Chemistry, Magadh University, Patna, India.

Summary

The mechanism of the reaction of lac, its fractions or hydrolysed lac with sulphur have been investigated. When lac is heated alone, a reduction in acid and hydroxyl values is noticed while in the presence of sulphur, in addition, a lowering in the iodine value is also noticed. A similar mechanism as in the case of the reaction of sulphur with oils explains both evolution of H₂S and the reduction in unsaturation is proposed. The possibility of cross-linking taking place simultaneously is also indicated. The presence of a rubber accelerator like M.B.T. at the time of the reaction, enhances the sulphur content of the sulphurated products to a little extent.

A process for improving the bond strength and colour of lac resin recoverable from kiri, a by-product of the lac industry.

by S. K. Saha, B. P. Banerjee and L. C. Misra, Indian Lac Research Institute, Namkum, Ranchi, India.

Summary

Kiri, a by-product of lac industry, was treated with aqueous sodium carbonate and the extracted lac resin was subjected to partial hydrolysis using a low concentration of sodium hydroxide in order to improve the bond strength of the product. Hydrolysis using 2% NaOH for 4 hours resulted in a product with a bond strength better than that of D. L. Shellac. The colour of the hydrolysed *kiri* lac could also be improved by treatment with sodium sulphite or sodium chlorite. Based on these studies a simple and inexpensive method for the recovery of a superior quality of lac resin from *kiri* could be envisaged.

Modification of lac, hydrolysed lac and lac-oil combinations with polyisocyanates.

by P. M. Patil and B. B. Khanna, Indian Lac Research Institute, Namkum, Ranchi - 834010, Bihar, India.

Summary

Shellac, hydrolysed lac and lac-oil combinations (with linseed oil and castor oil) were treated with some of the recently developed polyisocyanates such as Desmodur N, Desmodur VL, Desmodur L and Desmodur E21. The film properties of the resultant polyurethanes were studied.

The best results in respect of water, acid and alkali resistance were obtained from the lac-linseed oil combination using Desmodur N (75%) with even air dried films resisting water, acid and alkali. The films obtained were also uniform, glossy, flexible and hard.

Solubility parameter of aleuritic acid and its alkyl esters by contribution techniques.

by B. C. Srivastava, Indian Lac Research Institute, Chemistry Division, Namkum, Ranchi - 834010 Bihar, India.

Abstract

Aleuritic acid is a major component acid of natural versatile resin lac. It is a unique hydroxy acid (9,10,16-trihydroxy palmitic acid) containing one carboxyl and three hydroxyl groups of which two are on adjacent carbon atoms. The solubility parameter (δ) is a fundamental physical property of any organic material and is the square root of the cohesive-energy density.

Utilisation of aleuritic acid free-gummy hydrolysed lac in surface coatings.

by S. C. Agarwal and B. C. Srivastava, Indian Lac Research Institute, Namkum, Ranchi-834010, India.

Summary

The gummy hydrolysed lac (~50%), obtained as a waste product after the isolation of aleuritic acid, has been modified with ethylene glycol and subsequently reacted with Desmodur N. The final product possesses good adhesion, hardness, flexibility and impact resistance. The possible use of it in place of normal hydrolysed lac in the field of surface coatings is discussed. During the process of isolating aleuritic acid (9,10,16-trihydroxy hexadecanoic acid) from shellac through alkaline hydrolysis, a gummy hydrolysed material to the extent of about 50 per cent is left as a waste product which has no known use and whose disposal hitherto has been a problem.

Aqueous adhesive from Rebulac for metallic surfaces.

by R. K. Banerjee and P. C. Gupta, Technology Division, Indian Lac Research Institute, Namkum, Ranchi-834 010, India.

Abstract

The optimum pressure, temperature and time of bonding of metallic surfaces with aqueous Rebulac were found to be 3000 lbs/sq. inch, 200°C and 45 minutes. The bond strengths of aqueous Rebulac on copper and steel surfaces were found to be 0.40 and 0.38 ton/sq. inch respectively, which were 5

and 6.3 times higher than those obtained by using shellac under similar conditions. Aqueous total hydrolysed lac was also found to impart significant bond strength to copper (0.23 ton/sq. inch) and steel (0.15 ton/sq. inch) surfaces, which were, again, 3 and 2.5 times higher than those obtained by using Shellac.

Modification of rebulac with glycol, acids and polyisocyanate.

by S. C. Sengupta*, D. Ghosh and R. K. Das, Indian Lac Research Institute (Indian Council of Agricultural Research), Namkum, Ranchi-834010, India.

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Summary

Rebulac has been modified with ethylene glycol and/or dicarboxylic acids, linseed oil fatty acids and diphenyl methyl diisocyanate. Their properties and those of their varnishes and paints have been studied. Baked films from aqueous media of a few of these resins were found to have outstanding hardness, good water resistance, adhesion, flexibility and elasticity, in contrast to aqueous lac varnishes. The polyurethane coating with DMDI (40%) plus a small amount of castor oil cured at room temperature to give hard, glossy, flexible and water, acid, alkali and solvent resistant films. Their properties suggest their use in the field of metal lacquering, surface coating, wood finishing, indoor painting, etc.

Thermal degradation and characterization of poly(methylmethacrylate) prepared in presence of burnt mazote boiler deposit.

by A. B. Moustafa, M. A. Diab*, S. M. Sayyah** and A. A. Abd El-Hakim, Laboratory of Polymers and Pigments, National Research Centre, Dokki, Cairo, Egypt; Faculty of Education at Domiat, Mansoura* Univ. and Faculty of Science, Al-Azhar** Univ., Nasr City, Cairo, Egypt.

Abstract

The thermal degradation of poly(methylmethacrylate) was previously studied by Grassie and Melville in 1949 to give a monomer in high yield. The principal features were revealed by a combination of molecular weight and rate measurements. The possibility of applying thin layer chromatography (TLC) to the molecular weight study of homopolymers and their molecular weight distribution has been mentioned by Inagaki and coworkers.

In this work is studied the degradation of some poly(methylmethacrylate) samples prepared by different methods in the absence and presence of burnt mazote boiler deposit (BMBD) substance and TLC experiments were made for the original and resulting degraded polymers in order to study the mechanism of degradation and the polydispersity of the obtained polymers.

Nature of shellac in alcoholic dilute solutions
by M. K. Mishra, Chemistry Division, Indian Lac Research Institute, Namkum, Ranchi- 834 010, Bihar, India.

Abstract

Study of particle size, conductivity and dielectric properties of shellac in dilute alcoholic solution shows that shellac is colloidally dispersed. ■

The effects of grain refining chemicals on the morphology and corrosion resistance of zinc-phosphate coating on mild steel substrates

by N. C. Debnath, G. N. Bhar and Sandip Roy*, Application Research and Development (Paints), IEL Limited, Chemicals Division, P. O. Rishra, Dist. Hooghly 712248 West Bengal, India

Abstract

A comparative study of the effects of two industrially important grain refining chemicals — based on oxalic acid and titanium compounds — on the morphology, structure and corrosion resistance of zinc phosphate coatings on mild steel surfaces has been carried out. A combination of analytical techniques viz. SEM, XRD, EPMA and AAS were used to characterise the phosphate coating. The data shows that the performance of titanium based formulations is much better in respect of coating texture and corrosion resistance when compared with that of oxalic acid based formulation. The superior performance of the former may be attributed to the special features that characterise the zinc phosphate coating obtained from it viz. smaller grain size, lower coating weight, lower Zn:PO₄ ratio and lower P/P+H ratio. It has been postulated that smaller grain size/compact coatings results from increased number of nucleating sites generated by titanium incorporation into the coating. XRD data clearly indicates such incorporation.

Introduction

The morphology and structure of phosphate coatings on a mild steel surface depend substantially on the type of pretreatment process (degreasing, derusting, surface activation etc.) used in the preparation of the metal surface before phosphating. For industrial phosphating processes, particularly those used in the Automobile Industries, a grain refining stage is invariably incorporated just before phosphating stage in order to modify the morphology of the phosphate coating and thereby improve its corrosion resistance and ensure better adhesion of the paint film to the substrate.

The two most important grain refining agents that have found wide application in the industrial zinc phosphating process used in automobile industries are based on either oxalic acid or Titanium compounds. While quite a few References^{1-3,5-6} are available showing the effect of the grain refining stage on the quality of phosphate coating, in general, no systematic study has so far been made to find comparative results on the efficacy of these two refining agents with a particular type of zinc-phosphating chemical in a standard pretreatment process involving both degreasing and derusting stages prior to surface activation stage. Very recently Freeman has reported some results⁴ on the effect of grain refining agents on the quality of phosphate coating on mild steel surfaces. However, his work is confined mainly to morphological studies.

In the present study the effects of two grain refining agents on the morphology, crystal structure and composition of the phosphate coating on degreased and derusted mild steel surfaces have been investigated. An attempt has also been made to explain why one formulation gives better results than the other. The phosphate coating has been characterised by a combination of analytical techniques. The coating

morphology has been studied by Scanning Electron Microscopy (SEM), coating crystal structures by X-ray Deffraction (XRD), the relative proportion of Hopeite and Phosphophyllite determined by Atomic Adsorption Spectroscopy (AAS) and the elemental analysis by Electron Probe Microanalysis (EPMA). Finally, the corrosion resistance properties of the phosphated and painted coatings were evaluated in a salts-spray chamber.

Experimental procedure

Panel preparation

Mild steel panels (6' × 4") were cut from cold rolled sheets used for manufacturing car bodies. Two proprietary grain refining chemicals A & B based on oxalic acid and titanium compounds respectively were used as a pre-dip before phosphating the degreased/derusted panels in a nitrite/nitrate accelerated dip phosphating chemical. Four sets of panels were cleaned phosphated and passivated as per the sequence described in Table 1 and Table 2.

Painting

The dried phosphated panels were painted with stoving finish based upon Alkyd Melamine Formaldehyde to a film thickness of 55-60 μm. The paint was applied on all the panels by conventional spray method and the panels were baked at 140°C for 20 mins. The painted panels were allowed to mature for 24 hours, before exposing the scribed panels to salt-spray experiments (as per ASTM B-117).

Scanning electron microscopy

Electron micrographs of the phosphated panels were taken at different magnifications in a Hitachi S-415 A model SEM at 25 KV and using secondary emission mode.

X-ray diffraction

The diffractograms of the sample stripped from the substrate with the help of a scalpel were taken in a Philips PW 1010 X-ray crystallographic unit using a copper target.

Table 1

Surface preparation and phosphating

Sample No.	Process sequence
1	DA+PH+CR
2	DA+SA1+PH+CR
3	DA+DR+SA1+PH+CR
4	DA+DR+SA2+PH+CR

DA = Degreasing in Metasilicate based alkaline solution.

DR = Derusting in Phosphoric Acid based chemical.

PH = Phosphating in nitrate/nitrite accelerator based zinc phosphating chemical.

SA1 = Surface Activation chemical based on Oxalic Acid.

SA2 = Surface Activation chemical based on Titanium compound.

