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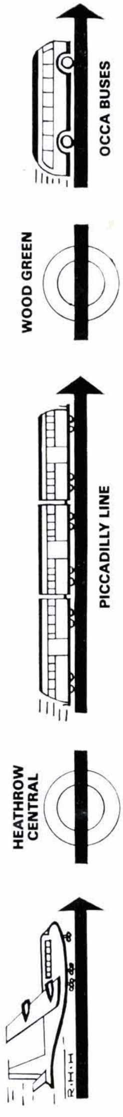

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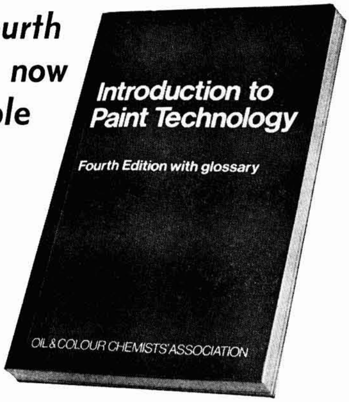

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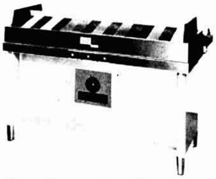

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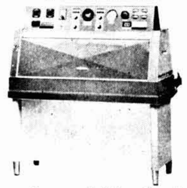

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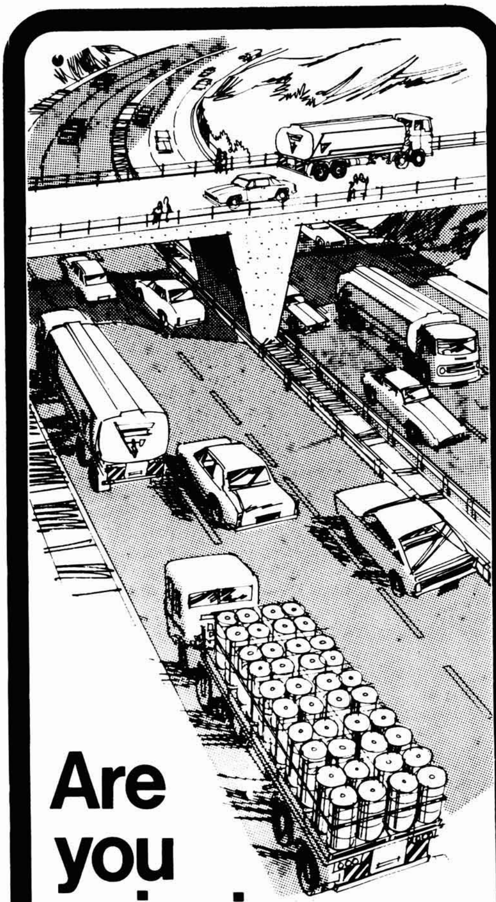

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Transactions and Communications

The UV curing of acrylate materials with high intensity flash*

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Summary

An examination has been made of the influence of photon flux and flux duration on the photoinitiated polymerisation of UV curing acrylates. Initial experiments with medium pressure mercury lamps showed that increasing photon flux density by the use of focusing elliptical reflectors gave an improvement in cure. The work was extended by using the very high photon fluxes available from xenon flash lamps.

Single and multiple flashes of light were used to cure a variety of formulations. The influence on cure, as measured by infrared spectroscopy, of photon flux density and pulse duration, is interpreted in terms of the elementary processes involved in free radical polymerisation.

Keywords

Raw materials for coatings
  binders (resins, etc)
    acrylic resin

Equipment primarily associated with drying or curing of coatings
    mercury lamps
    xenon arc lamps

Processes and methods primarily associated with drying or curing of coatings
    ultraviolet curing
    photoinitiation
    photon flux

Properties, characteristics and conditions primarily associated with dried or cured films
    viscosity

Le durcissement des revêtements à base d'acrylates au moyen des éclairs à haute intensité de rayons ultra violets

Résumé

On a étudié l'influence qu'exercent le flux photonique et la durée du flux sur la polymérisation photo-inititiée des acrylates durcissables par les rayons ultra violets. Les premières expériences, utilisant les lampes à vapeur de mercure à pression moyenne ont démontré qu'une amélioration du durcissement peut être assurée où l'on augmente la densité du flux photonique au moyen des réflecteurs élliptiques et de mise au point. L'étude a été étendue par l'emploi des fluxes photoniques très élevés qui sont disponibles grâce aux lampes-éclairs à vapeur de xénon.

On a utilisés les éclairs de lumière et simples et multiples pour durcir une gamme de peintures de diverses formulations. L'influence, dosée par la spectrographie à l'infra rouge, qu'exerce la densité du flux photonique et la durée de l'exposition a été interprétée sous termes des processus simples de polymérisation par radicaux libres.

Die UV Härtung von Akrylatmaterial mittels Blitzlicht von hoher Intensität

Zusammenfassung

Der Einfluss von Photonzustrom und Flussdauer auf die photoinitiierte Polymerisation von UV-härtbaren Akrylaten wurde untersucht. Anfangsexperimente mit Quecksilberlampen mit mittlerem Druck zeigten, dass wenn durch Focussieren elliptischer Reflektoren die Photonflussdichte erhöht wurde, die Härtung verbessert wurde. Durch Einsatz von sehr hohen Photonflüssen aus Xenon Blitzlichtlampen wurde die Untersuchung ausgedehnt.

Einzelne und vielfache Lichtblitze wurden zur Härtung einer Anzahl von Rezepturen benutzt. Der Einfluss von Photonflussdichte und Pulsdauer auf Härtung, gemessen mittels Ultrarotspektroskopie, wird mittels der für die bei freier Radikalpolymerisation vorkommenden, elementaren Prozesse benutzten Ausdrücke interpretiert.

Introduction

Refs. 1-3

UV curing requires an efficient source of UV radiation. The great majority of installations use medium pressure mercury discharge lamps. These vary in efficiency, but a typical lamp will convert about 20 per cent of input energy into UV radiation below 400 nm.

Several authors[1,2,3] have recently suggested that xenon lamps, if suitably operated, can be attractive radiation sources for UV curing. In order to obtain the optimum UV output from xenon lamps it is necessary to use them in the flash or pulsed mode, with a high peak current density. Under these circumstances the output of the lamp is a continuum with superimposed lines, and the peak photon flux may be several orders of magnitude higher than that given by a mercury lamp.

This paper describes work which has been carried out to examine the free radical polymerisation of acrylate materials initiated by intense flashes of light from xenon tubes. The

---

*Paper presented at the Newcastle Section Symposium on "Ultraviolet curing" held at the University of Durham on 14-15 September 1977.

results obtained have been helpful in obtaining a greater understanding of the processes involved in UV curing.

The kinetics of acrylate polymerisation

Refs. 4, 5

The acrylate monomers and oligomers used in UV curing formulations cure by a free radical addition polymerisation process. There are many reviews of free radical vinyl polymerisation in the literature. Particularly useful in this context is that of Oster and Yang[4].

There is a frequently quoted expression for the rate of free radical addition polymerisation:

$$\frac{-\mathrm{d}[M]}{\mathrm{dt}} = k_p\left(\frac{k_d f[S]}{k_t}\right)^{\frac{1}{2}}[M]$$

where $[M]$ is the monomer concentration, $[S]$ is the initiator concentration, k_p is the rate constant for propagation, k_d is the rate constant for the decomposition of initiator molecules into radicals, f is the fraction of radicals escaping the 'cage', and k_t is the rate constant for termination.

This expression has been modified for photoinitiated polymerisation to give[4,5]:

$$\frac{-\mathrm{d}[M]}{\mathrm{dt}} = k_p\left(\frac{I_o\varphi l\varepsilon[S]}{k_t}\right)^{\frac{1}{2}}[M]$$

where I_o is the incident light intensity, φ is the quantum yield (i.e. the yield of radicals from one excited photoinitiator molecule), l is the path length and ε is the extinction coefficient of the photoinitiator.

This expression indicates that the rate of polymerisation is proportional to the square root of the incident intensity, and implies that the degree of polymerisation obtained by a high intensity for a short time will be less than that obtained by a low intensity for a longer time.

The derivation of the above expression depends on the assumption of a kinetic steady state i.e. the rate of production of primary radicals is exactly balanced throughout by the rate of loss of radicals by termination processes. This may be valid when the intensity is low and polymerisation takes place in minutes or hours, but may not be valid when the total period of irradiation is only a matter of microseconds and the photon flux is varying enormously during that time.

The situation is further complicated by the fact that the reaction is exothermic and, as the conditions may be essentially adiabatic, the temperature may rise very considerably. Parts of the spectrum will be absorbed by the components of the formulation which will further enhance the temperature rise.

During cure there is a very rapid transformation from a liquid to a solid, and this increase in viscosity will have a profound influence on the rates of some of the processes taking place.

It is very difficult to predict the overall outcome of increasing the photon flux density. It is, however, instructive to estimate the effect on the rates of the elementary processes:

Production of primary radicals

$$e.g.\ \text{Initiator} \xrightarrow{h\nu} R^{\cdot}{}_1 + R^{\cdot}{}_2$$

The rate of production of primary radicals from the photoinitiator, for thin films, is given by:

$$V_R = I_o\varphi\ l\varepsilon[S]$$

With homolytic splitting processes, one quantum absorbed should result in two primary radicals, and therefore φ should be 2. However, in a polymerising system, the initiator will be surrounded by a 'cage', and if radicals cannot escape from the cage recombination may occur. This competing process will reduce the effective quantum yield. The 'cage' effect will depend on the cross-link density and the size of the primary radicals. The separation of the primary radicals is a diffusion controlled process and will be temperature dependent insomuch as a rise in temperature will produce a more mobile system. Because, with flash, I_o is very high, a very high value of V_R would be expected. However, although the rate will be very high it will also be very short lived.

Addition of primary radicals to monomer (initiation)

$$R^{\cdot} + M \xrightarrow{k_i} RM^{\cdot}$$

$$v_i = k_i[R^{\cdot}]\,[M]$$

Propagation

$$M + RM^{\cdot}{}_n \xrightarrow[k_p]{} RM^{\cdot}{}_{n+1}$$

$$v_p = k_p[P^{\cdot}]\,[M]$$

$[P^{\cdot}]$ is the sum of the concentrations of growing chain radicals. The peak rates of the last two processes will be high. The reactions are exothermic and, therefore, under adiabatic conditions there should be a considerable rise in temperature. Propagation is claimed not to be diffusion controlled, but to have an activation energy. However, in a system with polyfunctional monomers which has reached a high degree of polymerisation, steric effects will become important.

Termination processes

These are various radical – radical interactions which can reduce the concentrations of primary radicals and radicals in growing chains. They also limit the extent of polymerisation. Four termination processes are worth considering:

(a) *Recombination of primary radicals outside the cage.* This is quite a common reaction in UV curing and can account for some of the coloured byproducts *e.g.* two benzoyl radicals can combine to give benzil, which is yellow. The rate of the reaction is proportional to the *square* of the primary radical concentration. Radical combination reactions have no activation energy requirements and are diffusion controlled and, therefore, most prevalent in low viscosity systems. This means that if a low viscosity system is irradiated with a short intense pulse there will be a high rate of radical recombination.

(b) *Reaction of primary radicals with growing chain radicals.* The rate will be proportional to the product of the concentrations of the two radical types, and again will be diffusion controlled, and particularly by the diffusion of the primary radicals.

(c) *Reaction of growing chain radicals with one another*, is again diffusion controlled, but limited by mobility of polymer chains.

Termination processes *a*, *b* and *c* will be important in flash curing because, in simple terms, if radical concentrations are increased by a factor n (and n can be quite large in going from a mercury lamp to a xenon flash lamp) the rate of termination reactions is increased by a factor n^2. Because all these processes are diffusion controlled, they will be most rapid in low viscosity systems.

(d) *Reaction of atmospheric oxygen with primary radicals or with growing chain radicals*. The rate of termination by atmospheric oxygen will be affected by the rate at which oxygen can diffuse to the surface and into the film. Oxygen is a ground state free radical. Another example of termination by a ground state free radical is that due to the presence of carbon black pigments.

Curing of acrylate materials with mercury lamps

Ref. 6

One aspect of the curing of acrylate materials with mercury lamps, which is of importance in the design of UV dryers, is the choice of intensity of irradiation (i.e. watts cm^{-2}). The alternatives are either a high photon flux density for a short time, or a lower photon flux density for a longer time, by a suitable choice of reflector. A high photon flux density has, generally, been favoured and most UV dryers for sheet fed applications use focusing semi-elliptical reflectors.

The optimisation of photon flux density can be studied by moving an elliptical reflector/lamp combination relative to the print and measuring the influence on cure. Figures 1 and 2 show the results of two such experiments conducted on a laboratory sheet fed UV conveyor dryer. A Primarc medium pressure mercury lamp was placed at one focus of an anodised aluminium reflector, and the combination was placed across the conveyor. Photosensitive paper (Green Detex, William Sessions Ltd, York) indicated that peak photon flux on the belt was obtained when the lamp-belt distance was 7.8 cm. By moving the lamp/reflector combination vertically relative to the conveyor belt, the photon flux density could be varied greatly without appreciably altering the total number of photons striking the belt.

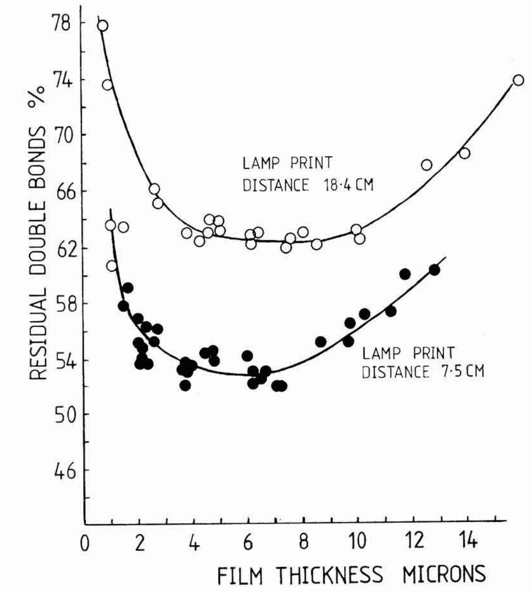

Fig. 2. Effect of intensity distribution and film thickness on cure of experimental red ink

In the first experiment a specially formulated Fishburn Ultraking red offset ink with only 9 per cent of the normal initiator concentration was printed at 3 μm film thickness on tinplate sheets. The number of passes through the UV dryer required to give surface cure, as measured by zero set-off on to white paper under hand roller pressure, was recorded. This was done for a variety of conveyor speeds and lamp-print distances. Figure 1 shows that as the lamp

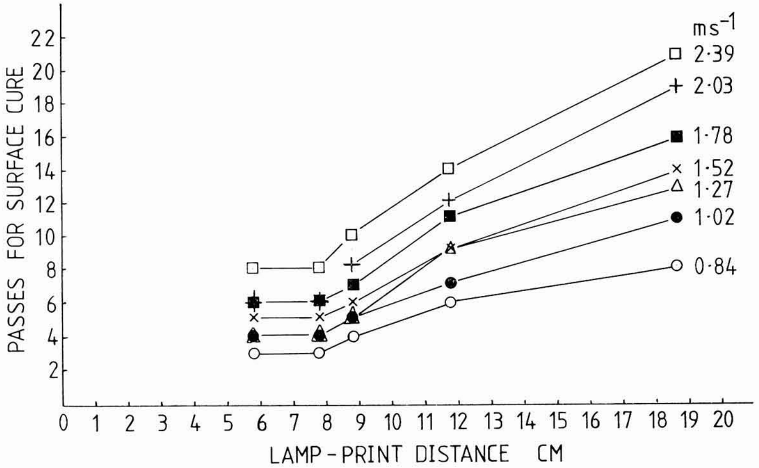

Fig. 1. Effect of intensity distribution on surface cure

module is raised from the optimum focus position cure deteriorates.

In the second experiment, an experimental red ink was formulated, whose components were:

Lankro	RCP 99 urethane acrylate resin	61.3 per cent
	trimethylol propane triacrylate	13.3 per cent
	pentaerythritol tetra-acrylate	13.3 per cent
	Irgalite NBS red pigment	10.0 per cent
	2,2-dimethoxy-2-phenylacetophenone	2.0 per cent

The monomers were supplied by Lankro Chemicals, the pigment by Ciba-Geigy and the photoinitiator by Ward Blenkinsop. The ink was printed at various film thicknesses on sodium chloride discs using a Duncan Lynch proof printer. The cure of the ink was measured from changes in its infrared spectrum. Infrared spectroscopy has been found to be a precise, rapid and objective method of examining the chemical changes which take place on cure, and in particular the depletion of double bonds. Sodium chloride was used as a substrate as it is transparent to infrared radiation and transmittance spectra can, therefore, be used.

The printed disc was placed in a holder and its infrared spectrum measured using a Perkin Elmer 157G spectrophotometer. The holder was then attached to a white coated sheet and passed through the UV dryer at 1.8 ms^{-1}. The spectrum was then measured again. The various absorptions associated with the acrylate double bond were found to have decreased. It was found convenient to calculate how many of the original double bonds had reacted from changes in the absorbance of the 810 cm^{-1} peak (acrylate CH_2 twisting mode[6]).

The 1525 cm^{-1} absorption (urethane) did not change on cure and was used to measure the thickness of the ink film. The experiment was done for a wide range of film thicknesses at two different lamp–print distances.

The results in Figure 2 show the average degree of cure in ink films of different thickness. It is possible to make a rough estimate from the curves of the distribution of cure within a thick film and this shows that, near the surface, cure is relatively poor. It is greatest at about 4-5 μm below the surface and then deteriorates at deeper levels. Whilst with high flux density curing, the surface cure is markedly better than with low flux density curing, cure at deeper levels—say 10 μm below the surface, is less affected by the photon flux density.

The probable explanation for this is that the poor surface cure is caused by oxygen inhibition, and a high photon flux density can help to overcome this. Cure in the deeper levels is less affected by photon flux density, but deteriorates because of poor radiation penetration.

An attempt was made to repeat the experiment in the absence of oxygen by sandwiching the ink film between two sodium chloride discs. It was found that even the low level of ambient light in the laboratory would cure the film before an infrared spectrum could be taken.

The conclusion from these experiments is that at the low film thicknesses provided by most printing processes, oxygen inhibition has an important influence on the cure obtained and a high photon flux is desirable to minimise this effect.

A logical extension of the use of focusing reflectors with mercury lamps is to examine lamps which can provide much higher photon fluxes.

The characteristics of xenon lamps

Refs. 7–11

Xenon lamps are manufactured in a wide range of shapes and sizes. The spectral output obtained depends on the dimensions of the lamp, the envelope material, its filling pressure and the driving circuit employed. Xenon is preferred to the other rare gases, argon and krypton, as it allows more efficient conversion of the electrical energy into light.

Xenon lamps may be operated in the continuous mode, with the discharge maintained by a low DC potential across the lamp. The current density is relatively low. In this mode a line spectrum is obtained for low pressure operation, changing to a continuum for high pressures. The efficiency of conversion of electricity into light is poor and little UV is produced.

In order to obtain higher conversion efficiencies and a greater proportion of UV it is necessary to increase the current density through the lamp. This is done by discharging a capacitor through the lamp to give a high current pulse of short duration. The arc formed acts as a black body radiator with a characteristic colour temperature which increases with the current density[7]. Although some lines may be observed, there is an intense continuum.

The purpose of this paper is not to discuss the viability of using xenon lamps for production line UV curing, but rather to use the high photon fluxes available to study the kinetics. The viability of xenon lamps for production purposes would depend on, among other factors, the conversion efficiency of electrical energy into UV radiation and the lifetime of lamps. The conversion efficiency varies very greatly. Xenon lamps are not generally as efficient as mercury lamps in producing UV radiation, and the high current density required for optimum efficiency tends to reduce lamp life. However, both efficiency and lamp life are dependent on the driving circuit and, for instance, by use of double pulse techniques[8, 9] very high current densities (25,000 amps/cm^2) can be passed through a lamp without damage. Useful reviews of xenon lamp operation are given by Barnes[10] and Edgerton[11].

Viscosity effects

Refs. 12-15

The first experiments were carried out with a small Mecablitz photographic flashgun with the diffusing screen removed. Prints were held close to the gun so that only about 5 cm^2 were irradiated. A large number of UV curing formulations were tried and it was found that whilst the higher viscosity materials, *e.g.* offset inks, overprint varnishes, cured well after a single flash, low viscosity formulations, *e.g.* coating varnishes, required several flashes for satisfactory cure and the first flash appeared to have little effect.

This finding, which agrees well with the predictions from kinetic considerations, was further examined by making a series of four unpigmented formulations to the general formula:

Dow XD 8008.01	epoxy acrylate resin	53.0 per cent
	Difunctional monomer	44.0 per cent
	Benzophenone	1.5 per cent
	Michler's Ketone	1.5 per cent

The difunctional monomers used, and the viscosities of the formulations obtained, were: butane diol diacrylate (3.2 poise), hexane diol diacrylate (4.4 poise), diethylene glycol diacrylate (4.8 poise) and neopentyl glycol diacrylate (7.6 poise). All monomers were supplied by Lankro Chemicals, and viscosities were measured at 25°C with an I.C.I. cone and plate viscometer. A similar, but higher viscosity formulation, was made with the trifunctional monomer trimethylol propane triacrylate.

The formulations were coated at about 5 μm film thickness on sodium chloride discs using glass rods and each sample was given several flashes of light. The cure after each flash was measured by infrared spectroscopy by changes in the ratio of the 810 cm^{-1} (acrylate) and 830 cm^{-1} (para substituted aromatic) absorptions.

The results are shown in Figure 3. The shapes of the curves and their dependence on the viscosity of the formulation illustrate the importance of the relative rates of propagation and termination reactions, and are an example of the "Tromsdorff effect"[12,13]. An auto-acceleration takes place when vinyl monomers are polymerised, either in the absence of solvent or in a solvent in which the polymer is not soluble, *i.e.* when there is a rapid increase in viscosity on polymerisation. The increase in viscosity causes an increase in $k_p/k_t^{\frac{1}{2}}$, and the result is an increase in the rate of polymerisation

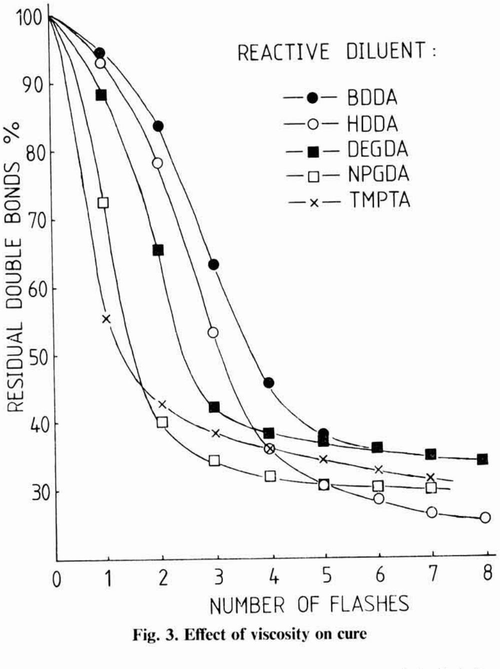

Fig. 3. Effect of viscosity on cure

Lee and Doorakian[14] have recently measured $k_p/k_t^{\frac{1}{2}}$ for the radiation curing of several pure monomers, including butane diol diacrylate, hexane diol diacrylate and diethylene glycol diacrylate, and have found that, relative to 2-ethyl hexyl acrylate, values of $k_p/k_t^{\frac{1}{2}}$ are 2.0, 4.0 and 6.1 respectively.

Collins, Costanza and Young[15] used infrared spectroscopy to measure the polymerisation of hexane diol diacrylate with 5 per cent benzoin isobutyl ether, curing with 1/60 sec. flashes from a mercury lamp obtained by using a shutter. They found that the first four flashes gave little cure, but subsequent flashes were much more effective. This effect was not found with the more viscous trimethylol propane triacrylate. Although the authors draw no conclusions from this aspect of their data, it does appear that the Tromsdorff effect can be observed with the photon fluxes obtained from mercury lamps if the viscosity is low enough.

Pulse duration and photon flux

The Mecablitz flashgun was used in ignorance of both its spectral output and the fraction of the total radiance of the lamp falling on the sample area. In order to examine carefully the effects of photon flux density on cure, a more versatile and characterised lamp was required. This was supplied by Applied Photophysics Ltd. It allows the capacitance, voltage and inductance of the driving circuit to be varied over quite a wide range.

There are two banks of capacitors, each consisting of five identical capacitors which can be connected together in parallel so that Bank *A*, where each individual capacitor is 250 μF, allows a capacitance of 250, 500, 750, 1000 or 1250 μF to be chosen. Similarly with Bank *B*, where each capacitor is 33 μF, capacitances of 33, 66, 99, 132 and 165 μF can be chosen. Each capacitor bank has its own high voltage supply unit. For Bank *A* this goes up to 900 V, whilst for Bank *B* the maximum is 2500 V. A separate inductance unit with a choice of inductances up to 215 μH is available for placing in series with the lamp, and this has the effect of lengthening pulse duration.

Linear quartz lamps of 150 mm arc length are used. A triggering electrode is wrapped around the lamp envelope. Discharge is unreliable when the capacitor voltage is less than 700 V, and spontaneous discharge may take place above 2400 V.

The input energy of the lamp is given by $\frac{1}{2}CV^2$, so that a wide range of pulse energies are possible. Pulse duration is a function of inductance and capacitance and is proportional to $(LC)^{\frac{1}{2}}$, so that the pulse width can be varied greatly.

A simple spectral radiometer was built to allow the measurement of the output of the Applied Photophysics flash equipment in the region between 250 and 400 nm.