CR = Chromic Acid Rinse.

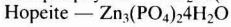
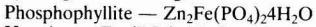
* The author is currently associated with research and development activities of the Water Treatment Chemicals Group.

Electron probe microanalysis

Microanalysis of the phosphated samples were carried out in a Cambridge Stereo SCAN 150 model with the Link Systems ED 290 at a voltage 20 KV and a beam current 360 micro amperes.

Atomic absorption spectroscopy

The amount of zinc and iron in the phosphate coatings were determined by dissolving them in 5% chromic acid solution at 70°C and analysing the solution with Atomic Absorption Spectrophotometer (AAS). The proportion of Hopeite (H) and phosphophyllite (P) were then determined by using the following molecular formulae:



Results and discussion

The scanning electron micrographs of the four phosphated panels, processed as per the sequency in Tables 1 and 2, are shown in Figures 1 to 4. Comparison of Figures 1 and 2 clearly indicate the extent of grain size, reduction and the corresponding increase in coating compactness imparted by oxalic acid based formulation for non-derusted surfaces. However, a comparison of Figures 3 and 4 which shows the

effects of two grain refining agents on the derusted surface clearly show that the titanium based formulation (Figure 4) is much more superior than the oxalic acid based fomulation (Figure 3). The better grain refining effect in Process 4 can be attributed to the presence of titanium compounds in the coating itself as is evident by the X-ray diffractogram (Figure 5) showing a distinct peak of a titanium compound which is absent in the other two panels in Figure 5. Incorporation of Titanium in the coating increases the density of nucleating centres^{1,2} on substrate substantially thereby reducing the grain size. The EPMA data incorporated in Table 3 show a significant decrease in Zn:PO₄ ratio for the Process 4 compared to Process 3. The number of zinc atoms available for lattice formation, therefore, gets reduced when Titanium based formulation is used as a grain refining agent in place of oxalic acid. In other words, the incorporation of zinc in the lattice is relatively hindered in the former case. This "lattice hindrance" may be another reason for the grain refining effect shown in the SEM photograph (Figure 4).

Salt spray results (after 216 hours of exposure) shown in Table 3 clearly demonstrate that the rust creep from the scratched line is almost half (1.5 mm) when treated with titanium compared to oxalic acid (3.0 mm). The coating weight also gets reduced to about half in the former case (2.80 compared to 5.54 mg with oxalic acid). Reduced coating

Figure 1

SEM micrograph of Sample 1 (Degrease/Phosphate/Chromate Passivation)

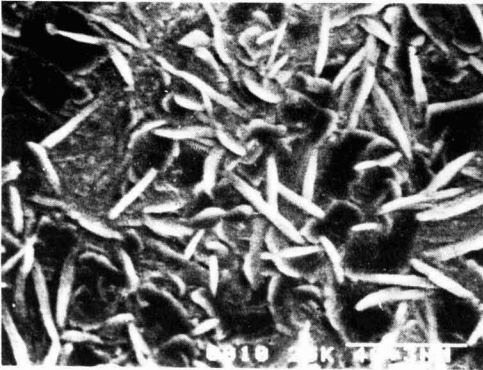


Figure 2

SEM micrograph of Sample 2 (Degrease/Oxalic acid Activation/Phosphate/Chromate Passivation)



Table 2

Surface pretreatment procedure

Process Sequence	Operation	Mode of Operation	Parameters of the bath	Concentration of the bath	Temperature of the bath (°C)	Time of Operation (minutes)
1	Wiping with dry cloth	—	—	—	—	—
2	Degreasing	Dip	—	5%(W/V)	80-85°C	4 mins
3	Rinse in Tap water	Dip	—	—	Room Temp(RT)	1 min
4	Derusting	Dip	—	25%(V/V)	RT	4 mins
5	Rinse in Tap water	Dip	—	—	RT	1 min
6	Surface Activation					
	A	Dip	pH = 2.15	0.4%	RT	1 min
	B	Dip	pH = 7.86 Ti = 23.20 ppm	0.145%	RT	1 min
			Content			
7	Phosphating	Dip	TA = 27 Toner Titration = 1.8-2.0 ml pH=2.8	4.4%(V/V)	70°C	4 mins
8	Rinse in Tap water	Dip	—	—	RT	1 min
9	Passivation	Dip	—	0.2%(V/V)	RT	½ min
10	Rinse with demineralised water	Spray	—	—	RT	½ min
11	Drying in air	—	—	—	110-120°C	2 mins

weight and smaller grain size in the Process 4 results in a more compact coating; thus increasing the corrosion resistance, and hence reducing rust-creep.

The Phosphophyllite/Hopeite ratio (Table 3) in the phosphate coating pretreated with oxalic acid is much higher than the coating produced by the titanium pretreatment. The corrosion resistance in the former case is much inferior to that of the latter. The contention⁷ that increased phosphophyllite in the phosphate coating increases the corrosion resistance of the coating is not valid at least in the case of zinc phosphate coatings produced on derusted surface.

Figure 3

SEM micrograph of Sample 3 (Degrease/Derust/Oxalic Acid Activation/Phosphate/Chromate Passivation)



Figure 4

SEM micrograph of sample 4 (Degrease/Derust/Titanium Activation/Phosphate/Chromate Passivation)

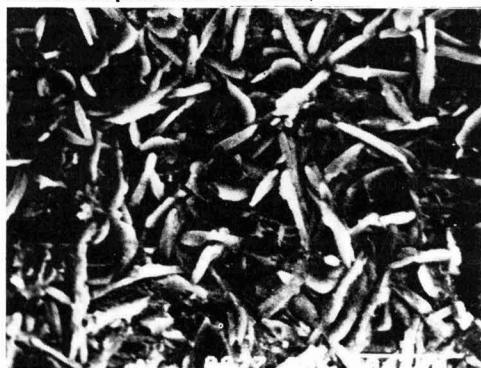


Table 3

Characterisation of the phosphate coating

Sample No.	Coating wt (gms/m ²)	Coating composition (XRD data)	P/P+H ratio (AAS data)	P	Fe	Zn	Weight ratio in The coating Zn:PO ₄	Salt Spray Results 216 hours (mm)
				Wt. = x10 ³ (EPMA data)	arbitrary unit			
1	4.00	Mixture of P* and H†	0.49	4.1	104	38.7	4.08	‡BS=4.0
2	5.32	—	0.77	—	—	—	—	BS=3.0
3	5.54	Mixture of P and H	0.77	6.8	379	67.0	3.21	BS=3.0
4	2.80	Mixture of P, H probably TiO	0.57	3.1	100	11.0	1.16	BS=1.5

*P=Phosphophyllite, Zn₂Fe(PO₄)₂·4H₂O

†H=Hopeite, Zn₃(PO₄)₂·4H₂O

‡=Blister Spread from the scribed line

Figure 5

X-ray diffraction results where P=Phosphophyllite, H=Hopeite and Ti=Titanium Compound

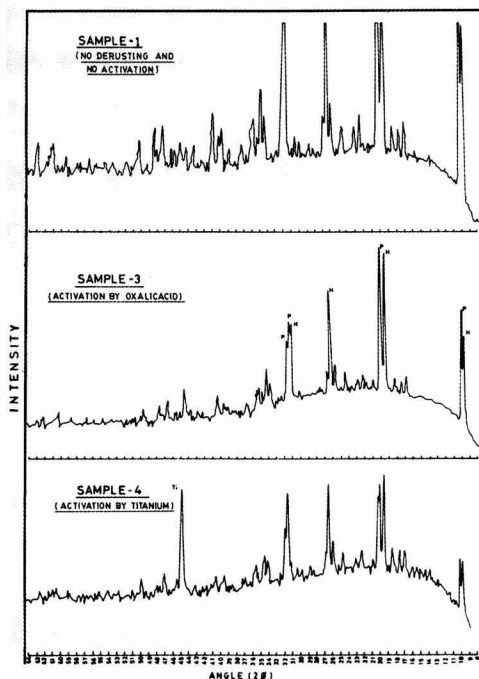


FIG. 5. XRD RESULTS
P = Phosphophyllite, H = Hopeite, Ti = Titanium compound.

Conclusions

In the present work, a comparative study on the effect of grain refining chemicals on the morphology, coating composition and corrosion resistance of a zinc phosphate coating on derusted mild steel panels has been carried out in detail. It has been clearly demonstrated that:

(a) Titanium based pre-rise gives the best quality zinc-phosphate coating (reduced grain size, lower coating weight and increased corrosion resistance).

(b) The reduction in grain size takes place because of incorporation of a titanium compound into the substrate/coating leading to more nucleating sites on the

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The Role of a Research Association: Industry sponsored research projects at the Paint Research Association

by D. J. Walbridge, Paint Research Association, Waldegrave Road, Teddington, UK

The Paint Research Association fulfils a number of separate functions in its provision of services to the surface coatings industry. It offers a comprehensive information service covering all aspects of coatings, including critical appraisal of relevant scientific developments and reviews of health and safety matters. It operates a consultancy service to Members, and it has a well-equipped analytical laboratory and paint testing facilities available for contract work. In addition to this core of essential supporting activities for the industry, it has always been recognized that the Association has a role to play in carrying out original research, with the aim of increasing basic knowledge in the science of surface coatings.

For a number of years this research was directed towards inventive developments of potential use in paints. Recently there has been a change of emphasis and the PRA now aims to identify areas of fundamental interest, by discussion with industry, leading to larger long-term projects which will allow staff to develop special expertise and utilize the latest research tools. Since the funds available for capital purchases are limited, it often is necessary to seek the co-operation of the universities and other research laboratories with appropriate instruments and skills to assist in carrying out the research. Funding for these research projects is sought from the UK government or the EEC, both of which will provide up to 50 per cent of the cost of an approved project. The remainder has to come either from the Member's subscriptions or more usually by forming a consortium of industrial sponsors with an especial interest in the project. The costs are then divided equally between the sponsors. In return they have immediate access to the results of the research and they play an active part in guiding the project throughout its lifetime so as to ensure its continuing relevance to their interests.

The selection of a suitable project is not an easy matter. The research must lead to real advances in knowledge and understanding in problem areas of interest to a broad range of the member companies. It must be 'pre-competitive' in nature to avoid a conflict of interest with the member's own research activities and to qualify for government funding. Government committees when screening proposals are liable to say that if the problem is of genuine importance to industry, then industry should pay for the research. Conversely, if the project does not command that degree of industrial support then why should it be given government funds? It is a difficult course to steer!

Despite these difficulties there is in fact a range of scientific problems for which a research association is particularly suited. Surface coatings, for example, are invariably complex systems which, if simplified and investigated in the traditional academic fashion, would give results not immediately applicable to real-life situations. On the other hand, industrial research departments are chiefly concerned with developing new products and only rarely can justify investigating mechanisms in depth. In a research association the more fundamental problems can be addressed, but in a way that accepts complexity and seeks sufficient, rather than absolute, explanations. There is then a better chance that the results can be utilised in solving everyday industrial problems.