A Barr and Stroud UMS type strip spectrum filter is housed between two 800 μm slits to provide a simple monochromator. By adjusting the position of the filter relative to the slits a narrow section of the spectrum between 250 and 400 nm can be chosen. Light passing through the slits and filter strikes an International Light PT 100 vacuum photodiode detector (S5 response) and the current produced is passed through a 48 kΩ resistor. The voltage drop across the resistor is recorded as a function of time on the screen of a Tektronix 564B storage oscilloscope. The trace gives the half peak width of the pulse, and the area under the curve is a measure of the number of photons striking the detector.

The flashtube was housed in a black box 75 cm from the detector with a shield arranged so that only light from the central 100 mm of the lamp could strike the detector. Similar measurements were made with various medium pressure mercury lamps in a module with a blackened reflector. A direct comparison was possible because the mercury lamps were placed in exactly the same position as the xenon lamp and all but the central 100 mm of the lamps was shielded from the detector. Although the output from an AC operated mercury lamp is not continuous but fluctuates at twice mains frequency, a microammeter could be used to measure the RMS current from the detector.

Figure 4 shows the spectral output of a 8 mm bore Spectrosil envelope 150 mm arc Wingent lamp at various combinations of capacitance, voltage and inductance. Also shown is the spectral output of a Primarc 80 watt/cm medium pressure mercury lamp.

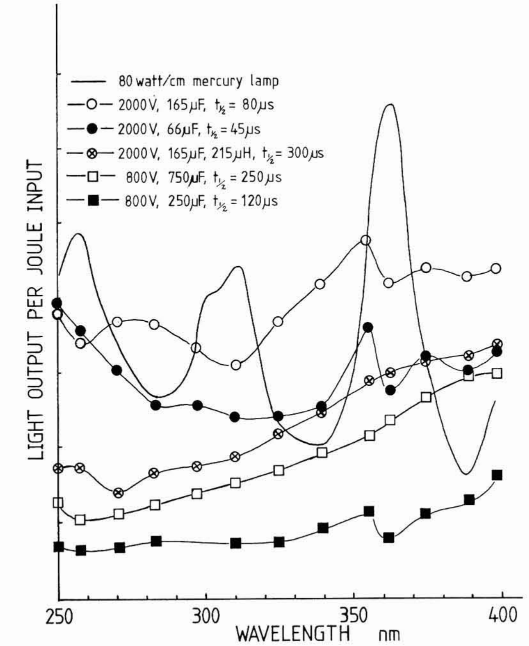

Fig. 4. Spectral outputs of xenon and mercury lamps

The xenon lamp varies a great deal in its efficiency and, in very general terms, high energy short duration pulses give the highest efficiency. At its most efficient the xenon lamp approaches the efficiency of the mercury lamp.

The lamp was used without a reflector. Samples to be cured were placed near the centre of the lamp, so that the lamp-sample distance was small compared with the length of the arc. The assumption was made that areas of uniform photon flux density form concentric cylinders around the length of the lamp. As the areas of these cylinders are proportional to their radii, the implication is that the photon flux density striking a sample is inversely proportional to the lamp—sample distance. Varying the lamp—sample distance allowed the photon flux density to be altered independently of pulse duration or spectral distribution.

A variety of experimental and commercial unpigmented formulations were cured with single flashes of radiation. Variables examined were capacitance, voltage, inductance, lamp-sample distance, film thickness and ambient temperature. Cure was measured by infrared spectroscopy. Certain phenomena were noted:

(i) Over the range of film thicknesses studied—up to about 15 μm, the cure measured usually improved as the film thickness increased, although this depended on the photoinitiator.

(ii) Increase of ambient temperature improved the cure obtained. The range examined was 9–47°C.

(iii) With capacitor bank *B* (low capacitance, high voltage, short duration, high photon flux density), as the lamp-sample distance was decreased, cure improved to an optimum value and then decreased. Even with quite viscous materials (e.g. 200 poise) a short duration (less than 100 μs half peak width) high flux flash can produce remarkably little cure, and the cure deteriorates as the flux density is increased above a certain level.

(iv) With capacitor bank *A* (high capacitance, low voltage, long duration ($t_{1/2}$ = 120 – 900 μs), low photon flux density), as the lamp-sample distance was decreased, cure improved to the lowest lamp-sample distance used (0.5 cm), although a stage could be reached where further increases in photon flux density gave little further improvement in cure.

Measurements with the Applied Photophysics flash unit were supplemented by measurements with a small U shaped flash tube housed in a semi-cylindrical aluminium reflector fed from a large (3000 μF) capacitor bank charged to 450 V. This flash tube gives high flux density long duration flashes. The input energy per unit area of sample irradiated is high.

A very large number of measurements were made, but because it was not possible to examine every possible combination of variables the data are inevitably fragmentary. However, a pattern emerges which appears to unify the results. This pattern is shown in Figure 5, which shows how the cure of a typical material at constant film weight and ambient temperature is dependent on the photon flux density and duration of the radiation pulse.

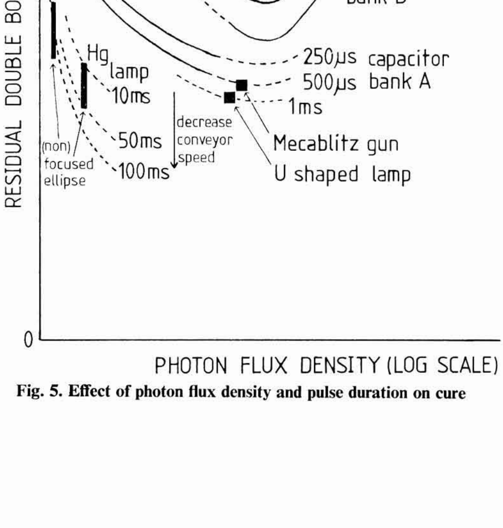

Fig. 5. Effect of photon flux density and pulse duration on cure

Both photon flux density and pulse duration are difficult to quantify satisfactorily because photon flux varies rapidly during the pulse, and a suitable average value is needed, and the half peak measurement of pulse duration may not be entirely satisfactory when there are large variations in pulse shape.

Another problem with photon flux is knowing which photons to count and which to neglect. Should all photons be counted within a certain band of wavelengths, or would it be better to give each wavelength a weighting according to the absorption spectrum of the photoinitiator?

Results obtained with mercury lamps at various conveyor speeds and with focusing and non-focusing elliptical reflectors are fitted into the pattern.

The pattern can be interpreted in terms of the kinetic scheme described above. Whatever the overall rate of polymerisation, it is to be expected that increasing the duration of radiation will increase the cure obtained. The effect of changing photon flux density is more complex, and it can be seen that for short duration pulses, increasing the photon flux density beyond a certain level actually decreases the cure obtained. There are two possible explanations for this striking phenomenon. The first, and less likely, is that absorption of radiation by the sample causes a rapid exceeding of the ceiling temperature of the polymer and, therefore, cure is prevented by thermodynamic factors. The second is that the extra radicals produced are more likely to enter into recombination and chain termination reactions than into chain initiation.

The ceiling temperature explanation is unlikely because the effect is so viscosity dependent. As will be seen in the next section, high flux short duration pulses are very effective for increasing cure in a partly polymerised system. However, temperature rise effects can be very striking in pigmented systems because of absorption by the pigments of the visible part of the flash. High flux density curing of ink films on sodium chloride discs may cause the discs to crack.

There may be vaporisation of part of the formulation, and if the ink film is on a plastic substrate the latter may degrade.

Curing with multiple flashes

The results shown in Figure 3 indicate how advantageous it can be to irradiate a sample to be cured with several flashes, provided that the interval between flashes is sufficient to allow partial polymerisation to take place so that the effects of termination processes are reduced.

Several experiments were performed in order to assess the effectiveness of combinations of pulses for curing acrylate materials:

(i) It can be seen from Figure 5 that a single flash from the xenon lamp fed from capacitor bank *B* often gives poor cure. It was decided to try using different numbers of flashes whilst keeping the total irradiation constant. The formulation used was:

Lankro RCP99 resin	66.0 per cent
trimethylol propane triacrylate	14.5 per cent
pentaerythritol tetra-acrylate	14.5 per cent
2,2-dimethoxy-2-phenylacetophenone	5.0 per cent

A capacitance of 99 μF was used, and a voltage of 2000 V, with no added inductance. The lamp-sample distance was chosen to give the sample 1.25 joules (into the lamp) per sq cm of sample. A single flash left 63 per cent of the double bonds unreacted. The lamp-sample distance was then doubled to halve the photon flux density, and two flashes then left only 39 per cent of the double bonds unreacted. Reducing the photon flux density to only a fifth of the original value, and giving the sample five flashes, improved the cure still further, leaving only 33 per cent of the double bonds unreacted. In this experiment the interval between flashes was about 2 sec. The experiment was repeated with capacitor bank *A* (1250 μF, 800 V, no added inductance) and it was found that a single 1.25 joules/cm^2 flash left 36 per cent of the double bonds unreacted. Two 0.625 joules/cm^2 flashes gave the same result.

(ii) In order to examine the importance of the interval between flashes, a second flash unit was built similar to the first, but lacking capacitor bank *A*. This second unit fed a 150 mm arc 8mm bore lamp which was mounted parallel to and 2.5 cm from the first. A photodiode picked up the flash from the first lamp and the signal was fed into a delay unit which then triggered the second lamp after any preset delay time of 1 ms or longer. The sample was placed equidistant from the lamps. A first experiment was performed using the formulation described in (i). The first lamp was fed from capacitor bank *A* with 750 μF and 800 V, whilst the second lamp was fed from capacitor bank *B* with 165 μF and 1800 V. The sample disc was positioned so that for each lamp the input energy was 0.31 joules/cm^2. The cure obtained as a function of delay time is shown in the following table:

Table 1

Delay time	% Residual double bonds
1 ms	43
10	40
100	38
1,000	37

These results were compared with cure obtained with the first flash alone (48 per cent), two 'first' flashes with 3 minutes delay (44 per cent), and a single first flash with the lamp-sample distance halved (42 per cent).

A second experiment was performed with the RCP99 resin based ink. The same combinations of capacitance and voltage were used as before, but the lamp-sample distance was halved so that for each lamp the input energy was 0.62 joules/cm^2. The ink was printed on sodium chloride discs at 4 μm film thickness. The results are given in the following table:

Table 2

Delay time	% Residual double bonds
1 ms	63
10	56
100	49
1,000	47
10,000	49

These results were compared with cure given by the first flash alone (65 per cent), a single 'second' flash (76 per cent), two 'first' flashes with 10 sec delay (55 per cent), and two 'second' flashes with 10 sec delay (53 per cent).

These experiments to examine the influence of delay time show that, in order to optimise cure, the delay between flashes should be at least 100 ms, in these particular situations. The optimum delay time will depend on both the formulation and the curing conditions. When the conditions used with the red ink were used to cure an unpigmented formulation with only 1 per cent initiator, no improvement was found when the delay time was increased beyond 10 ms.

Conclusions

The purpose of the work described in this paper has been firstly, to demonstrate the complexities of the kinetics of acrylate curing, and in particular the adverse effects of termination reactions, and secondly, to look for the most effective way of irradiating a sample to be cured with a fixed number of photons. It has been demonstrated that the photon flux density/time relationship must be chosen correctly for satisfactory cure to be obtained.

The variable delay technique used with double flash curing is a potentially useful analytical tool somewhat analogous to conventional flash photolysis and could provide useful information about the rates of these very rapid polymerisation reactions. The results given in this paper suggest that typical reaction rates may be such that, when curing with mercury lamps, most of the polymerisation takes place during the period of irradiation.

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Parameters in UV curable materials which influence cure speed*

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Summary

A study is presented on the influence of some parameters of polyesteracrylates, such as molecular weight and functionality, on the cure speed. The effects that these parameters have on curing characteristics of mixtures with polyethyleneglycoldiacrylate are also studied. The experiments were performed in presence and absence of air oxygen.

A method is described for the measurement the amount of double bonds remaining after UV curing.

Several parameters are found to be of importance in determining cure speed in the presence of air. In the absence of air oxygen, high cure speeds are achieved and relatively low degrees of cure are obtained.

Keywords

Raw materials for coatings
  binders (resins, etc)

acrylate resin
polyester acrylate

Raw materials used in manufacture or synthesis of ingredients for coatings

monomer
oligomer

Processes and methods primarily associated with manufacturing or synthesis

oxygen inhibition

drying or curing of coatings

ultraviolet curing
radiation curing

service or utility

photopolymerisation

Properties, characteristics and conditions primarily associated with coatings during application

curing rate

Les paramètres qui exercent une influence sur la vitesse de durcissement de revêtements durcissables par les rayons ultra violets

Résumé

On présente une étude sur l'influence qu'exercent certains parametres de polyesteracrylates, tels que leur poids moléculaire et leur fonctionalité, sur la vitesse de durcissement. On a étudié également les influences qu'exercent ces paramètres sur les caractéristiques de durcissement des mélanges avec le diacetate de polyéthylène glycol. On a effectué les expériences avec et en l'absence d'oxygène atmosphérique.

On décrit une méthode pour déterminer le taux de double-liaisons qui reste encore en vigueur après le durcissement par les rayons ultra violets.

On trouve que plusieurs paramètres exercent une influence importante sur la vitesse de durcissement en présence de l'air. En l'absence de l'oxygène atmosphérique, des vitesses de durcissement élevées sont réalisées et en même temps des degrés de durcissement assez faibles sont obtenus.

Die Härtungsgeschwindigkeit beeinflussende Parameter in UV-härtbaren Materialien

Zusammenfassung

Es handelt sich um eine Untersuchung des Einflusses einiger Parameter, wie Molekulargewicht und Funktionalität, bei Polyesterakrylaten auf die Härtungsgeschwindigkeit. Ebenfalls untersucht wurden die Auswirkungen dieser Parameter auf die Härtungscharakteristika von Mischungen mit Polyäthylenglykoldiakrylat. Die Versuche wurden in Gegenwart, sowie Abwesenheit von Sauerstoff der Luft durchgeführt.

Eine Methode zur Messung der Anzahl von Doppelbindungen, welche nach UV Härtung verbleiben, wird beschrieben.

Es wird gefunden, dass verschiedene Parameter für die Bestimmung der Härtungszeit bei Gegenwart von Luft wichtig sind. Bei der Abwesenheit von Sauerstoff der Luft werden hohe Härtungsgeschwindigkeiten, aber verhältnismässig niedrige Durchhärtung erhalten.

Introduction

Much has already been said and written about many aspects of UV–curing. The considerable economic advantages of this technology: saving of energy, as well as of raw materials and contribution towards a cleaner environment are very well known.

Less knowledge is available about what can be done on a molecular basis to control the curing characteristics of the monomer/oligomer blend.

The systems that are marketed or described in literature recently can be seen to be formed mainly from an oligomer, a monomer and a photoinitiator.

Of course, all the components in this type of formulation are of great importance in determining the cure speed. This paper describes a research programme carried out by the product Development Department, the Technical Marketing Department, and the Analytical Laboratory of Synthese.

The influence of some parameters of the oligomers, such as

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molecular weight and functionality on the cure speed have been investigated.

Cure speed was measured by judging the film after it had been rubbed by the finger tip. The degree of cure was also measured by means of infrared spectroscopy. In the first part of the investigation experiments were performed in air. In the second part, the experiments were repeated with the exclusion of air. The results of this latter part were rather surprising.

Monomers

Monomers are generally low molecular weight acrylate esters. They have to be added to a system so as to act as crosslinking agents and to give the systems a workable viscosity. To obtain low viscosity, much monomer has to be added. However, to retain good reactivity in the mixture, earlier literature indicates that as little monomer as possible should be used. Therefore, low viscosity, highly reactive liquids with good thinning properties are required.

As little monomer as possible should be added to a system for toxicologic reasons, such as skin irritation, which is largely determined by the amount of monomer present.

Some properties of monomers used in UV curing are shown in Table 1.

Furthermore, monomers, such as for example low molecular polyesteracrylates such as UVE 70, can be used. This is included in Table 1.

An important advantage of this kind of monomer is that a wide range of similar materials can be prepared, so that properties such as compatibility with oligomers, molecular structure, flexibility, hardness, etc can be adjusted.

For these experiments one "reactive diluent crosslinker", polyethyleneglycol diacrylate has been chosen. This monomer has good overall properties.

Oligomers

Oligomers can be chosen from a wide range of chemical structures. In the author's opinion, oligomers determine the properties of the final coating film. Properties such as hardness, flexibility, durability and adhesion can to a large extent be controlled by varying the chemical composition of the oligomers.

Figure 1 gives an idea of the range of chemical possibilities.

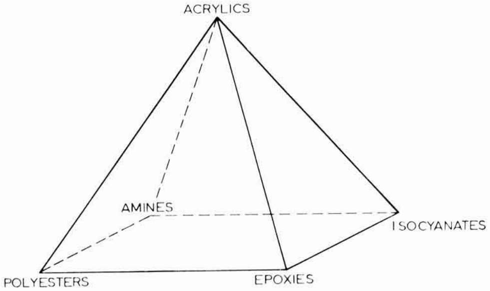

Fig. 1. Building blocks for oligomer synthesis

At the angles of this pyramid are the building blocks that can be chosen to form an oligomer. As oligomers for these experiments, a series of polyesteracrylates has been chosen. Polyesteracrylates have the advantage that characteristics of the final product can be carefully selected. By means of a

Table 1

Some properties of monomers used in UV-curing

	Average mol. wt. $M\bar{n}$	Viscosity (cP)	Acrylic functionality F_A	Acid value	B.P. (°C, at 1mm Hg)	Primary irritation index (PII)
Neopentylglycol-diacrylate	212	8	2	<1	96	6.0
Hexanediol 1,6 diacrylate	226	8	2	<1	125	6.0
Polyethyleneglycol-diacrylate	310	30	2	<2	>120	4.9
Trimethylolpropane triacrylate	296	250	3	<2	>200	5.1
Pentaerithritol-triacrylate	300	2500	3	<2	>200	5.2
Dianol 2,2–diacrylate	400	8000	2	<5	>200	0.4
UVE 70 polyesteracrylate	450	300	2	<15	>200	4.4

Table 2

Properties of some polyesteracrylates

Polyester-acrylate	Number Average mol. wt. $M\bar{n}$	Acid value	Hydroxyl value	Acrylic functionality F_A	Maleic functionality F_M
02	918	13.4	33	1.71	1.30
03	934	12.4	33	1.74	0.74
06	839	11.9	33	1.78	1.11
18	530	12.6	34	1.85	0.97
30	805	8.2	30	2.11	0.87

computer programme, the end – properties, such as number average molecular weight, hydroxyl value, acid value, molecular functionality, and compatibility can be calculated from the properties of the raw materials to be used.

The properties of a number of polyesteracrylates are outlined in Table 2.

Photoinitiators

Photoinitiators are the driving force of the total system. If they are not used, the system will not function properly.

The class of substance which can be advantageously used are aromatic ketonic compounds, which generate reactive radicals upon exposure to UV radiation. These radicals subsequently initiate the polymerisation reaction of the double bonds in the binder.

For these experiments the photoinitiator chosen was the dimethylketal of benzil.

Experimental

Application and curing conditions

All the formulations were applied as a layer of 30 microns thick on tin plate using a wire-wound rod. The plates were placed on a conveyer belt whose speed could be varied from 2–12 metres/minute. The UV radiation was produced by a Philips HOK 5 lamp of 80 watts/cm. The lamp was placed at a distance of 12 cm from the substrate giving a band width of 15.5 cm at substrate level.

This band width could be varied by means of a slit, thus the effective belt speed could be accelerated artificially by narrowing the slit width.

In order to obtain comparable cure speed values, the exposure time (ET) was used as a measure of UV energy input, and an estimation was made of the belt speed for the fully opened slit.

Table 3 gives an idea of these speeds.

Cure speeds were defined as the fastest belt speeds at which the formulations could be cured under UV. A film was judged to be cured when rubbing with the fingertip did not cause any damage to the film.

Table 3

Comparison of exposure time with the belt speed at fully opened slit

Exposure time (sec)	Belt speed (m/min)
4.65	2
2.33	4
1.33	7
1.05	9
0.80	12
0.60	15
0.40	23
0.20	46
0.10	93

The estimated belt speeds were then converted from the setting of the slit width into exposure times by means of the formula:

$$\mathrm{ET} = \frac{\text{slit width (m)}}{\text{belt speed (m/sec.)}}$$

Formulations

The polyesteracrylates were tested in the following formulations:

Pure oligomers

Polyesteracrylate	100 parts
Dimethylketal of benzil	4 parts

Set percentage of oligomers

Polyesteracrylate	50 parts
Polyethyleneglycol diacrylate	50 parts
Dimethylketal of benzil	4 parts

Set viscosity

Polyesteracrylate	X parts
Polyethyleneglycol diacrylate	100–X parts
Dimethylketal of benzil	4 parts

X was chosen so as to achieve a viscosity of 10 poise for the blend.

The cure speed of pure polyethyleneglycol diacrylate (PEGDA) was also measured.

Some other blends of two polyesteracrylates were additionally made to get an idea of the influence of the percentage of polyesteracrylate in the blend on cure speed.

Table 4

Experiments with pure oligomers

Polyester-acrylate	Number Average mol. wt. $M\bar{n}$	*viscosity dP	Acrylic functionality F_A	# 1000 A	F_A x $M\bar{n}$	ET (sec.)
18	530	71	1.85	3.49	981	2.33
09	639	85	1.84	2.88	1176	1.55
13	780	235	1.93	2.47	1505	1.33
06	839	1480	1.78	2.12	1493	1.17
27	898	340	2.02	2.25	1814	0.80
02	918	339	1.71	1.86	1570	1.17
48	925	658	2.08	2.25	1924	0.93
31	979	931	2.07	2.11	2027	0.80
33	1070	919	2.57	2.40	2750	0.60

*Viscosity is given in decipoise measured at 20°C

Results

Pure oligomer

In order to find the parameters of the oligomers which influence cure speed, it is obvious that the clearest relationship will be found between these parameters and cure speed when testing the oligomers in the pure state.

The following formulation was therefore tested:

Polyesteracrylate	100 parts
Dimethylketal of benzil	4 parts

Experiments were conducted with nine pure polyesteracrylates whose properties are given in table 4.

Figure 2 gives the relationship between ET and *Mn̄*. ET decreases with higher *Mn̄*. This relationship is to be expected, because when relatively larger molecules are polymerised, bigger polymers are formed more rapidly.

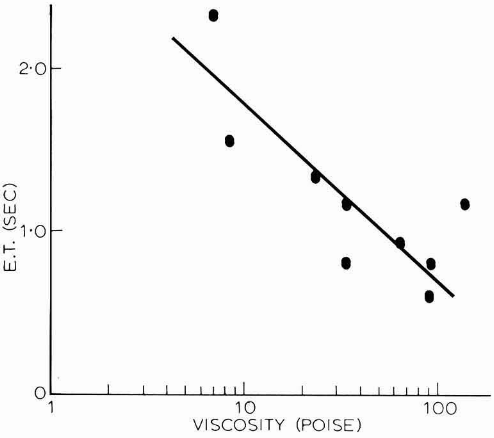

Fig. 4. Exposure time as a function of viscosity of pure polyesteracrylates

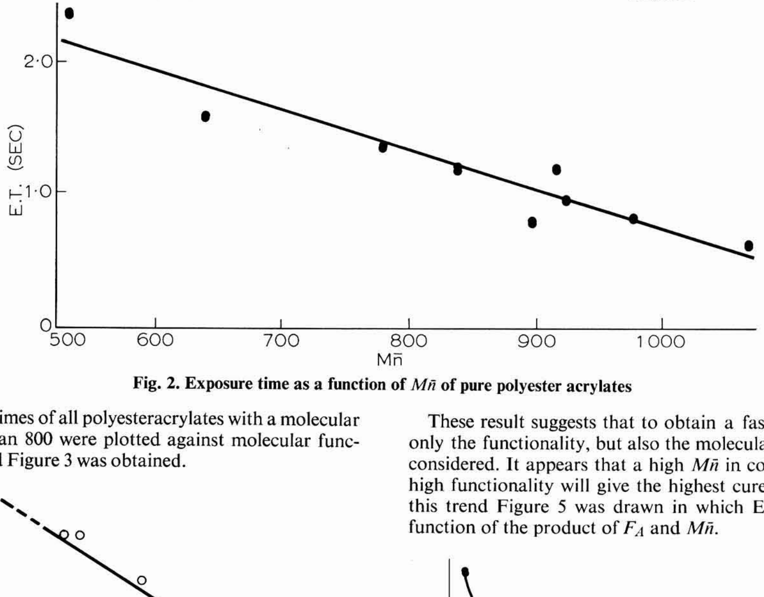

Fig. 2. Exposure time as a function of *Mn̄* of pure polyester acrylates

The exposure times of all polyesteracrylates with a molecular weight higher than 800 were plotted against molecular functionality (F_A) and Figure 3 was obtained.

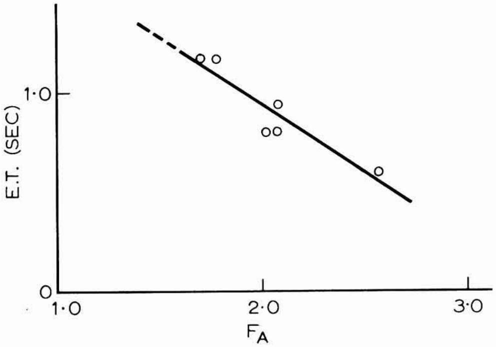

Fig. 3. Exposure time as function of functionality of pure polyesteracrylates

A clear relationship is shown: the higher functionalities giving shorter exposure times.

In Figure 4 the relationship between ET and viscosity is shown. A very clear relation is observed here: the higher viscosities give shorter exposure times and higher cure speeds.

These result suggests that to obtain a fast cure speed, not only the functionality, but also the molecular weight must be considered. It appears that a high *Mn̄* in combination with a high functionality will give the highest cure speed. To check this trend Figure 5 was drawn in which ET is plotted as a function of the product of F_A and *Mn̄*.