Collaboration with industry is of vital importance. The choice of topic and the aims of the research must be clearly defined at the outset in conjunction with the sponsors. Once

the project is underway, close contacts must be maintained. The PRA holds regular Steering Committee meetings for each project which are intended to provide a forum in which the experimental results are presented and debated and where new directions are decided. Experience has shown that the members also benefit from meeting their suppliers' and customers' representatives and their opposite numbers from their competitors.

To carry out this kind of research requires well-qualified, imaginative scientists. They must be able to forge links with universities in order to evaluate new techniques and to enlist the help of expert academic consultants when this is necessary. The PRA has been fortunate in attracting the right quality of staff for its new projects, but the longer term problem of retaining or replacing staff in the face of the predicted shortage of science graduates is likely to be severe.

In the sections which follow several of the current projects at the PRA are reviewed, in order to illustrate the different methods of funding and to give an indication of the range of the scientific problems under investigation.

Mechanism of action of auxiliary metal driers

Under the terms of the voluntary agreement between the Paintmakers' Association and the Department of the Environment, lead driers ceased to be used in decorative paints after June 1987. Lead has always been regarded as an essential component of oil-based paints because of its property of promoting good drying under adverse conditions and in thick films. The choice of a replacement was made largely on empirical grounds, because almost nothing was known of the mechanism by which secondary or auxiliary driers function when used in combination with cobalt.

The PRA had already been investigating novel complex driers as possible replacements for cobalt and lead and was therefore well qualified to propose an investigation of the mechanism of auxiliary driers. The proposal received the support of the manufacturers of metal driers and the leading paint companies, making a total of fifteen sponsors. The Department of Trade and Industry gave approval for 50 per cent support for a three-year period beginning in February 1987. The studies have concentrated on the actions of lead, zirconium, calcium and aluminium in a variety of combinations with cobalt.

The investigation has focused on three facets of the drying process:

- (1) The differences in composition between the breakdown products generated during drying by the use of different drier combinations. Initially, these were analysed by gas-liquid chromatography/mass spectrometry (GCMS) but more recently with the purchase of liquid chromatography (HPLC) equipment, the non-volatile products from drying soya-bean oil have been studied. We are currently discussing with Dr Hancock of Royal Holloway College the identification of these fragments by LC-MS.
- (2) The spectroscopic analysis of the drying film. At an early stage in the project, the PRA purchased an FTIR spectrophotometer fitted with a horizontal attenuated total reflectance (HATR) stage. Films are cast on the HATR crystal and infra-red spectra are measured at intervals over several hours. The infra-red beam penetrates the bottom 10 μm of the film in contact with the

crystal. By varying the thickness of the applied film it is possible to study the progress of the drying process at different depths below the air interface. It is also relatively simple to study low temperature drying by this method.

Carbon-13 NMR studies at the Universities of Kent and East Anglia have proved more difficult to interpret because of the complex mixture of reaction products. A combination of preparative chromatography and NMR may prove more fruitful.

- (3) The state of the metal drier in the film. Although the role of cobalt in catalysing the breakdown of hydroperoxides is reasonably well understood and can be represented in terms of an oxidation/reduction cycle between Co^{II} and Co^{III} , nevertheless the exact nature of the cobalt atom complex is still uncertain. It is generally believed that the active cobalt species is a dimer in which one cobalt is divalent and the other trivalent. Whether the secondary drier metals, which are present in much greater amounts than cobalt, interact with cobalt, whether they catalyse the oxidation process independently, or whether they contribute to drying in some other way is not known.

The technique of Extended Absorption X-ray Fine Structure (EXAFS) can in principle be employed to study the state of metal atoms and their immediate environment within a paint film. Previously no techniques have been available for such studies at the low metal concentrations employed. One of the industrial sponsors, ICI plc, undertook EXAFS studies of drying paint films at the SERC Daresbury synchrotron facility as a contribution in kind to the programme. The results are still being analysed but they have already given a valuable insight into the state of aggregation of cobalt, lead and zirconium in the film.

While we are still some way from being able to write down a definitive mechanism of action for the auxiliary metals, it can already be seen that lead and zirconium operate in very different ways and also that calcium has a surprisingly important role in the drying process.

Mechanism of action of anti-corrosive paints

This is another major research project supported by the DTI and fourteen industrial member sponsors, made up of pigment and paint manufacturers and major users of anti-corrosive paints. The need to involve outside experts was recognised from the outset. A senior consultant, Dr J. G. N. Thomas, was appointed and arrangements were made for some aspects of the research to be carried out in collaboration with Dr J. Sykes (Oxford University) and Dr Turgoose (UMIST).

Traditionally, anticorrosive paints have been based on lead and chromate pigments which are now seen to present unacceptable toxic hazards. The search for alternatives is hampered by the complexity of the mechanisms of anti-corrosive protection. It is probable that there is no general mechanism and that successful compositions are effective because each presents a unique combination of physical and chemical protection.

The initial objectives were the study of the phosphate class of inhibitive pigments, complemented by a study of an established inhibitor, zinc chromate. The particle size distributions of zinc phosphate and the orientation within the paint film of the lamellar particles have been studied for a range of commercial pigments. These pigments have been found to vary quite widely in their chemical composition, particle size and lamellar characteristics.

A major effort has also been directed to the assessment of electrochemical methods for following the early stages of corrosion. At the PRA, emphasis has been placed on the

study of AC impedance. Samples used in these experiments have then been examined by the transient potential technique which is being developed at Oxford. An exercise is also under way, using mathematical techniques, to determine the validity of the equivalent electrical circuits employed in interpreting the electrical data, and to attempt to assign practical significance to the electrical elements in the circuit. At UMIST a post-graduate student is studying the mechanism of action of water-borne anticorrosive coatings on steel.

Expert systems – a coating selector for ferrous manufactured goods

This project was 50 per cent funded by the EEC under the SPRINT technology-transfer initiative. It brought together the PRA and three kindred organisations, CoRI in Belgium, EOLAS in Ireland and TIC in Denmark, in a collaborative venture to develop a computer-based system for choosing the correct coating for the general industrial finishing of steel articles. The specialist fields of automotive, can and coil coating were deliberately excluded.

The selector is available on floppy disk for use with an IBM compatible PC and it aims to simulate the advice which would be given by an experienced consultant. In its operation it poses a series of relevant questions which lead the user through the sequence of cleaning, pretreatment, application method, cure and performance in relation to the construction, shape and purpose of the article to be coated. It provides a guide to good practice which could be used by a consultant to lead a client through the questions which need to be considered, by an industrial finisher faced with a new problem, or as a training aid for technical service staff.

It was written at the PRA, using a proprietary software 'shell' and bringing together the expertise available from the four contributing associations. Copies of the Coating Selector in its present form may be purchased from the PRA. A demonstration disk is available. The extension of the project to include coatings for non-ferrous substrates has been approved by the EEC.

Self stratifying coatings

This project arose out of a meeting between the coatings research associations within the Common Market, which sought to identify joint ventures which would utilise their individual strengths. As a result a successful application was made under the EEC BRITE programme for a 50 per cent funding of a four-year project to investigate the formulation of self-stratifying coatings – single coat systems based on incompatible resins which spontaneously separate into two functional layers of primer and topcoat in one application.

The project, which is under the overall management of the PRA, brings together CoRI (Belgium), CERIEPEC (France), FPL (W Germany), EOLAS (Ireland), NIF and TIC, both in Denmark and TNO in the Netherlands. In the UK, the PRA will have the support of eighteen industrial sponsors.

The selection process was highly competitive. There was a first screening, in which the original 471 submissions were reduced to 112, followed by the final selection of 46 projects for immediate funding. The process is not recommended for the faint-hearted and it requires considerable stamina! Almost two years elapsed between the first application and the final approval for the commencement of the project on 1 January 1989.

The project's objectives are fourfold: (1) to formulate a number of experimental self-stratifying coatings for applications from solution or dispersion on steel, wood or plastic; (2) to identify the parameters controlling stratification, including application techniques; (3) to characterise promising systems by the use of modern analytical techniques and standard per-

formance tests and (4) to establish a computer-based model embodying the scientific principles of stratification, on the basis of which industry could develop proprietary systems.

The basic idea of a self-stratifying coating is, of course, not original. The concept has been discussed in the academic literature and several patents have been published. Reports have also been received of working systems in the form of powder coatings. Liquid coating systems appear not to have been explored in detail and there is much to be discovered by the investigation of the parameters controlling the phase separation and stratification of polymers in thin films.

The protection of exterior woodwork by modern surface coatings

Most projects are planned to run for three or four years. However, the process of drafting a project, seeking industrial support and approval, and finally securing government or EEC funding can take between one and two years. Consequently, long-term planning is essential and the Association needs to generate research proposals at a steady rate, avoiding the temptation to relax once a major project is launched.

The Protection of Wood project is at the stage when government funding has to be sought. Industrial sponsors have indicated their willingness to support the research as being both relevant and timely. Collaborative programmes have been agreed in outline with two universities. The rules for DTI funding are currently undergoing some changes so it remains to be seen what the outcome will be.

Coatings for exterior joinery have been the subject of major improvement in recent years with the introduction of 'flexible' microporous and 'breathing' pigmented paints, and with the growth in the use of clear wood stains and varnishes. The performance of these systems is likely to be very different from the previous generation of coatings.

The project aims to study the performance of these new coatings, paying particular attention to the wood/coating interface and looking for signs of incipient degradation of the wood structure or the adhesion of the coating which may be caused by the action of light, moisture or microbiological organisms at the interface. Dr Dickinson (Imperial College) and Dr Banks (Bangor), who are both authorities on the surface structure and degradation of wood, will undertake studies in support of the PRA programme.

Conclusions

The number of major research projects which can be undertaken at the PRA is determined by the size of the institution, the balance which has to be maintained with consultancy and contract work for the Members, and by the extent of the need within the industry for fundamental studies. The current research programme comprising three major and two or three smaller projects does provide a range of topics of interest to a wide spectrum of Members. The maintenance of an active programme of research also helps to ensure that new techniques and developments of potential value to the members are not overlooked.

Martindill, Continued from p.486

(4) by selecting the best quality non-yellowing binder (acrylics generally give best results),

(5) by keeping processing temperatures down, particularly when high speed stirring, and

(6) by applying as high a filmweight as possible.

A lesser known technique for improving apparent persistence of colour is to incorporate a very small percentage of a bright non-fading colourant with a similar hue to the fluorescent so that a residual colour remains after the fluorescent fades.

Conclusion

Fluorescent coatings based on daylight fluorescent pigments are all around us, on police and emergency vehicles, safety and sports clothing and equipment, road markings and cones, on printed textiles, in extruded, injection moulded and calendered plastics, in cosmetics, on posters, labels and price tags, and a thousand other items. They are there for one reason only - high visibility. The range of any coatings manufacturer is incomplete without daylight fluorescent colours.

Denbath et al, Continued from p.494

surface. The lower Zn/PO₄ ratio in the coating (1.16), when titanium based pre-rinse is used, indicates some kind of lattice hindrance to the entry of zinc atoms thereby further helping the grain refining process.