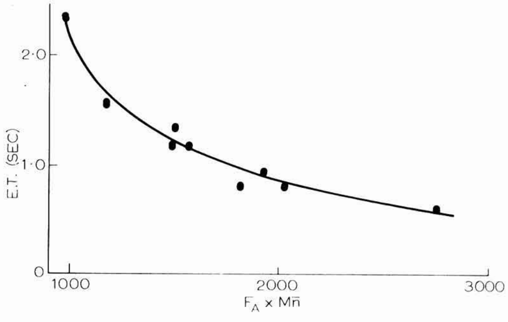

Fig. 5. Exposure time as a function of F_A x *Mn̄* of pure polyesteracrylates

This graph shows that there is a correlation between exposure time and the parameter F_A x *Mn̄* which all nine polyesteracrylates demonstrate.

Mixtures

In order to get an impression of the behaviour of oligomers and their parameters in admixtures with reactive diluents, polyethyleneglycol diacrylate was used as reactive diluent and several mixture properties were studied in different formulations.

As mixture parameters the properties chosen were:

X = percentage of polyesteracrylate in the mixture
$M\bar{o}$ = number average mol. wt. of mixture
\# 1000A = amount of acrylic double bonds per kg.

Influence of the percentage of polyesteracrylate

To find the influence of the parameter X two polyesteracrylates were tested in different mixing ratios with polyethyleneglycol (200) diacrylate (PEGDA). Results of these experiments are outlined in Table 5.

Table 5
Influence of percentage oligomer on mixture properties

	Polyesteracrylate 03			Polyesteracrylate 31		
X	\# 1000 A	$M\bar{o}$	ET	\# 1000 A	$M\bar{o}$	ET
0	6.45	310	3.49	6.45	310	3.49
50	4.16	465	2.33	4.28	471	2.33
60	3.70	517	2.33	3.85	525	1.55
80	2.78	666	1.55	2.98	684	1.33
100	1.86	934	1.17	2.11	979	0.80

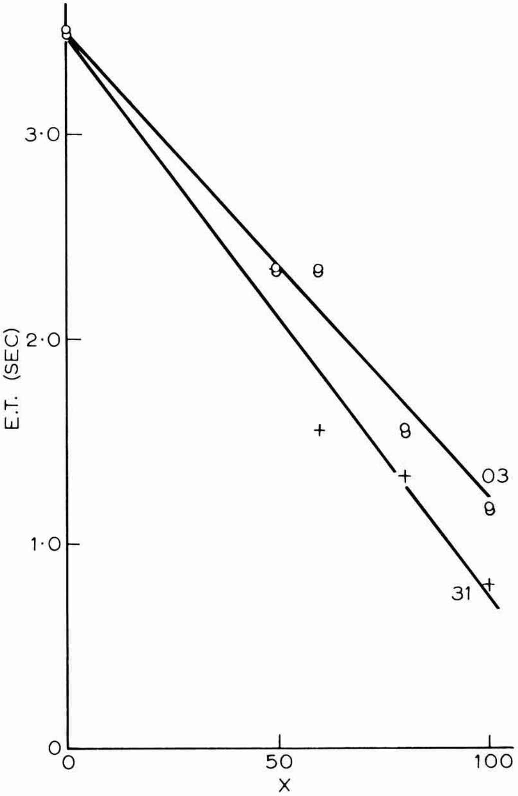

Fig. 6. Influence of percentage of polyesteracrylate on exposure time

Figure 6 shows that there is a straight line relationship between ET and the percentage of polyesteracrylate in the mixture; higher percentages giving shorter exposure times.

Influence of molecular weight

From the experiments with pure polyesteracrylates, it was concluded that the number average molecular weight was a rate determining parameter.

The number average molecular weight $M\bar{o}$ of a mixture can be derived from the following equation:

$$M\bar{o} = \frac{N_1 M\bar{n} + N_2\, M\bar{n}_{PEGDA}}{N_1 + N_2}$$

where:

N_1 = number of molecules polyesteracrylate
N_2 = number of molecules PEGDA
$M\bar{n}$ = number average molecular wt of polyesteracrylate
$M\bar{n}_{PEGDA}$ = number average molecular wt of PEGDA.

Substituting into this equation:

$$N_1 = \frac{X}{M\bar{n}}$$

$$N_2 = \frac{100 - X}{M\bar{n}_{PEGDA}}$$

$$M\bar{n}_{PEGDA} = 310$$

gives

$$M\bar{o} = \frac{31000\, M\bar{n}}{310\, X + 100\, M\bar{n} - X\, M\bar{n}}$$

This formula shows that $M\bar{o}$ can be varied in three ways:

(1) by changing $M\bar{n}$ at constant X
(2) by changing X at constant $M\bar{n}$
(3) by changing both X and $M\bar{n}$

The first change was accomplished by mixing seven different polyesteracrylates in a 50/50 ratio with PEGDA (see Table 6).

Table 6
Experiments with constant percentage of oligomer ($X = 50$)

Polyester-acrylate	\# 1000 A	$M\bar{o}$	ET
02	4.16	463	1.05
18	4.97	391	1.86
09	4.67	417	1.86
13	4.46	444	1.55
06	4.29	453	1.33
27	4.35	461	0.93
33	4.43	481	1.05
PEGDA	6.45	310	3.49

The second change was accomplished by mixing one polyesteracrylate in different mixing ratios (see Table 5).

The third change was brought about by testing nine polyesteracrylates at a constant viscosity of 10 P (see Table 7) with different percentages of polyesteracrylate. These experiments with a constant viscosity were made because viscosity was found to be a parameter controlling the cure speed.

Table 7

Experiments with constant viscosity of 10 *poise* (20°*C*)

Polyester-acrylate	X	# 1000 A	$M\bar{o}$	ET
02	79	2.82	650	1.55
09	100	2.88	639	1.55
13	83	3.15	620	1.33
27	74	3.36	601	0.80
48	70	3.51	580	0.80
03	76	2.98	630	1.86
32	72	3.52	581	0.93
27B	65	3.72	546	0.80
30	80	3.39	610	1.17

Figure 7 shows three graphs of ET as function of $M\bar{o}$ as $M\bar{o}$ is changed by the three means described above.

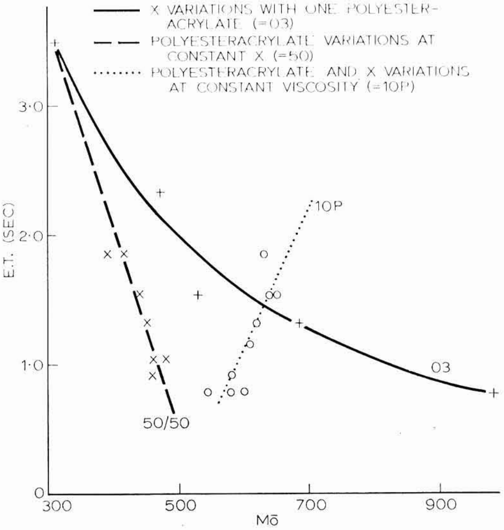

Fig. 7. Exposure time as a function of $M\bar{o}$

The graphs show that there is a correlation between ET and number average molecular weight of the mixtures. The graph in which $M\bar{o}$ is changed by changing $M\bar{n}$ at constant X and the graph in which $M\bar{o}$ is changed by changing X at constant $M\bar{n}$ show qualitatively the same relationship as was found in the experiments with pure polyesteracrylates (see Fig. 2): shorter exposure times are observed at higher molecular weights.

The third graph of Figure 7 shows an opposite effect: higher molecular weights giving longer exposure times. This can possibly be explained by the fact that mixtures having a high concentration of polyesteracrylate are involved.

For this reason the influence of the polyesteracrylates is increased and it is better to take here the number average molecular weight of the polyesteracrylate as a rate determining parameter.

This gives a correlation as shown in Figure 8 which is comparable to the other relations found: higher $M\bar{n}$ requiring shorter exposure times.

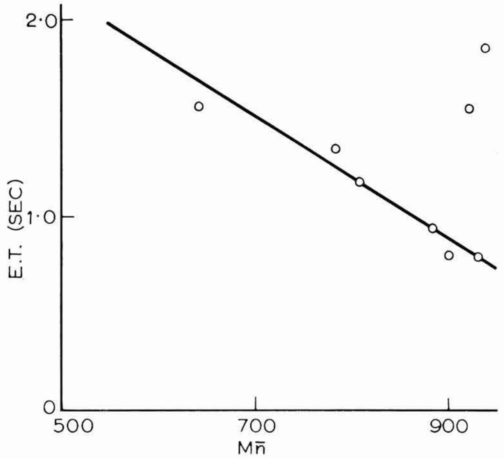

Fig. 8. Exposure time as function of $M\bar{n}$

Influence of the amount of acrylic double bonds per kg

The amount of acrylic double bonds per kilogram can be derived from the following equation and is thought to be a direct measure for the reactivity of the mixture.

$$\# \, 1000\mathrm{A} = \frac{10 \, . \, X \, . \, F_A}{M\bar{n}} + \frac{10 . \, (100 - X) \, . \, F_{PEGDA}}{M\bar{n}_{PEGDA}}$$

This parameter can again be varied in three ways:

(1) By changing the polyesteracrylate, so changing $M\bar{n}$ and F_A and keeping X constant.

(2) By changing X using one polyesteracrylate; so keeping $M\bar{n}$ and F_A constant.

(3) By changing X and the polyesteracrylates, keeping constant the viscosity.

Figure 9 shows three graphs of ET as a function of the amount of acrylic double bonds per kg.

The graph which gives the results of the experiments at a fixed viscosity of 10 p shows the relationship expected: more acrylic double bonds per kg giving shorter exposure times and so faster cure speeds.

The other two graphs demonstrate an anomalous behaviour: more acrylic double bonds giving longer exposure times, and so slower cure speeds. This effect does not seem logical and cannot be explained at the moment, but this observation will be discussed later.

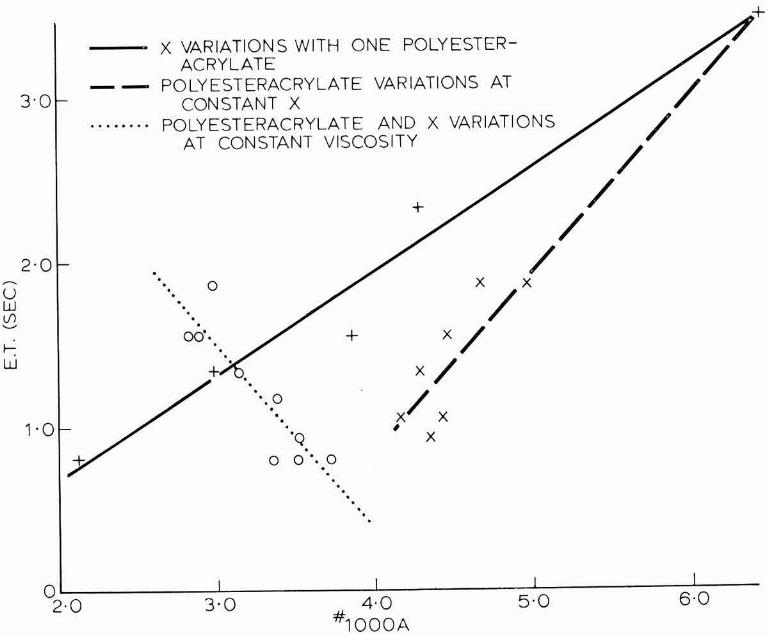

Fig. 9. Exposure time as a function of the amount of acrylic double bonds per kg

From these experiments it was concluded that cure speed is governed by several parameters, namely:

$M\bar{n}$	for pure polyesteracrylates
F_A	for pure polyesteracrylates
$M\bar{o}$	for mixtures
X	for mixtures
# 1000A	for mixtures
$M\bar{n}$	for mixtures with a high percentage of polyesteracrylates.

Infrared spectroscopy

So far, cure speed has been measured by judging the paint film visually after it had been rubbed by the fingertip. Because with this method it is always possible that personal judgment will influence the experimental results and some results were found which were not according to expectations, it was desireable to have a more objective method of measuring cure speed.

It was required to measure a chemical change occurring during the exposure to UV radiation.

Basically, in the resin the main change during UV curing is the disappearence of the double bonds. The change in amount of double bonds can be measured instrumentally, for instance by NMR or IR spectroscopy.

Extraction and titration could also be carried out on the paint film. To do this, however, the paint film has to undergo many operations which would be time consuming and errors would be more likely to occur.

Infrared spectroscopy was chosen because this method would give the least experimental difficulty.

The lacquer formulation used was:

Acrylic ester epoxy resin	28 parts
Polyethyleneglycoldiacrylate	72 parts
Dimethylketal of benzil	4 parts

Figure 10 is the infrared spectrum of the uncured product. In this spectrum two absorption peaks caused by ethylenically unsaturated double bonds are indicated by the arrows. For measurements, the absorption peak on the left hand side of the spectrum was chosen (near 1640 cm^{-1}).