(c) Unlike the phosphate coatings obtained on a rust-free surface, the increasing amount of phosphophyllite in the coating does not increase its corrosion resistance when a fine grained thin compact zinc phosphate coating is produced on a derusted surface.

Acknowledgements

The authors are thankful to the management of IEL Limited for the permission to publish this work. Thanks are due to Dr. C. S. Narshimhan of Alchemie Research Centre, Thane, India for the assistance provided in getting EPMA analysis carried out.

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Appendix

Trade names of the surface activation chemicals based on the Titanium Salt and Oxalic acid are PHOSFIX 504 and PHOSFIX 502 respectively. Both the products are manufactured and marketed by IEL Limited, Chemicals Division. Both the products are used in a very low percentage in distilled water. PHOSFIX 504 when dissolved in distilled water offers a slightly alkaline colloidal solution of titanium containing polyphosphate as the colloid stabiliser. On the other hand, the aqueous solution of PHOSFIX 502 is a very dilute solution of oxalic acid.

Edited versions of the discussions that followed the presentation of a paper at the OCCA Chester Conference 1989 are published below. These papers were published in September and October JOCCA. Only those questions are published for which discussion slips have been received.

Keynote Address: Profitable R&D by **D. Pirret, Shell Chemicals UK Ltd**

Published in September 89 JOCCA

Mr B. Hayton, Colas Products: Could Mr Pirret provide any guide to the level of expenditure on R&D typical for companies in relation to their size or other factors?

Mr D. Pirret: The usual measure of relative R&D spend is a percentage of proceeds, which is not an ideal measure as it does not reflect the scale effect — for example there is a threshold spend below which the technological position would be critically weak therefore a low market share player may spend a relatively higher percentage of proceeds than the high share player with lower absolute return.

The industry in which companies operate will also affect the spend e.g. pharmaceuticals and agrochemicals typically require more R&D per pound of proceeds than base chemicals. Therefore one must always compare "apples with apples and not pears".

4-5% of proceeds is not unusual for a major wide portfolio chemical company, with base chemical companies spending say 1-2%, drug companies often spend more than 10%.

Care needs to be taken with company definitions of R&D which will also affect the percentages.

Satisfying the Safety Regulations by **Mr A. J. Hinton, Hinton Safety Consultants, UK** Published in October 89 JOCCA

Mr J. Gent, Fulmer Yarsley: 1) What is a new substance? 2) How does it apply to polymers? 3) How long do you maintain written records for R&D purposes?

Printing Inks for the 90s

Joint Symposium organised by the Oil & Colour Chemists' Association and the Society of British Printing Ink Manufacturers

22 March 1990

Grand Hotel, Birmingham

- **The impact of ecology on the development of printing and packaging inks**
- **Challenge of newspaper printing to ink makers**
- **Radiation curing — Where does it fit in?**
- **Will lithographic inks continue to meet the demands of high speed publication and packaging printing?**
- **New pigment developments for printing and packaging inks**
- **Resin developments for tomorrow's inks**
- **Developments in screen printing**

Contact the Oil & Colour Chemists' Association for further details.

Mr A. J. Hinton: 1) A new substance in the EEC is one which is not listed in the European Inventory of Existing Chemical Substances (EINECS Inventory). 2) In the current regulations, polymers are considered as having been notified (i.e. exempt from notification), unless they contain 2% or more of a new "monomer" (the proposed 7th amendment changes "monomer" to "substance"). Basically, therefore, a polymer counts as a new substance as is subject to notification requirements if it contains, in combined form, 2% or more of a new substance. 3) If you consider the Consumer Protection Act, which incorporates the EEC Directive on Product Liability, there is an implied need to maintain records on any specific batches of substances to which persons may be exposed for a period of at least ten years after they have been supplied.

Mr J. E. O. Mayne, Dept. Materials Science & Metallurgy, Cambridge University: What is a toxic substance? Water if taken in sufficient quantity is toxic.

Mr A. J. Hinton: 1) A "toxic" substance has certain very specific properties under EEC regulations. Being less specific, substances or preparations are "dangerous under the EEC regulations if they fall into any of a number of categories (such as "very toxic", "toxic", "irritant", "flammable", etc.) on the basis of test results or human experience. 2) Water does not classify as a dangerous substance under these regulations, although it has been argued that its lethal properties are inversely proportional to the height of the test species!

Mr D. W. N. Clayton, Crown Berger: 1) Whilst under notification, what is the maximum amount that can be supplied to individual customers for research purposes? 2) Can the supplied company place a product on the market which contains a substance which is the subject of notification?

Mr A. J. Hinton: 1) New substances placed on the market at less than 1 tonne/annum for research and analysis purposes and intended solely for laboratories are exempt from notification requirements (Article 8(1), 3rd indent, Directive 79/831/EEC); Although there is some ambiguity in this exemption, it is generally interpreted as referring to chemicals used for laboratory-conducted research and analysis only and not to those used to produce a subsequently-marketed product. In theory, reduced notification requirements also apply for 12 months only to new substances marketed at more than 1 tonne/annum for the purpose of research and development. The notifications must be made to the Competent Authority of every country where you wish to carry out the R&D, and, in practice, the liberal interpretation by many C. A.'s that the notifier "complies with any conditions imposed" by the C. A. has led to a considerable lack of harmony in the requirements of different authorities; this, in turn, has made it almost impossible to obtain such a clearance in some Member States, short of placing in a full notification dossier. 2) Until the new substance has been fully notified, a company supplied with it under any of the exemptions or derogations is no more free to market it, either alone or in a preparation, than was the original notifier or supplier. The original supplier should make it clear to his customer that he is supplying a new substance which is not yet fully notified. ■



Benevolent Fund

Members of the Association will be aware of the long standing negotiations that have been undertaken with the Customs & Excise Department to exempt OCCA membership subscriptions from VAT and to reclaim the VAT paid on past subscriptions.

Provisional agreement was reached some twelve months ago for membership subscriptions to be exempt from VAT and thus enabled the 1989 subscriptions not to carry this tax. Full approval has now been granted for this exemption and the 1990 subscription notices currently being prepared for dispatch will not include VAT. The full member rate of £40.00 for 1990 will, therefore, show a saving of £6.00 for all those members unable to recover VAT and will still be only 90p greater than the 1988 subscription of £34.00 plus VAT.

I am very pleased to report that the Customs & Excise have now also agreed to repay a significant amount of the VAT previously paid on membership subscriptions since the tax was first introduced in 1974. The Association had applied to recover the full amount paid, but technical difficulties in the application of VAT during the period 1974 - 1978 has restricted the repayment to 1979 onwards. The net result is that after allowing for the fees of the Association's professional advisers, some £40,000 will be available to the Association for the benefit of its members.

Council recognises that it would be impossible to identify the individual members and companies to return the VAT previously paid

and has, therefore, decided to use the money to establish a Benevolent Fund to assist members in hardship. Recommendations will go to Council in February concerning the operation and control of the Fund and the precise details have not yet been determined. However, it is likely that only the interest from the Fund will be used, although repayable loans from the capital base cannot be ruled out.

The principle and immediate benefit will be to subsidize the subscriptions of those members in financial difficulties who would otherwise be unable to continue with their membership. Opportunities will arise for members and companies to contribute to the Fund and the Fund may be broadened to be applicable to all persons within the surface coating industries.

The establishment of the Benevolent Fund is a further important step in the development of the Association and it gives me great satisfaction to be able to make this announcement.

BSI Committees

The Technical Committee has invited Peter Munn to join the Committee and to act as a focus for the Association's Standards activities. Peter has long been active in this area, including service on PA Committees and we hope this strengthening of our efforts in this area will result in more information on British, European and International Standards being made available to our members and for publication in *JOCCA*.

The Association still requires representatives on the following BSI Committees

- PVC/1/5 Red Lead Pigments
- PVC/27/1 Organic Finishes for Aluminium
- PVC/27/-/3 Zinc Phosphate and MIO Paints
- GME/29/1 Test Sieves
- GME/29/2 Test Sieving and other Sizing Methods

Please contact the General Secretary if you wish to serve on any of these Committees.

Long Serving Members

Council has decided to consider marking the service of those members whose membership extends over 25 years. A preliminary analysis of the membership database shows that by 1 January 1990, 90 members will have completed over 40 years, including 13 members with over 50 years, membership. Dr H. J. Stern of the London Section joined on 1 January 1925 and is the Association's longest serving member. A full list of members with 40 years or more service will be published next month. ■

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Bristol Section

Polysulphide technology

The first lecture of the 1989-90 session was presented on 26 September by Mr Tom Rees, Laboratory Manager of Thiokol Chemicals, a division of Morton Thiokol — the Raw Materials supply unit of Morton International. Mr J. C. Shaw chaired the meeting.

Recent Advances in Polysulphide Technology covered two main areas:
1 Modification of Epoxy systems
2 Modification of Water Thinned Systems.

The use and characteristics of these modifications were discussed and illustrated to the members and guests present plus the visiting General Secretary.

Following question time the vote of thanks for the lecture was given by past chairman Maurice Prigmore and the buffet was kindly provided by Morton International.

G. Fowkes ■

Manchester Section

PRA and its role in Europe

The Sections first technical meeting of the 1989-90 session was held on Monday 2 October, 1989, at the Mechanics' Institute, Manchester, where a paper entitled "PRA and its Role in Europe" was presented by Mr J. Bernie of the Paint Research Association.

The lecturer commenced by giving an overview of the paint industry worldwide, with production in the region of 12,000 million litres per year and £20 billion turnover. In the last 5-7 years there has been globalisation of demand, and major restructuring, with large companies looking for world products. With huge R&D expenditure, there is a large gap developing between major producers and medium and smaller companies, with medium sized companies under pressure to keep up.

The spread of sales worldwide for the major world producers was outlined, and the spread of sales in Europe for Europe's top 10 companies also illustrated. As Europe and North America are mature markets, there will be little increase in sales in these areas, and the main growth will be in South America, the Far East, and China.

Technical challenges facing the paint industry were outlined with a trend towards cheaper, plentiful, and environmentally friendly raw materials anticipated, with the big multinationals being the pace setters.

The lecturer continued by outlining the PRA organisation, and the services it offers were outlined in detail. One way smaller companies can compete with the multinationals is by the use of PRA services, and particularly by being involved in collaborative projects, the funding of these being outlined in detail, with particular examples being used to illustrate the system.

There are a number of comparable organisations to the PRA in Europe, but these are all smaller and do not offer the same range of services. There is a large amount of E.E.C. cash available to fund research in Europe, and attempts have been made to introduce corporate projects in Europe, but they are very difficult to instigate, and there is considerably delay in obtaining E.E.C. financing. Current PRA projects in Europe were outlined, with PRA's role in them illustrated, current fund sources outlined, and possible future projects proposed.

The lecture was followed by a prolonged, and lively question and answer session, and the vote of thanks proposed by David Love.