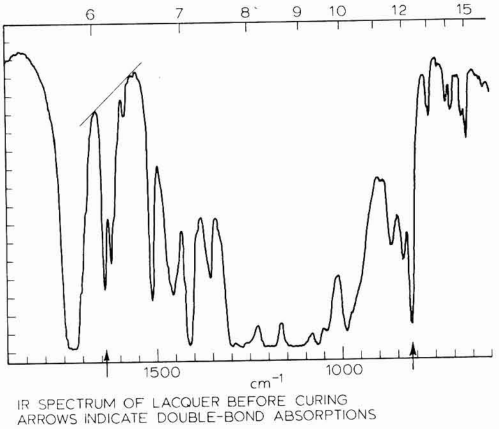

Fig. 10. Infrared spectrum of the uncured product

The lacquer was applied to potassium bromide discs by a wire wound rod in layers of about 30μm thick.

The wet films were exposed to UV radiation several times. Figure 11 gives the important range of the infrared spectra of the uncured film and the spectra of the films after being subjected to three different amounts of UV energy as indicated by the exposure times. As shown, the absorption peak due to the double bonds decreases after UV irradiation.

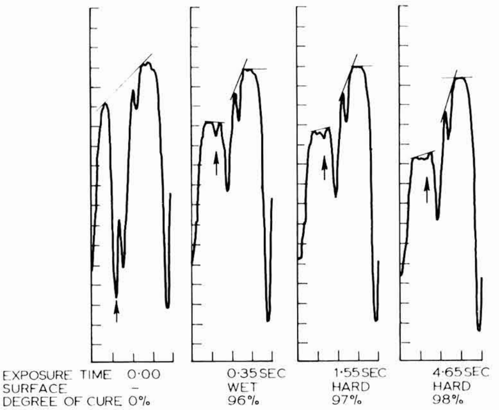

Fig. 11. Degree of cure

After curing the films were examined by rubbing the film with the fingertip. After an exposure time of 1.55 seconds the film could no longer be damaged by rubbing.

Table 8 shows the exposure times used, the results of the films surface judgments and the corresponding degrees of cure. The degree of cure is defined as the extinction of the double bond absorption after exposure, taken as percentage of the extinction of the double bond absorption of an uncured film.

Table 8
Degree of cure of UV systems

Exposure time	Degree of cure	Film surface
13.95 sec.	98%	Hard
4.65 sec.	98%	Hard
2.33 sec.	97%	Hard
1.55 sec	97%	Just hard enough
1.17 sec.	97%	Soft
0.93 sec.	97%	Very soft
0.35 sec.	96%	Wet
0.18 sec.	95%	Completely wet

Corrections for variations in film thickness were made from the change in an appropriate absorption band which does not change during the curing process.

It is remarkable that the curing degree is high under all the conditions used, even in those cases where the surface of the film is still wet.

It is difficult to understand how such a small difference in the degree of cure (97 per cent to 95 per cent corresponds to such a great difference in film appearance (hard and dry film *vs.* wet film).

The insufficiently cured films were examined more closely and the surface washed off. It was found that a film of acceptable hardness lay underneath. Evidently the cure speed at the surface is much slower than deeper in the film and this possibly may be due to the well-known phenomenon of air inhibition. To check this hypothesis an experiment was done with the exclusion of air.

A foil was placed over the applied coating which was subsequently exposed to UV radiation. With this foil, an exposure time of 0.15 seconds was found to be required for complete curing as judged by the fingertip method. A degree of cure of 87 per cent was achieved.

This demonstrates clearly that although a relatively low degree of cure is reached, a hard coating can be obtained if air inhibition can be overcome. It further proves that air inhibition is a very important cure determining factor.

Experiments under foil

In order to exclude the disturbing influence of air, further experiments were performed in such a way that air inhibition could no longer occur.

Therefore, the experiments carried out earlier were repeated by placing a foil over the coatings before curing. The cure speeds were measured in the same way as before. A general effect observed in all experiments carried out in this way was: a decrease in the exposure time necessary to achieve a hard and dry film.

The effects of the parameters on the cure speed observed during the previous experiments and described earlier disappeared when working with the exclusion of air. The next Figures demonstrate the results obtained. For instance, Figure 12 shows the relationship between exposure time and

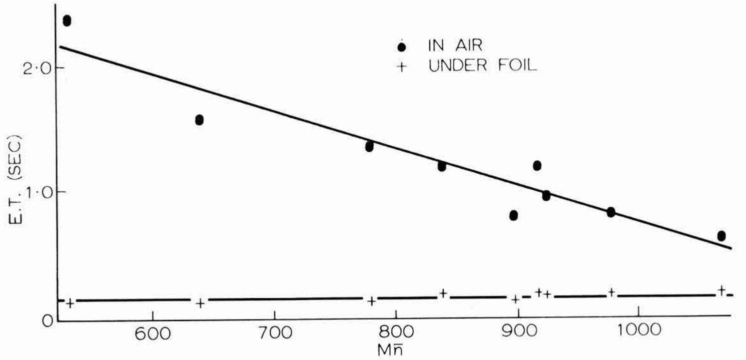

Fig. 12. Exposure time as a function of *Mn̄* of pure polyesteracrylates

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$M\bar{n}$ for pure polyesteracrylates. The upper line is reproduced from the line from Figure 2 .The lower line gives the results of the experiments under foil.

Figure 13 demonstrates the disappearance of the dependence of ET on the viscosity when working with the exclusion of air.

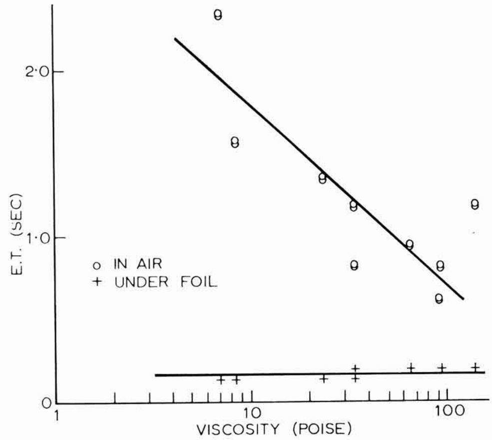

Fig. 13. Exposure time as a function of viscosity of pure polyesteracrylates

Figure 14 shows that the anomalous effect of the amount of acrylic double bonds on exposure time as observed in the presence of air (where this factor was changed by changing the polyesteracrylate at constant X) also disappeared.

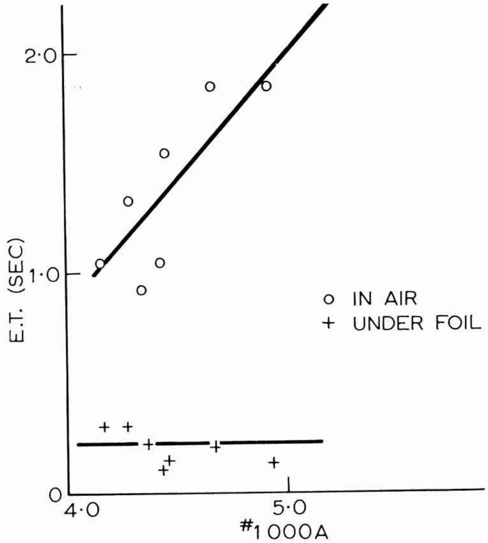

Fig. 14. Exposure time as a function of amount of acrylic double bonds. Experiments at constant X.

Discussion

The parameters found to influence the cure speed when the experiments were carried out in air were found to be:

$M\bar{n}$	for pure polyester acrylates
F_A	for pure polyester acrylates
$M\bar{o}$	for mixtures
X	for mixtures
# 1000A	for mixtures
$M\bar{n}$	for mixtures with high percentage of polyesteracrylate.

An analytical method to measure the amount of double bonds reacted was outlined. Using this method it was proved that in addition to the oligomer parameters, air inhibition is an important factor influencing cure speed.

Finally, experiments under foil were described which showed that when air is excluded:

— fast cure speeds are achieved

— relatively low degrees of cure give hard films.

Working with the exclusion of air, achieved either by a foil or by using inert gas equipment, gives a very great improvement in cure speed. When making calculations regarding the economics of UV curing, the possibility of working under foil or in an inert atmosphere must certainly not be forgotten. If very fast cure speeds are required at very low viscosities, it is worthwhile to consider working with the exclusion of air.

It should be borne in mind, however, that working under inert atmosphere will give hard films, but at the moment when this hard film is obtained the degree of cure is still in the order of only 87 per cent. Possibly this will cause troubles, such as postcure by sunlight or toxic residues of monomers in the coating.

On the other hand, when working in air there are possibilities in formulating oligomers with the right properties.

The last Figures suggest that there are formulations that come close to exposure times needed in the experiments done under a foil.

Finally, a comparison has been made of the two conditions under which UV curing can be carried out.

Table 9 shows the advantages and drawbacks of both curing methods.

Table 9

A comparison between two conditions under which UV curing can be carried out

	UV curing in air.	UV curing under inert condition.
1)	Special formulation required for high cure speed.	Wider formulation possibilities for obtaining high cure speed
2)	Little residual monomer content in final coating.	Monomer content in final coating relatively high.
3)	Limited choice of oligomers for formulating blends.	Wider range to choose from for formulating blends.
4)	No extra investments required.	Additional equipment needed.

Conclusions

As already stated, oligomer properties are not the only factors determining cure speed.

All the elements in the formulations are of importance. The lamps are also very important.

It is now possible to change energy output and spectral lines of the lamp. The first experiments show that also here there is a possibility of influencing cure speed.

It is, of course, very important to choose the right photoinitiator to react with the spectral lines supplied by the lamps.

Acknowledgments

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The use of differential scanning calorimetry in photocuring studies*

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Summary

A method has been developed for measuring the reaction kinetics of photoinitiated surface coating materials. A Perkin Elmer differential scanning calorimeter Model IB has been adapted to allow such measurements to be made under a variety of conditions which are accurately controlled. The effect of varying curing temperatures and light intensity on a polyester/styrene composition is demonstrated and discussed.

Keywords

Processes and methods primarily associated with analysis, measurement or testing

calorimetry

drying or curing of coatings

ultraviolet curing

Properties, characteristics and conditions primarily associated with materials in general

reactivity

coatings during application

curing rate

L'emploi de calorimétrie differentielle à balayage au cours des études du processus de photodurcissement

Résumé

On a mis au point une méthode pour déterminer la cinétique de réaction des revêtements photo-initiables. Un calorimètre differentiel à balayage du type Perkin Elmer Modele 1B a été modifiée afin de permettre la détermination de telles mesures sous les conditions qui sont précisement contrôlées. A la fois on discute et démontre l'effet qu'exerce la variation de la température de durcissement et de l'intensité de lumière sur un revêtement à base de polyester et de styrolène.

Die Anwendung der Differential Abtastfarbmessung für Photohärtungsuntersuchungen

Zusammenfassung

Es wurde eine Methode zur Messung der Kinetischen Reaktionsenergie von photoinitiierten Beschichtungsmitteln entwickelt. Ein Perkin Elmer Differential-Abtastcolorimeter Modell IB wurde so angepasst, dass es solche Messungen unter verschiedenen, genau kontrollierten Bedingungen erlaubte. Die Auswirkung verschiedener Härtungstemperaturen und Lichtintensität wird an Hand einer Polyester/Styrol Rezeptur aufgezeigt und besprochen.

Introduction

Ref. 1

The work described in this report was carried out as a service activity to a research programme directed towards the development of photocuring surface coatings.

In initial experiments in this programme the effect of varying various parameters on the cure rate had been estimated by measuring the time for the system to reach a gel point. It was realised that this was not an ideal measurement, since the point at which a gel is formed is itself a dependent variable.

Earlier work in these laboratories by K. E. J. Barrett and H. R. Thomas had utilised the Perkin Elmer differential scanning calorimeter (DSC) to follow reaction kinetics[1]; it was, therefore, decided to modify the calorimeter in order that photoinitiated reaction kinetics could be examined. By using the DSC the conditions under which reactions were carried out were accurately controlled and flexible.

The object of the experiments was to aid research chemists in producing rapid curing, commercially viable photocuring surface coatings, rather than to examine the mechanism of the photocuring system itself.

Experimental

Apparatus

Ref. 1

In the conventional design of the Perkin-Elmer DSC IB it is not possible to use the instrument below about 50°C without using a low temperature cover made of steel and filled with liquid nitrogen to provide a cold "heat sink". Since this completely encloses the sample pans, it is impossible to carry out any photocuring work with the existing system at temperatures below 50°C.

Modifications were made such that:

(1) The instrument was capable of operating down to 0°C.

(2) Light could be shone into the reaction can.

(3) Access to the sample pans could easily be obtained.

(4) The whole assembly could be reproducibly assembled.

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*Paper presented at the Newcastle Section Symposium on "Ultraviolet curing" held at the University of Durham on 14-15 September 1977.

Accordingly, the apparatus shown in Figs. 1 and 2 was constructed. It consists of a brass cover which has a cooling coil wrapped round the exterior surface, and a removable lid, into which a brass-clad rigid light guide fits. All seals are made by "O" rings. To ensure reproducibility in assembling the apparatus, a key-way was cut in the cover lid which aligns with a peg fitted to the DSC. Thus, the light guide is always in the same position with respect to the DSC sample pan holders. A rigid light guide provides a cold light source, and is easier to align than normal optical systems.