Printing inks in perspective

The Sections first printing ink meeting of the 1989/90 session, was held on Monday, 16 October, 1989, at the new venue of "Smithys", Longbarn, Warrington, the usual venue of the "Silver Birch" not being available due to redecoration work. The session was attended by 40 members and guests, and a paper entitled "Printing Inks in Perspective, Some thoughts on Future Development" was presented by consultant Geoff Hutchinson.

In the past 15 years the priorities ink manufacturers have had to face, have been faster and faster presses, increased variety of substrates, and improving the efficiency of manufacturing plant. The 1974 Health and Safety at Work Act put pressure on safety as regards the handling of inks, and brought about an increased awareness of environmental pollution. The direct result of antipollution legislation in the USA has been the development of UV inks, and similar developments can be anticipated in Europe, the new COSHH regulations in particular, putting increasing emphasis on health and safety.

Statistics showing the current position regarding printing ink production and value in Europe compared with the USA and Japan were outlined, the effect of 1992 in Europe speculated upon, and taking UK figures alone, some suggestions regarding future trends made. Expected changes in methods of printing newspapers, and the changes required in newspaper inks,

were discussed, in future, greater attention to improving colour, and perfecting the flexographic system were anticipated. It was anticipated that quicker setting inks for offset Litho would become available. IR setting in offset printing is becoming quite widely used, and a theory for the mechanism involved put forward. The use of water-based varnishes over litho inks to prevent sticking is becoming common, and new devices for applying these varnishes were outlined. Heat set in web offset is expected to continue to develop with faster and faster print speeds with lower temperatures being used, and advances in plant design for this were illustrated. UV curing is the most significant technical advance of the last 20 years, but the cost of the inks has precluded its widespread adoption. Considerable development in this field is anticipated, with a reduction in the price of the inks expected.

There are a wide range of raw materials available for gravure and flexographic inks, but the solvents used are very smelly. Ethanol is the most acceptable, but there are no resins which are purely alcohol soluble. The air pollution regulations concerning solvent emissions in Germany, were outlined, and two systems of solvent recovery which could be used, were described. The UK industry is not affected by these regulations, but they are likely to become a factor in 1992. The US regulations created research in the areas of UV, 100% solids, and water based, and this is likely to occur in the UK and Europe also. There are a number of problems which have to be overcome before water-based inks can be made to work in a similar way to solvent based systems, these problems were outlined, different methods of drying inks described, and future resin systems suggested.

The whole tone of the lecture was to be somewhat controversial, with a view to creating discussion, this the lecturer provoked, the presentation being followed by a lively discussion period of some 30 minutes, with the vote of thanks proposed by Bob Kinkead closing the evenings proceedings.

M. G. Langdon ■

Newcastle Section

Rheological agents

Forty members and guests attended the first meeting of the 1989/90 session at St Mary's College, University of Durham, to hear Dr Ralph Schwindt of NL Chemicals talk on "Modern Rheological Agents for the Paint Industry".

Dr Schwindt began by reviewing the

structure of bentonite and hectorite minerals as sheets of silica separated by layers of alumina or magnesia, with some isomorphous replacement of aluminium by magnesium, and magnesium by lithium, electrical balance being maintained by additional alkali/alkaline earth metal cations. The organoclays sold to the paint industry are made by treating bentonite/hectorite with special quarternary ammonium chlorides which contain a C₁₈-C₂₀ chain covalently linked to the nitrogen. The basic sheet, or platelet, structure is maintained, but the substitution by ion-exchange the bulky organic cation increases the inter-platelet separation and allows for further separation and the formation of smaller platelet stacks and single platelets during dispersion in paint media.

The rheological effects of organoclay dispersions stems from the absorption of water, either added or contained within any pigments used, which results in swelling, together with gelation due to hydrogen bonding between water and available hydroxyl ions along the platelet edges. The dispersion increases fluid viscosity by the viscoelastic inter platelet attractive forces, and thixotropy from the degree of hydrogen bond breakdown according to applied shear rate and shear time.

Dr Schwindt showed some excellent slides illustrating the various stages of de-agglomeration with shear and wetting and subsequent gelation through further high-shear incorporation of chemical activator with water. These slides followed from conclusions drawn from infra-red absorption and X-ray diffraction analysis of dispersions made by different techniques and reported at FATIPEC in September 1988. A summation of such work suggest that:

1. Water is essential for gelation
2. Chemical activators such as methanol or propylene carbonate can help to make water more accessible to a system, but can act as degellants — propylene carbonate is more of a problem here, but methanol has the disadvantage of being low-flash and more toxic.
3. With some paint media e.g. alkyds, no chemical activator is needed — they accept water at sufficient shear.
4. Surfactants are often degellants during paint storage.

Dr Schwindt recounted the case of xylene from an external storage tank being used successfully in an organoclay gel before Christmas 1986, but being unsuccessful after Christmas 1986. There had been a sharp drop in the temperature during that Christmas and all traces of suspended water had

frozen out of the xylene. That this was the reason for the different performance was demonstrated by the addition of one drop of water to a small sample under high shear and the formation of a gel.

Arising from the derived knowledge of a dispersion/gelation mechanisms, new more easily dispersed and gelled organoclays were made available in recent years. Versions exist for different solvent/resin systems which require no activation additive; only 3% of water after initial dispersion, based on the weight of organoclay. Experience since their introduction has shown that these organoclays are not always direct substitutes for the older types and both complement one another. One area where neither type can be used is with moisture-cured urethanes, for obvious reasons.

Turning to alternative thixotropes, Dr Schwindt discussed the hydrogenated cator oil (HCO)-based materials. He showed a number of slides illustrating the mechanism of de-agglomeration and dispersion by shear and heat to the fully dispersed and active state. The process is dependent on shear rate/shear time and temperature/temperature duration and is much more sophisticated than with organoclays. Excessive temperature/time can easily lead to solution of the thixotrope, which then re-crystallizes on cooling, forming large 'seeds' unless continually mixed during the cooling process. Correction of seeding can be very time consuming and is a serious problem in large scale production.

Modern developments of alternatives to the HCO-type thixotrope have produced good results with a polyamide type: this is also activated by high shear and specific temperature attainment, without any seeding effect if excessive temperature/time conditions occur. Unfortunately, it is not effective with polar solvents, such as alcohols: it is very effective with hydrocarbons and its performance in chlorinated rubber paints is excellent.

Other widely-used thixotropes are the polyolefin wax paste additives, which also require high shear and heat to activate. In some systems they are surprisingly good in producing very stable rheological structures.

Summing up, Dr Schwindt stated that all thixotropes need the input of individual paint chemists according to the paint product involved. NL Chemicals are currently working on liquid products with a different activation chemistry—with waterborne coatings glycols are needed for activation.

Question time covered the max/min

water contents for organoclays, temperature effects on organoclay dispersion, organoclays versus fumed silicas, and the problems of HCO thixotropes in paints for beadmilling or for application on hot steel surfaces e.g. tanks in Saudi Arabia. Afterwards an excellent buffet with refreshments was provided by NL Chemicals.

J. Bravey ■

Thames Valley Section

Works visit: Volstatic Ltd

The first meeting for the 1990 session was held at Volstatic Ltd, Slough on 14 September 1989. The meeting was preceded by a buffet kindly provided by Volstatic Ltd.

After a brief introductory talk on the evolution from the 1950's to present state application and basic principles of electrostatic powder coating, we were taken on a tour of the development laboratory. Here we were given a demonstration of the high voltage (c. 80000 V) low current (c. 50 micro amps) and high safety factors required.

We were then taken to the demonstration room where we were shown the three main systems. The first was a fully automated, conveyor, system shown in operation. Also demonstrated were the associated systems for powder collection, recovery and recycling.

The second system demonstrated was a smaller, manual, system where we were given the chance to show our expertise in spraying items ourselves.

The third system shown was totally portable, and of large briefcase size, ideal for laboratory evaluation and customer demonstration work.

The cleanliness of the operation, and reduction of hazardous waste and solvent emission, was apparent to all.

The evening was concluded with our retaining a small sample of the work demonstrated, and a lively question time. Further demonstrations on the toughness of finished articles, and developments in speedy colour-change down-times were also discussed.

The vote of thanks was proposed by R. Stephens at the close of a very successful first meeting of this session.

D. Horn ■

Successful wood coatings symposium

Over 100 delegates and speakers attended a one-day symposium organised by Thames Valley OCCA on "Decorative and Protective Coatings for Exterior woodwork" held at the

Building Research Establishment (BRE), Watford on Thursday, 12 October. The symposium attracted delegates from a very wide background including manufacturers, specifiers (especially from County Councils) and end-users.

Mr J G Sunley, Consultant and former Director of TRADA chaired the symposium. In his opening remarks he outlined the major developments that have occurred in wood finishes over the last forty years stressing the change from on-site finishing to off-site pre-finishing. He then introduced Dr E. R. Miller, Finishes Section BRE, who spoke on the "Characteristics of Wood as a substrate". Dr Miller outlined the structure of wood and then discussed the differences between hardwood and softwood. He described the characteristics of wood as a substrate: Texture, Porosity, Extractives, Moisture, Absorption, Dimensional Instability and Photodegradation. Dr Miller then went on to describe the apparatus developed at the BRE for the determination of water absorption. From this research he stressed the importance of sealing the end-grain of wood to prevent water entering through the joint leading to the consequent deterioration of the wood. There then followed a lively discussion where Dr Miller discussed his controversial statement that sawn timber would have better performance than planed timber as a substrate for coatings. Wood coatings manufacturers in the audience felt that for aesthetic reasons it would be difficult to persuade consumers to use sawn rather than planed timber. One specifier in the audience also voiced criticism that joinery manufacturers refused to comply with his specification for timber articles especially with regards to the sealing of end grains of wooden windows.

The second speaker of the morning session was Dr J Carey of the Timber division of the BRE who spoke on "Micro organisms for coatings Performance". Micro-organisms have the potential to affect coatings performance in several ways:

- cause disfigurement of the coating
- cause physical deterioration of the coating
- affect the wood beneath the coating and this indirectly affects performance.

The organisms responsible can be bacteria, fungi, yeast or algae. Dr Carey sub-divided the fungi into several sub-groups and discussed the effects of each sub-group on wood. Algae and mould fungi are usually responsible for surface disfigurement of coatings whereas the deterioration of the coatings is usually associated with fungi. Once the coating is penetrated by water the underlying



Thames Valley Wood Coatings Symposium Speakers and Organisers (o) — L to R: F Sykes, R Tuckerman, J Carey, D Horn (o), J Worringham, E. R. Miller (o&s), G Drinkwater (o), J. G. Sunley, M. Palmer, R Stephens (o) and J Gant (o).

wood becomes wetted and colonisation by micro-organisms quickly follows. For a coating to perform at its best on exterior woodwork it is important that growth of micro-organisms is controlled either by the exclusion of water and/or effective biocidal treatments.