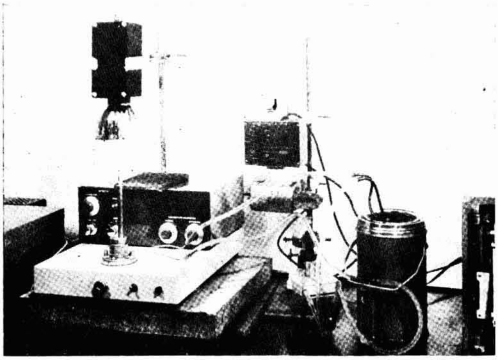

Fig. 1.

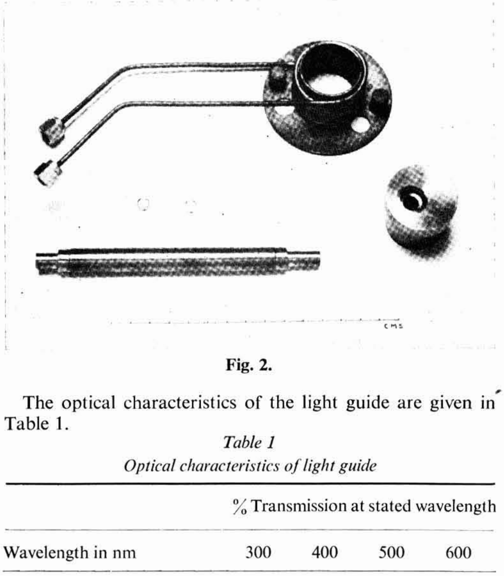

Fig. 2.

The optical characteristics of the light guide are given in Table 1.

Table 1

Optical characteristics of light guide

	% Transmission at stated wavelength			
Wavelength in nm	300	400	500	600
150mm visible conducting light guide	~0	72	86	86

Following the method of Barrett & Thomas[1], transistor cans were used as sample holders. These were modified as shown in Fig. 3. The mouth of the can was enlarged by a simple press and die, such that a conventional Perkin Elmer non-volatile sample pan could be used as light-tight lid, and a shoulder was formed in the can to support a disc of transparent sheet which lessened the loss of volatiles during high temperature curing.

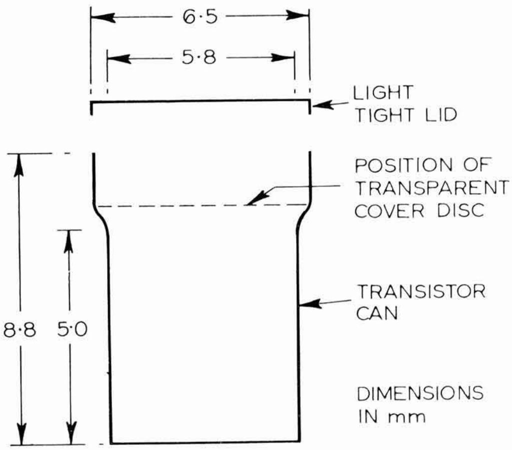

Fig. 3. Cross section of modified transistor can

The diameter of the base of the transistor can is significantly less than that of the DSC sample pan holder, so that to ensure reproducible positioning of the transistor cans, small crescent shaped spacers were used which were a snug fit inside the sample pan holders, and into which the sample cans were themselves a tight fit. (See Fig. 1).

Since the DSC operates on a differential principle, if light is shone on only one pan a deflection proportional to the incident energy is observed, i.e. the instrument may be used as a bolometer. The reproducibility of the system was checked by repeatedly assembling the apparatus with the lamp position unaltered, and measuring the deflection on the instrument due to the incident light energy. This deflection showed no variation.

The only part of the apparatus which has no positive location is the light source. For the following experiments a Phillips HPR, MBR/U 125 watt mercury vapour lamp, was used. This lamp has been aligned by:

(a) measurement from the top of the light guide.

(b) adjustment until the DSC gave a set deflection between the lamp shuttered, and lamp on, positions.

The cooling system consists of a coil wrapped round the DSC cover, which is connected by flexible tubing to a copper coil immersed in a Dewar flask filled with acetone/Drikold mix. The heat transfer liquid, ethanol, is pumped round the system by a peristaltic pump, silicone rubber tubing being used for this section of the circuit, since it does not stiffen at the temperatures attained. A small expansion/contraction chamber is included in the circuit.

To enable measurements of the effect of intensity on curing rate to be carried out, a series of filters was made by partially vacuum-coating glass panels with aluminium. Some neutral density filters of commercial origin were also used, but many such filters were far from neutral. Transmission spectra of the filters used are given in Fig. 4.

To check that the range of light intensities being used for DSC experiments was comparable with that being used for practical work elsewhere, a crude light meter was constructed. This consisted of a photoresistor (RCA type SQ2535), the resistance of which was monitored by a AVOmeter. The

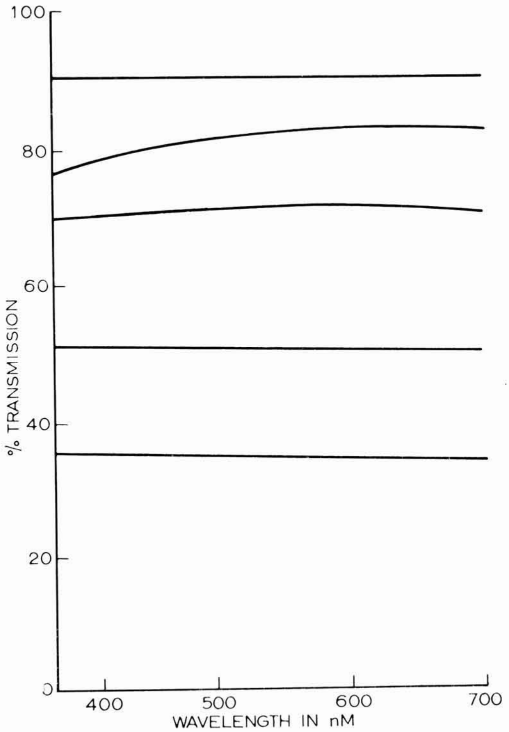

Fig. 4. Characteristics of neutral density filters

photoresistor was mounted in a reaction-type transistor can so that it was possible to place it on the DSC in exactly the same way as reaction cans. The light intensities used for curing were higher than the upper limit of the photoresistor.

Neutral density filters were therefore incorporated into the aperture of the transistor can to reduce the intensity reaching the light sensor. Intensities commonly used for large scale curing of surface coatings were found to be approximately 50 per cent of the maximum intensity used in DSC experiments.

Technique

In the light of early experiments, a technique of determining kinetic data for photocuring systems was evolved. This is as follows:

(a) A transistor can is modified as in Fig. 3.

(b) A transparent cover disc is cut from acetate sheet and a Perkin-Elmer sample pan is selected to act as a "light tight" lid.

(c) Can, cover and lid are weighed (±0.1 mg).

(d) Approximately 50mg of initiated reactant is placed in the can, the cover disc is placed on the surface of the reactants and the lid placed on the can to make it "light tight".

(e) The total assembly is weighed to obtain the weight of reactants by difference.

(f) The can is placed on the DSC pan at 45°C. The transistor can lid is removed, and the brass lid and light guide re-positioned. The lamp is shuttered during this operation.

(g) A short time is allowed for a nitrogen gas purge to remove any damp air before switching on the cooling system.

(h) Reaction temperatures, sensitivity, chart speed etc. are selected. Typical settings are:

Reaction temperature: 25°C
Sensitivity : 32m cals/sec. Full scale deflection.
Chart speed : 30 inches/hr
Sample weight : ~50mg.

(i) When the baseline has become steady, the lamp is unshuttered, and the reaction exotherm is traced on the chart.

(j) When the rate of reaction drops to near zero, the temperature of the reaction is raised to 80°C and then 120°C to obtain complete reaction ("post cure").

(k) When the rate of post cure has reached zero, the temperature is lowered to the original reaction value and the lamp shuttered.

(l) When temperature stability has been achieved, the lamp is unshuttered. The resultant trace gives the deflection of the recorder pen due solely to the light energy falling on the DSC. By subtracting this from the reaction trace, the exothermic heat of reaction may be measured.

(m) The temperature is again increased to 80°C and 120°C. The deflection of the trace is subtracted from that previously obtained to give the true reaction energy.

(n) The cured sample is removed from the DSC and weighed to check that no excessive weight loss has occurred (usually <2.0 per cent).

(o) Since the area under the DSC trace represents the total heat of reaction, this is calculated by photo-copying the trace, cutting out the required area and weighing. Comparison with a standard area then allows an estimate of the total heat of reaction.

An obvious improvement to the above system is to use rigid light guides over both sample and reference pans, to eliminate the displacement of the recorder pen due solely to the incident light energy. This should remove the necessity of steps (l) and (m) above, with a consequent saving of time, and improvement in accuracy.

Results

Preliminary experiments

Ref. 2

Originally calorimetric work was requested, to determine the amount of residual monomer in systems which had been photocured, by measurement of exothermic energy of thermal polymerisation of any such residual monomer. No evidence of thermal polymerisation could be found.

However, when a mercury vapour lamp was shone on the uncured reaction mix an immediate exotherm was observed.

On shuttering the lamp the exotherm decayed away. A typical trace of this type is illustrated by Fig. 5. This initial work was carried out without modification to the DSC, merely placing a lamp above the conventional high temperature cover. All other experiments utilised the equipment previously described.

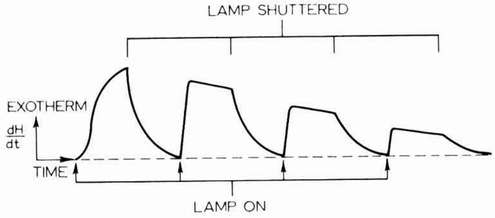

Fig. 5.

To check the reproducibility of the technique, four cures at 57°C and three at 70°C of polyester/styrene mix initiated by a ketone/amine system were examined. The results are shown in Figs. 6 and 7. Better reproducibility has been obtained with more reactive resins, which allow the instrument to be operated at lower sensitivities, giving a better signal to noise ratio.

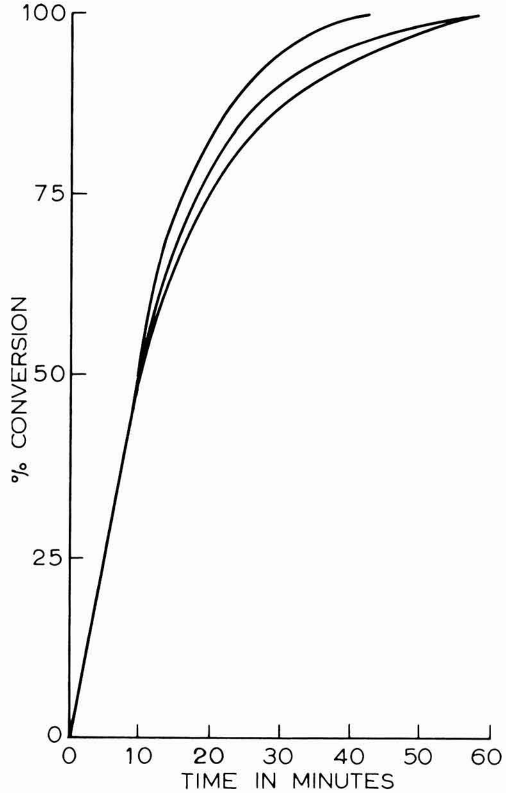

Fig. 6. Polyester/styrene (60/40) cured at 57°C

A source of inaccuracy inherent in measuring kinetic data in photochemistry is the variation between local and average rates of reaction due to the absorption of light as it passes through the sample. However, it has been calculated[2] that there is no large difference between the total measured rate and the local rate, as long as the percentage of incident light absorbed by the reaction mixture is kept below about 50 per cent.

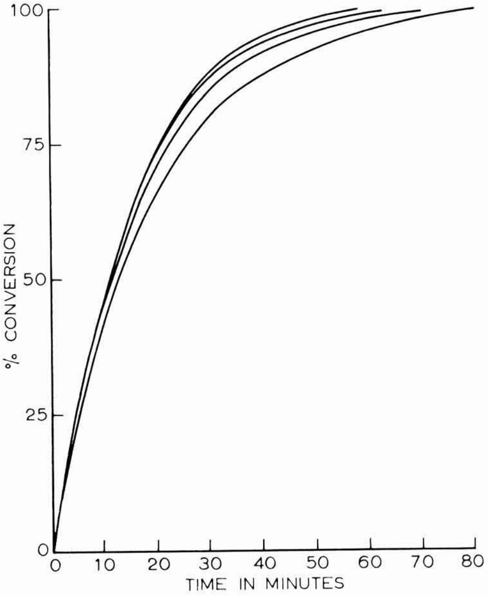

Fig. 7. Polyester/styrene (60/40) cured at 70°C

To measure the absorption of samples, a wedge of each was prepared and the absorption spectra at various thicknesses measured. This gave a calibration curve of absorption at any given wavelength, versus thickness. From this it was calculated that, under the experimental conditions defined above, less than 50 per cent of the incident light is absorbed in the reaction mixture.