There then followed an animated discussion where a questioner asked if there was any biological treatment that could be used to interfere with the colonisation sequence of wood. Dr Carey replied that it was difficult to stop colonisation but there were a number of new preservative methods:

1. Many new more environmentally-friendly toxic alternatives.
2. A gel-type reservoir of preservative only activated when the timber becomes wet.
3. Wood being chemically treated, but this is expensive i.e. wood fittings for kitchens utensils. The problem with this treatment is that it allows water in.

Reference was made in both BRE lectures to a number of useful relevant BRE publications on "Paints and Surface Finishes". These are available from Publication Sales, BRE, Garston, Watford, Herts WDZ 7JR.

Following the coffee interlude Mr J. Graystone of ICI Paints spoke on the "Standardisation of Wood Finishing systems and Future Effects on the UK Market".

Mr Graystone commented that in previous years there has been no acceptable general standard for wood finishes leading to confusion caused by product proliferation, performance claims and mixed terminology. Mr Graystone said that the situation improved with BS 5358 — a standard for a solvent-based wood primer and BS 5082 — a standard for a water-borne

wood primer. The importance of these standards is that they are performance-based. With this in mind he then went on to discuss the philosophy behind a new standard introduced last year — BS 6952. The philosophy behind the new standard is that it is user-driven, no single product is singled out and that it is a performance standard. The sections of the standard are:

1. Scope and Definitions
2. Terminology and Performance Factors
3. Classification Proposals

The standard will enable:

- a description by the user of factors such as appearance and intended end-use situation
- the manufacturers confirm that the product has the desired appearance and be recommended for the proposed use.

The classification would depend on:

1. The Appearance which is made up of 36 combinations i.e. build (4 levels) × opacity (3 levels) × sheen (3 levels)
2. The Exposure Condition: sheltered, average, exposed
3. The End Use: Free movement, some control, minor movement
4. The Substrate Condition: new untreated wood, weather-degraded wood, preservative-treated wood, factory-primed timber, weathered coating.

The overall idea of the standard would be to finish up with a manufacturer's Product Information Table.

BS 6952 is available from BSI Sales, Linford Wood, Milton Keynes MK14 6LE.

Mr Graystone then went on to discuss the current European Standards for wood finishes. CEN is the body

involved with the preparation of European Standards and is made up of members from the EEC and EFTA which comprises over 16 member countries. The CEN Paint and Varnishes Technical Committee is CENTC 139 which has a Working Group WG2 on wood coatings. It is currently discussing the adoption of BS 6952 as the basis of a European Standard.

Following lunch, Mr J. H. M. Worringham of Cuprinol Ltd spoke on the Progress in Natural Finishes for Wood. Five years ago there were four types of wood finish: Wood preservative, varnish, oil and wood stains. In the last five years there have been major changes in wood finishes. In his paper Mr Worringham concentrated on wood stains discussing new developments such as high-film build stains, water-based wood stains, transparent and synthetic iron oxide wood stains, and non-drip stains.

The second lecture of the afternoon was given by Mr F. C. Sykes of Akzo Coatings who spoke on "Factory Applied Joinery Finishes", stressing the growth of off-site prefinished and finished joinery (especially windows). He also commented upon the need to improve site practice. Mr Sykes illustrated numerous factory finishing installations and outlined the important advantages of factory finishing for manufacturers and specifiers.

The last talk of the afternoon session was given by Mr R. Tuckerman of ICI Paints who spoke on Exterior Decorative Coatings for Wood. In his wide-ranging talk he described the substrates and construction for US housing.

In the US wood is used as a cladding, trim and joinery material in housing construction. From 1985 to 1987 the major siding (cladding) material for new US housing is summarised below:

	1985 (%)	1987 (%)
Plywood	14	15
Hardboard	26	20
Lumber siding	6	12
Brick	18	18
Stucco	13	14
Aluminium	11	6
Vinyl	6	11

Mr Tuckerman then went on to discuss the consumer, product and future trends of the US Wood Coatings market.

Mr Sunley closed the meeting at 4pm and thanked the symposium speakers and Thames Valley Section organising committee.

It is planned to publish a selection of these papers in future issues of JOCCA.
P. J. Fyne ■

Transvaal Section

Marketing

On 20 September 1989 the Section was addressed by Mr H. L. Starik, Divisional Manager of the Agfa-Tech Division of Bayer South Africa on the subject of Marketing.

Compared to many professions marketing is very young despite the fact that successful manufacturers have used marketing principles for centuries.

At the turn of the century demand for manufactured goods exceeded production so producers concentrated on increased capacity. The two World Wars brought about a continuation of consumer demand that continued through the sixties. However by the seventies supply outstripped demand and indications now point to a society concerned with quality and the individual, factors that require marketing.

At the base of a marketing philosophy, is the so called 'Product Life Cycle' which distinguishes four stages in the life of a product. These are an Introductory Phase, Growth Phase, Mature Phase and finally the Decline Phase.

It is at the Mature Phase that good marketing can make most difference to a product's future because two possible developments can take place. A successful modification of the original product can bring about a new life cycle or alternatively the product becomes a 'lame duck' that slowly declines into oblivion.

Marketing is the philosophy that recognises that it is the Consumer who decides on the future of a product. Therefore the producer must define the consumer and thereafter satisfy the consumer's need better than competitors. Marketing therefore recognises that:

1. company activities start and end with the consumer
2. the consumer's needs must be identified
3. all resources must aim at customer satisfaction
4. continuous innovation must maintain advantages over competitors.

For companies to succeed it is essential that they recognise that "Marketing Means Better Business".

After an active 'question and answer' period the vote of thanks proposed by Mr R. E. Cromarty was heartily endorsed by the audience.

R. E. Cromarty ■

**JANUARY JOCCA:
CORROSION**

OCCA News

OCCA Golf

This years OCCA National Golf Trophy was played for at Cannons Brook Golf Club, Harlow under bright blue skies. A fairly strong breeze kept temperatures down, but made scoring difficult. The course was playing short after the prolonged dry weather, however the greens were in excellent condition despite this.

At the end of an enjoyable day play the winner of the National Trophy was Len Jennings of Croxton + Garry Ltd with a creditable 32 Stableford points under the difficult conditions. The Valentine Trophy for London Section members only was played for at the same time. Since Len Jennings of Croxton + Garry Ltd is a member of London Section he was able to defend the Valentine Trophy for the second year running.



Len Jennings

West Riding Golf

The West Riding Golf Trophy was once again competed for at Wetherby Golf Club on Thursday, 12 October 1989. The weather was reasonably kind and some fairly high scores were obtained. Unfortunately the greens were not in good condition due to the long dry summer which led to a certain amount of frustration. However, 33 members and guests completed the round with an excellent day's play.

The top scorer was Jim Bury with 42 points. The Trophy itself however was won by the defending champion Roger Wells with 39 points.

A particular feature of this competition is the number of beginners we attract, all of whom scored at least some points.

The day was rounded off with a good meal and the usual presentation of prizes. Our thanks to Pete Stanton, our Social Secretary, for the organisation and for Rob Lewis' hard work on the day.

G. C. Alderson ■

Manchester Section

Quiz Night

The Sections Annual Quiz Night was held at the Silver Birch, Birchwood Centre, Warrington on Monday, 18 September 1989. One hundred members and guests attended the event this year, making up 25 teams from 16 different companies. The quizmaster this year, by popular demand, was again John Bennett, with subjects ranging from Sport, Geography, TV and Films, to Literature, Mythology and Famous Women.

Freeman Chemicals "B" team were the winners (see full results table), with Crown Berger in second place, and Freeman Chemicals "A" team 3rd. Last years winners, Marcell Guest failed to hold onto their title, but again McPhersons Paints provided the strongest team, McPhersons "A" team picking up the booby prize.

Manchester Section would like to thank all the companies who donated prizes, and helped yet again to make this a most successful and enjoyable event.

Section Chairman's & President's Reunion Weekend



OCCA 1984-85 Reunion Weekend (L to R): Top row — Hugh Young: Chairman West Riding, Cecil Finlay: President, Ian Bollam: Chairman Newcastle, Roger Saunders: Chairman Bristol, Eric Wallace: Chairman Midlands, Gordon Robson: Chairman Manchester; Bottom Row — Shirley Bollam, Morfydd Saunders, Jean Finlay, Joyce Robson, Norma Wallace.

A very happy fifth 1984-85 Section Chairman & President's Reunion Weekend was held at the Alveston Hall Hotel, Nantwich, Cheshire.

We all arrived late Friday afternoon and quickly set up our own bar in an adjoining conference room and commenced a relaxing and social weekend — excellent food — a really gourmet weekend and wonderful company! Saturday was warm and sunny and during the morning we

ventured along part of the Sandstone Trail from Beeston Castle, before finishing up at the Copper Mine for bar snacks. Ian Bollam found a new route by car across the ridge along an unsurfaced track "Not suitable for Motor Vehicles" as recommended by one of the locals which was exciting to say the least! We spent the afternoon in near by Candle Workshops and at the Staircase Locks on the Shropshire Union Canal — altogether a splendid day rounded off by as swim in the hotel pool and a dinner dance (see photo).

Sunday we visited Staveley Water Gardens before taking our leave after lunch. Once again a superb weekend. Previous years we have visited the Last Drop Hotel at Bolton on three occasions and also the Dunkenhagh Hotel at Clayton-le-Moors, Accrington.

Next year it is Cecil Finlay's turn to arrange the weekend so we look forward to seeing what is in store for us.

G. Robson ■

News of Members

Australian member wins defence award

Larry Hill, an Australian Member (Overseas Section) was recently awarded a Defence Science and Technology Award for Excellence in Defence Consulting. Since 1962 Larry has been employed at Materials Research Laboratory (MRL), Melbourne where he handles coating

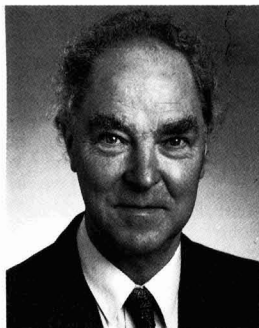
OCCA Manchester Section Quiz Night

Full Results

1st	Freeman Chemicals "B"	192 points
2nd	Crown Berger	183 points
3rd	Freeman Chemicals "A"	180 points
4th	McPherson Paints "C"	169 points
5th	Usher Walker	167 points
6th	Ciba Geigy "A"	160 points
7th	Process Colours	148 points
8th	Coats Lorilleux "A"	145 points
9th	McPherson Paints "B"	141 points
10th	Coats Lorilleux "B"	137 points
11th	BASF Inks "A"	131 points
12th=	Weilburger UK	130 points
12th=	Marcel Guest "A"	130 points
14th	Leigh's Paints	128 points
15th	Ellis Everard Specialties	127 points
16th	Ciba Geigy "B"	116 points
17th	Tioxide and others	115 points
18th	BASF Inks "B"	113 points
19th	Foscolor	112 points
20th=	Swale Winnet	110 points
20th=	Marcel Guest "B"	110 points
22nd	Johnstones Paints "A"	102 points
23rd	BASF Inks "C"	99 points
24th	Johnstones Paints "B"	89 points
25th	McPherson Paints "A"	73 points

M. G. Langdon ■

problems for the Australian Defence Services. Apart from a short break, occasioned by the formation of Australian OCCA, Larry has been a member of OCCA since 1949 and has contributed several papers to the Journal. He is also an Honorary Member of OCCA Australia, having been the Honorary Editor of the Australian Journal *Surface Coatings Australia*, since 1967.



Larry Hill

Prior to his joining MRL, Larry worked in the paint industries of England and South Africa. His first manager was the late Norman Arthur Bennett, then Technical Director of Griffiths Brothers, Bermondsey. Norman Bennett was President of OCCA 1957-9 and Griffiths Brothers was the first paint company to exploit the benefits of micaceous iron oxide. Many famous structures throughout the British Commonwealth are coated with MIO systems including Sydney Harbour Bridge.

Larry is an FSCT of both UK and Australian OCCA and is a member of the Professional Gratings Committee of OCCA UK.

Memorium

Sefton Nevil Hawley
(1925-June 1989)

Mr S. H. Codd writes:

Sefton was a friendly, lively personality, of whom it could be said, "if he couldn't do you a good turn — he certainly wouldn't do you a bad one", and indeed, he was a loyal friend and colleague who was always ready to do a good turn.

As a child he went to the Herbert Strutt School, Belper, and on leaving he joined the family firm of W. Hawley & Sons, until the time came for him to join the RAF, where he served as a pilot/navigator, trained in Canada and

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served in India and the Middle East, attaining the rank of Flight Sergeant.

After the war he became a Ground Instructor at Great Hucklow Gliding Club.

In 1954 he was given an award for bravery when he rescued a young boy who had fallen through the ice into the river Ecclesbourne at Duffield.

His career at work was that he started as a Laboratory Technician, followed by a move into production on the factory floor. In 1946 he became a Company Director. At that time they were making distemper for Woolworths, amongst other things.

He was made Joint Managing Director in December 1951, along with his brother, Donald. In 1973 he was Managing Director, and in 1984 he became chairman and Managing Director.

In 1963 he joined with eight others to form the Committee of the Trent Valley Branch of the Midlands Section of OCCA at the Inaugural Meeting of 24 October 1963. He worked as a Committee Member, becoming Social Secretary, then in 1971 he was made Treasurer. This was followed by a stint as Vice Chairman until, in 1976, he became the Branch Chairman and served in that capacity until 1978.

Many will remember the OCCA Socials which he helped organise and the fun he created. Fun and laughter were never far away on these occasions.

Perhaps his motto, "Life is not a rehearsal — you only have one chance" explains his life. Certainly, he put a lot into life.

Sefton left a wife, Joan Elizabeth, and a son, Jeremy, and a daughter, Karen.

It is intended to call future February Lectures "The Sefton Hawley Memorial Lecture", the first of which will be given by Colourgen, entitled "Instrumental Colour Measurement of Metallic Coatings" and sponsored by W. Hawley.

Obituary

Mr. M. H. M. Arnold

It is with regret that we announce the death of Michael Arnold; following a short illness he died at Moore in early October at the age of 74. He continued to operate his chemical consultancy business, Arnold Services of Moore and North America, until a few days before his death.

Michael Arnold, born in Crosby, was a renowned specialist within the Chemical, Paint and Food Industries throughout the world; he is also remembered as an author of many papers and as a past Honorary Editor of

JOCCA.

His career, starting with ICI Billingham to initiate their biological research programme, moved into the Paint and Resins Industry with Postans Ltd., of Birmingham where as Technical Manager he installed its synthetic resin manufacturing plant and set up the industrial paint development section. He encouraged new members to the industry through Postans paint apprentice scheme. He was always a supportive member at OCCA Midlands Section meetings, helping sensible and lively discussions to proceed.

After ten years at Postans Ltd, he moved on to Bowmans Chemicals of Widnes where he was appointed Technical & Managing Director and where he took their product range forward in lactic acid, malic acid and glucoheptonates manufacture to a prominent position with Europe.

More recently Michael set up a successful consultancy business established as a world authority on sugar acids. This called for travel and assignments in all regions, many of these on behalf of UNIDO into the third world areas.

Michael is survived by his wife Edith and two sons, to whom our thoughts go out at this trying time.

New Members

Ordinary members

Atkinson, K. (*West Riding*)
Broderick, T. P., BSc (*London*)
Campbell, D. J., BSc (*Newcastle*)
Duncan, I., BSc (*West Riding*)
Elbasir, A. A., MSc (*General Overseas - Libya*)
Fitzpatrick, R. J. (*West Riding*)
Houston, D. R., BA (*Ontario*)
Nurrish, C. M. (*Cape*)
Poyner, A. H. (*Manchester*)
Roberts, J., BSc (*Cape*)
Strydom, C. G., BSc (*Cape*)
Wood, P. L. (*Midlands - Trent Valley Branch*)

Associate members

Adam, D. W. (*Scotland*)
Morgan, W. D. (*Transvaal*)
Walton, R. J. (*Manchester*)
Warburton, B. N. (*Transvaal*)

Registered student

Bourne, R. G. F. (*Midlands-Trent Valley Branch*)
Kirk, L. (*Manchester*)
Scott, A. P. (*Manchester*)
Slater, P. W. (*Manchester*)

Professional Grade Register

List of successful candidates

As laid down in the report of the Working Party on Education, Training and Qualifications which was adopted on the institution of the Professional Grade, a list of those members in the Grade is published in the December issue of the *Journal* each year. The 18th such list appears below and includes the names of members resident in 35 countries.

The sections to which members are attached are given in italics.

Fellows

- Addenbrooke, Brian John (*Midlands*)
 Anneveldt, Jan Johan Willem (*Natal*)
 Apperley, Thomas William James (*West Riding*)
 Arbuckle, Kenneth Harold (*London*)
 Archer, Harold (*Manchester*)
 Ashworth, Norman (*Manchester*)
 Astfalck, Anthony Noel (*Transvaal*)
 Atherton, Donald (*Scottish*)
 Bailey, John Noel (*Newcastle*)
 Banfield, Thomas Arthur (*London*)
 Barrett, Ronald Leon (*Scottish*)
 Bayliss, Derek Arthur (*London*)
 Bester, Lawrence Percy (*Natal*)
 Bhumkar, Chidanand Jayram (*General Overseas – India*)
 Birrell, Peter (*Ontario*)
 Bishop, Eric Harold Abbott (*West Riding*)
 Boroky, Joseph Stephen (*General Overseas – Australia*)
 Bose, Sunil Kumar (*London*)
 Bosman, Herman Izak (*Transvaal*)
 Bourne, John Robert (*Midlands – Trent Valley Branch*)
 Boxhall, John (*Thames Valley*)
 Bridle, Peter Frederick (*London*)
 Brooks, Leo James (*London*)
 Brown, Arthur Ernest Girdlestone (*London*)
 Burwell, Barry (*Manchester*)
 Butcher, George Alfred (*Midlands*)
 Butcher, Kenneth William George (*Manchester*)
 Butler, Cecil (*West Riding*)
 Byrns, Arthur Robin (*Cape*)
 Caffery, George Francis (*London*)
 Campbell, George Alexander (*Manchester*)
 Canterford, Barry Albert (*London*)
 Carr, William (*Manchester*)
 Carter, Eric Victor (*Newcastle*)
 Churchman, Anthony Edward (*London*)
 Clarke, Harry James (*Midlands*)
 Clement, Donovan Harry (*Midlands*)
 Cole, Derek (*General Overseas – Australia*)
 Collier, Claude William (*Midlands – Trent Valley Branch*)
 Collings, Arthur Geoffrey (*London*)
 Coupe, Raymond Richard (*London*)
 Courtman, Frank (*Manchester*)
 Coverdale, Peter Frederick Muir (*Midlands*)
 Cowie, Edward Bruce (*General Overseas – Singapore*)
 de Jong, Jan Lauwrens (*Transvaal*)
 Dowsing, George Frederick (*London*)
 Draper, Patrick Albert (*Natal*)
 Duligal, Eric Arthur (*Transvaal*)
 Dunkley, Frederick George (*Midlands – Trent Valley Branch*)
 Easton, James Douglas (*General Overseas – USA*)
 Eglington, Roland Alexander (*Natal*)
 Ekeh, Godwin Chukoemeka (*General Overseas – Nigerian Branch*)
 Finn, Stanley Russell (*London*)
 Froggatt, Joshua John (*London*)
 Fullard, John Edward (*Transvaal*)
 Furuhjelm, Victor Henrik (*General Overseas – Finland*)
 Garratt, Peter Garth (*General Overseas – Austria*)
 Gate, Peter Atholl Jackson (*Transvaal*)
 Geddes, Kenneth Raymond (*Manchester*)
 Giesen, Mathias Franz (*General Overseas – Germany*)
 Gilliam, Brian Frederick (*London*)
 Gillian, James Graham (*Manchester*)
 Gollop, Percy Lionel (*London*)
 Goyal, Ramkumar Bhanwarlal (*General Overseas – India*)
 Graham, Thomas (*Manchester*)
 Grainger, William Alan (*Irish*)
 Gray, Alan (*Ontario*)
 Greenfield, Eric (*Midlands*)
 Grey, James (*Ontario*)
 Haken, John Kingsford (*General Overseas – Australia*)
 Hamburg, Herman Rudolf (*London*)
 Hanson, Robert Philip (*Newcastle*)
 Hill, Derek Alfred Wheeler (*London*)
 Hill, Gilbert Victor Geoffrey (*Thames Valley*)
 Hill, Lawrence Albert (*General Overseas – Australia*)
 Hipwood, Hubert Allan (*London*)
 Hodgson, Kenneth Vickerson (*London*)
 Holbrow, Gordon Leonard (*London*)
 Hoppood, Arthur Thomas Robb (*London*)
 House, Dudley James (*Transvaal*)
 Hutchinson, Geoffrey Herbert (*London*)
 Inshaw, John Lesley (*Thames Valley*)
 Iyengar, Doreswamy Raghavachar (*General Overseas – USA*)
 Jacob, Basil (*Thames Valley*)
 Johannsen, Ralf Peter (*Cape*)
 Johnsen, Svend (*General Overseas – Denmark*)
 Jolly, Anthony Charles (*Manchester*)
 Kalewicz, Zdzislaw (*General Overseas – France*)
 Kane, Joseph Richard (*Newcastle*)
 King, Raymond John (*Midlands*)
 Kotwal, Hoshidar Peshotan (*General Overseas – Pakistan*)
 Kut, Sigmund (*London*)
 Landmann, Axel Wolfgang (*London*)
 Lasser, Howard Gilbert (*General Overseas – USA*)
 Lewis, Fred (*Manchester*)
 Ley, John Barry (*Thames Valley*)
 Lomas, Harold (*Ontario*)
 Lunt, Walter Richard (*West Riding*)
 Martin, Christian Pierre (*General Overseas – France*)
 McLean, Angus (*Scottish*)
 McMillan, Stanley (*Newcastle*)
 McQuirk, Peter John (*Midlands*)
 McRobbie, David Martyn (*London*)
 Mitchell, John Edmund (*Manchester*)
 Mitchell, Seward John (*Midlands*)
 Monk, Cyril James Henry (*Bristol*)
 Moon, William Robert (*Manchester*)
 Morgans, Wilfred Morley (*General Overseas – Spain*)
 Morpeth, Frederick Johnson (*Manchester*)
 Morris, David (*Manchester*)
 Munn, Raymond Henry Edward (*London*)
 Munro, Hugh Anderson (*Scottish*)
 Myers, Gordon (*Natal*)
 Newman, Derek George (*Thames Valley*)
 Newton, Dennis Sydney (*Bristol*)
 North, Albert Graham (*London*)
 Pace, Graham (*Thames Valley*)
 Patel, Shirish Manibhai (*Ontario*)
 Pettit, Michael Robert (*London*)
 Piggott, Kenneth Elliot (*Natal*)
 Polaine, Sidney Alan (*London*)
 Porteous, Barry Milroy (*Ontario*)
 Prigmore, Maurice Henry (*Bristol*)
 Raaschou Nielsen, Hans Kristian (*General Overseas – Denmark*)
 Rampley, Dennis Neil (*London*)
 Ray, Stanley Arthur (*Midlands*)
 Rechmann, Heinz (*General Overseas – Germany*)
 Redman, Frank Benson (*Manchester*)
 Reid, John Rodney Stanford (*Transvaal*)
 Raleigh, Albert Kenneth (*General Overseas – Australia*)
 Robson, Gordon Reginald (*Manchester*)
 Roe, David Edwin (*London*)
 Rouse, Robert Earnshaw (*Transvaal*)
 Rubin, Wallace (*London*)
 Rudram, Arthur Thomas Stephen (*London*)
 Saunders, Laurence Frederick (*Transvaal*)
 Seymour, Norman Henry (*Manchester*)
 Sharpe, Eric Edward Victor (*Transvaal*)

Simon, Raymond (*Irish*)
 Simpson, Lesle Ainsley (*Newcastle*)
 Slade, Harold Aitken (*London*)
 Smith, Eric (*West Riding*)
 Smith, Francis Mark (*Manchester*)
 Smith, John George Nixon (*Newcastle*)
 Stead, Graeme Rupert (*Transvaal*)
 Stoyle, Francis Wilbert (*Irish*)
 Stretton, Elizabeth (*Manchester*)
 Talbot, Ernest Alexander (*Newcastle*)
 Tate, Donald Howarth (*Newcastle*)
 Taylor, Alexander Legg (*Irish*)
 Taylor, John Roberts (*Bristol*)
 Thukral, Prem Sagar (*London*)
 Tooth, John Henry Collins (*London*)
 Turner, John Harry Wallace (*Manchester*)
 van Londen, Anton Matthijs (*General Overseas – Netherlands*)
 Walker, Peter (*Thames Valley*)
 Watkinson, Leonard James (*West Riding*)
 Weineck, Terrence Graham (*General Overseas – Portugal*)
 Whiteley, Peter (*Midlands*)
 Wilkinson, Thomas William (*Hull*)
 Willis, Gervase Hewitson (*Manchester*)
 Windsor, Frederick Barry (*Manchester*)
 Wood, George (*London*)
 Woodbridge, Richard John (*Bristol*)
 Worsdall, Herbert Charles (*London*)
 Young, Hugh (*West Riding*)

Associates

Abel, Adrian George (*West Riding*)
 Acey, John Arthur (*London*)
 Adams, John Charles (*Midlands*)
 Adams, Terry Ernest (*London*)
 Adefarati, Francis Babasola (*General Overseas – Nigerian Branch*)
 Akam, Richard Brian (*General Overseas – Hong Kong*)
 Amand, Henri Gordon (*Bristol*)
 Anthony, Alan Sydney (*London*)
 Archer, Robin Jeremy (*Natal*)
 Armstrong, Herbert Walter Maynard (*London*)
 Arnold, Alan Edward (*Midlands*)
 Arnold, Frank (*Manchester*)
 Ashton, Ronald (*Manchester*)
 Asiedu-Dompheh, Jonathan (*London*)
 Asubonteng, Samuel Kofi (*General Overseas – Ghana*)
 Attwood, Stephen Charles Jackson (*Transvaal*)
 Ayre, Thomas Richard (*Midlands – Trent Valley Branch*)
 Bains, Ranjit Singh (*London*)
 Baldwin, George William (*Manchester*)
 Bandyopadhyay, Basabjit (*General Overseas – India*)
 Bannington, Donald Bertram (*London*)
 Barnes, Peter James (*London*)
 Barry, James (*Midlands – Trent Valley Branch*)
 Batch, Alan James Edward (*London*)
 Beadle, Roy William (*London*)
 Bell, Brian Robert (*Midlands*)
 Bell, Eric James (*London*)

Benley, Major Gordon (*West Riding*)
 Berberi, Anwar Edmond (*General Overseas – Lebanon*)
 Bolam, Ion Barrow (*Newcastle*)
 Borer, Keith (*Newcastle*)
 Bourne, Jonathan Paul (*General Overseas – Switzerland*)
 Brooke, Leslie John (*Bristol*)
 Calderbank, John Thomas (*Manchester*)
 Campey, Lesley John Randall (*Ontario*)
 Carr, Robin Gordon (*Newcastle*)
 Cartwright, Jeffrey (*London*)
 Catchpole, David (*London*)
 Catherall, Kenneth David (*Midlands*)
 Chambers, Anthony (*London*)
 Chaudhry, Mudassar Iqbal (*General Overseas – Pakistan*)
 Chellingsworth, Horace Thomas (*Midlands*)
 Chippington, Kenneth Alan (*Bristol*)
 Chua, Ah Guan (*General Overseas – Singapore*)
 Clarke, Raymond John (*Ontario*)
 Clayton, David Walter Norbury (*Manchester*)
 Clausen, Hans Christian (*Natal*)
 Coates, John Allen (*Manchester*)
 Cole, Francis William (*London*)
 Constantinides, Erricos (*London*)
 Cordwell, Terrence Allan (*Midlands – Trent Valley Branch*)
 Cox, Garth Anthony (*London*)
 Cromarty, Ronald Earling (*Transvaal*)
 Cunningham, Robin Roy Carol (*London*)
 Dalton, Frank (*General Overseas – Denmark*)
 Davis, Brian Frederick James (*London*)
 Davis, Reginald Albert (*Bristol*)
 Dent, Graham John (*Manchester*)
 Devine, James (*Transvaal*)
 Donkersley, Brian (*West Riding*)
 Dooley, David Brian (*London*)
 Downham, Stephen Airey (*Manchester*)
 Drury, James Roland (*Transvaal*)
 Dunn, Paul Alan (*London*)
 Durdey, Alan James (*Newcastle*)
 Dury, Ian Clifford James (*Thames Valley*)
 Eaton, Michael George (*Thames Valley*)
 Egan, Muireann (*London*)
 Eylers, Petrus Gerardus (*General Overseas – Zimbabwe Branch*)
 Fairless, Joseph (*General Overseas – Netherlands*)
 Fell, Alan William (*Thames Valley*)
 Fernades, Larry Raphael Frances Joseph (*London*)
 Finlay, Cecil Newton (*Newcastle*)
 Fisher, Leslie Alexander (*Natal*)
 Flood, Geoffrey Terence (*Scottish*)
 Flynn, Roy (*West Riding*)
 Ford, Keith Sydney (*Manchester*)
 Formanek, Leopold (*General Overseas – Czechoslovakia*)
 Freund, Kurt Alfredo (*General Overseas – Ecuador*)

Gallon, Mark Robert (*Manchester*)
 Gascoyne, John (*Midlands*)
 Gaynor, Helen McDonald (*Transvaal*)
 Gibson, Anne McArdle (*Scottish*)
 Gibson, Frank (*Manchester*)
 Gibson, John Carrington (*Hull*)
 Greeff, Mattheus Philip (*Transvaal*)
 Green, Basil Ray (*General Overseas – Trinidad*)
 Green, Brian James (*London*)
 Griffiths, Henry James (*Midlands*)
 Gunn, David John (*Midlands*)
 Gunn, Reginald (*Bristol*)
 Halsall, Raymond (*Manchester*)
 Hamilton, Alexander (*Scottish*)
 Harris, Edward Murray (*Scottish*)
 Harrison, Cyril Geoffrey (*Hull*)
 Harty, David Basil (*General Overseas – Australia*)
 Hasnip, John Anthony (*Hull*)
 Hemmings, James (*West Riding*)
 Herriott, Charles Edward (*London*)
 Hester, Nigel Lewis (*Bristol*)
 Hickman, Edwin Peter (*Midlands*)
 Hill, Raymond Forsyth (*Scottish*)
 Hitchmough, Rex Henry (*London*)
 Holden, William Desmond (*Manchester*)
 Holmes, Peter Neville (*Irish*)
 Holt, Clifford (*West Riding*)
 Honiball, Alan Edward (*Scottish*)
 Horn, David Michael William (*Thames Valley*)
 Howarth, Graham Alistair (*Manchester*)
 Howes, Edward John (*London*)
 Hughes, Anthony Harold (*Manchester*)
 Hutchins, Robert John (*London*)
 Hutchinson, Martin Alfred (*West Riding*)
 James, Tudor Herbert (*Bristol*)
 Johnstone, James William (*Manchester*)
 Jones, Derek Frederick Arthur (*Thames Valley*)
 Kaye, Dennis David (*London*)
 Kenna, Frank William (*Manchester*)
 Kerr, Michael Anthony (*Manchester*)
 Kirkwood, Thomas (*Scottish*)
 Kirlaw, Charles Wesley (*General Overseas – USA*)
 Kitchen, John Robert (*Midlands – Trent Valley Branch*)
 Laker, Bernard George (*London*)
 Lakshmanan, P. R. (*General Overseas – USA*)
 Lander, Wilfred Terence (*Hull*)
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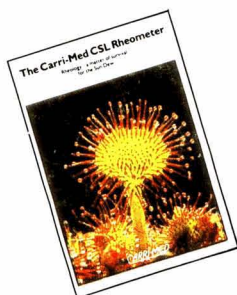
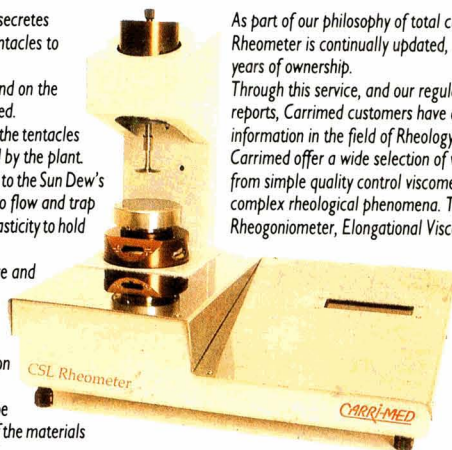
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