Effect of temperature on cure rate and degree of conversion

Isothermal cures of Roskydal 650/styrene (60/40) systems using a ketone/amine initiating system at temperatures covering the range from 0°C to 127°C were carried out. The initial part of the cure (i.e. up to 25 per cent conversion) appeared to follow first order kinetics and from an Arrhenius plot of the 1st order rate constants against reciprocal temperature, a value of the activation energy of the system of 3K cal/mole of double bonds was obtained (Fig. 8).

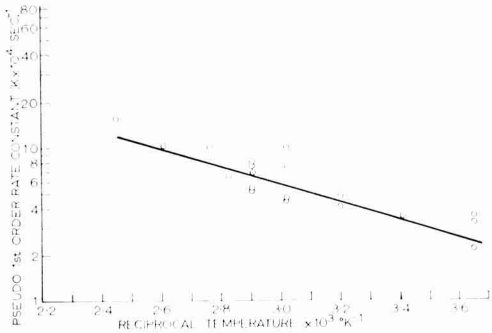

Fig. 8. Arrhenius plot for polyester/styrene (60/40)

Similar cures with a more reactive polyester mixed 70/30 with styrene gave the results shown in Fig. 9. The activation energy for this system was 5K cal/mole of double bonds. From Fig. 9 and from data on the Roskydal system, it is clear that not only the rate of reaction but also the final degree of conversion is temperature dependent. If the limiting mechanism in the final conversion is the ease with which monomer may diffuse through the polymer to active sites, then it could be expected that the glass transition of the polymer network would be the decisive factor. As the polymer network grows, so less monomer remains to act as a plasticiser, thus eventually a point is reached where the system becomes glassy. The higher the temperature of reaction, the smaller the amount of monomer required to retain the non-glassy state, therefore the higher the limiting percentage reaction. Attempts to measure the *Tg* of the resulting cured resins on the DSC were unsuccessful, since no discontinuity in the specific heat of the material could be observed.

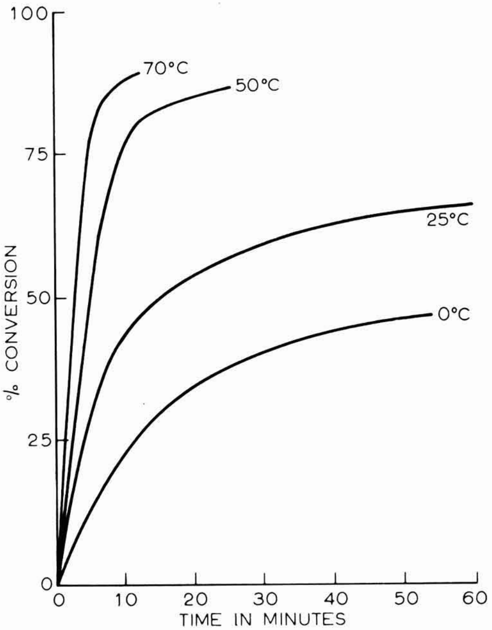

Fig. 9. Cure rate temperature dependence of experimental polyester/styrene (60/40)

In order that little residual styrene monomer should be left in the system, it would appear that curing should be carried out at temperatures at least as high as 50°C.

Dependence of cure rate on intensity

The intensity of the curing radiation was reduced from the maximum level used for all other experiments by steps down to 1/5th the maximum. Data obtained from cures at reduced intensities was plotted as pseudo 1st order reactions, giving linear plots up to 25 per cent conversion. The variation of the first order rate constants with light intensities for the Roskydal 650/styrene (60/40) systems at 57°C is shown in Fig. 10.

This plot gives the relationship:

Initial rate of cure $\propto$ Intensity $^{\frac{1}{2}}$

Data on the rate of reaction at higher conversions is considered unreliable, particularly at low levels of illumination, since the signal to noise ratio becomes critical.

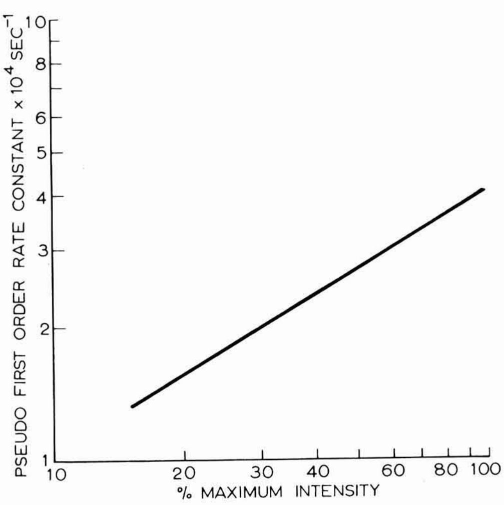

Fig. 10. Rate constant/intensity for polyester/styrene (60/40) cured at 57°C

Conclusions

The differential scanning colorimeter (Perkin Elmer Model 1B) has successfully been adapted to measure the reaction kinetics of photocuring polymer used in surface coatings. This makes possible the following conclusions for the systems studied, which are based solely on DSC results.

(a) If residual styrene monomer concentrations of less than 15 per cent of the total styrene in the formulation are required on the completion of photocuring, temperatures in excess of 50°C must be used.

(b) The activation energy of the total system is calculated to be 5K cals/mol of double bonds.

(c) The initial rate of cure is proportional to the square root of the incident light intensity.

Acknowledgements

The authors would like to thank Dr F. W. Hobden for his advice and encouragement and Mr G. Lamb for constructing the additional equipment.

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The UV-curing behaviour of some photoinitiators and photoactivators*

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Summary

The criteria for the selection of suitable photoinitiators for white pigmented resin formulations are outlined. A number of aromatic ketones, some of them novel compounds, have been synthesised and their properties examined with reference to these criteria. From this investigation, two thioxanthone derivatives were selected for examination with various amine photoactivators. On the basis of the results obtained, two aminobenzoate-type photoactivators are suggested for use in conjunction with the thioxanthones as safe, cost-effective UV-curing systems for titanium dioxide-pigmented coating formulations.

Keywords

Processes and methods primarily associated with manufacturing or synthesis

UV curing

analysis, measurement or testing

ultraviolet spectroscope

drying or curing of coatings

radiation curing

photoinitiation

photoactivation

Le comportement, en systèmes durcissâbles par les rayons ultra violets, de certains photo-initiateurs et photo-activeurs

Résumé

On trace les grandes lignes des critères qui gouvernent la sélection des photo-initiateurs convenables à être incorporés dans les formulations à base de pigments blancs et de résines. Un certain nombre de cétones aromatiques, dont quelques-unes sont de nouveaux composés, ont été synthésisées et leurs caractéristiques ont été étudiées à l'égard de ces critères. En raison de cette investigation, on a choisi deux dérivés de thioxanthone et qui ont été étudiés en combinaison avec de divers photo-amorceurs aminés. Basé sur les résultats obtenus, deux photo-amorceurs du type aminobenzoate sont suggérés à être utilisés en combinaison avec les thioxanthones en tant qu'agents de durcissement par les rayons ultra violets, qui sont sûrs et efficaces au point de vue du prix, pour les revêtements pigmentés avec le dioxyde de titane.

Das UV-Härtungsverhalten einiger Photoinitiatoren und Photoaktivatoren

Zusammenfassung

Es werden die Kriteria für die Auswahl geeigneter Photoinitiatoren für weiss pigmentierte Lackrezepte skizziert. Eine Anzahl von aromatischen Ketonen, darunter ganz neue Verbindungen, wurden synthetisch hergestellt, und ihre Eigenschaften mit Bezug auf diese Kriteria geprüft. Das Resultat dieser Untersuchungen führte zur Auswahl von zwei Thioxanthonen zur weiteren Prüfung mit verschiedenen Amin-Photoaktivatoren. Als Resultat dieser Arbeiten wird die Verwendung zweier Aminobenzoat-Typ Photoaktivatoren vorgeschlagen und zwar in Verbindung mit Thioxanthon, als verlässliche, wirtschaftliche UV-Härtungssysteme für mit Titandioxid pigmentierte Beschichtungsmittel.

Introduction

Refs. 1, 2, 3

The production of conventionally-cured white (usually titanium dioxide) pigmented coatings in the UK alone is at present in the order of 1000 tons per annum. Because of the potential energy savings[1] and other now well-known advantages, there is a considerable incentive within the surface coatings industry to develop UV-curable formulations for this application.

The problems encountered in the UV-curing of relatively thick pigmented films are two-fold: the pigment can reflect or absorb incident UV light, thus generally diminishing the availability of the light to the photoinitiator; and the opacity of thicker pigmented films can result in a poor through-cure. The complex question concerning the calculation of absorption of UV light by photoinitiators in pigmented coatings has recently been examined by Wicks and Kuhhirt[2].

Titanium dioxide shows a high reflectivity throughout the visible range of the spectrum and strong absorption characteristics at wavelengths below 340 nm. The region 350-400 nm represents a transition between the reflection and absorption of light, and is, therefore, optimal with regard to light availability to a photoinitiator (Figure 1).

2-Chlorothioxanthone (CTX) is an aromatic ketone with absorption bands at 260 nm, and 385 nm and it has been proposed that the latter band renders CTX particularly suitable as a photoinitator for titanium dioxide-pigmented formulations[3]. This proposal has been borne out subsequently by much laboratory experience. The mechanism by which a photoinitiator such as CTX operates is as follows: absorption of light by CTX results in the promotion of the ketone group

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*Paper presented at the Newcastle Section Symposium on "Ultraviolet curing" held at the University of Durham on 14-15 September 1977.

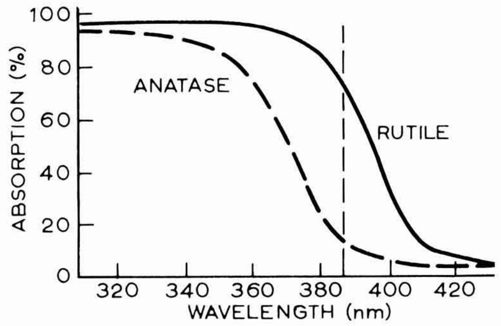
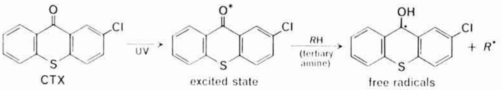


Fig. 1.

to a chemically reactive excited state. In the presence of a hydrogen donor such as a tertiary amine, the excited-state ketone group abstracts hydrogen, with a resultant formation of free radicals. These free radicals can then initiate the polymerisation of the resin binder system:

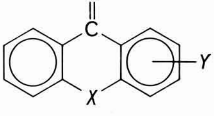


In white-pigmented formulations, the photoinitiator characteristics of main importance to the formulator and user are as follows:

(1) UV-curing performance.
(2) Colour (as far as it affects the colour of the cured film).
(3) Solubility/ease of incorporation.
(4) Effect on the stability of the formulation.
(5) Physiological aspects (toxicity; odour of decomposition products).
(6) Cost.
(7) Patent position.

The order and extent of importance of these criteria will vary according to the nature of the application of the formulation.

It was of interest to examine compounds similar to CTX with regard to the above criteria. CTX may be considered to be notionally derived from benzophenone:



Benzophenone: $Y = \mathrm{H}$; $X =$ nothing
CTX: $Y = \mathrm{Cl}$; $X = \mathrm{S}$.

The compounds investigated in this work programme are shown in Table 1, with X and Y as used in Figure 2.

Experimental

Synthetic routes

Refs. 4-11

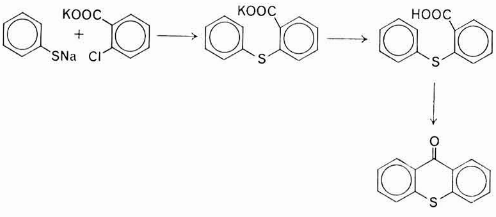
Examples of reaction sequences by which the compounds may be prepared are given below; in most cases alternative synthetic routes exist.

Table 1

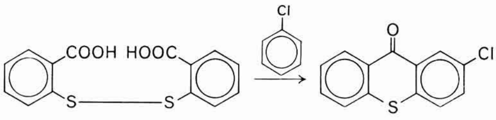
Compound	X	Y
Thioxanthone	—S—	H—
2-Chlorothioxanthone (CTX) (*a*)	—S—	Cl—
2-Methylthioxanthone	—S—	CH_3—
2-Ethylthioxanthone	—S—	CH_3CH_2—
2-Isopropylthioxanthone (ITX) (*a*)	—S—	$(CH_3)_2CH$—
2-tert-Butylthioxanthone	—S—	$(CH_3)_3C$—
*2-Phenylthioxanthone (M.Pt. 130-131.5°C)	—S—	C_6H_5—
2-Benzylthioxanthone	—S—	$C_6H_5CH_2$—
*2-Cyclohexylthioxanthone (M.Pt. 86-88°C)	—S—	C_6H_{11}—
*4-Isopropylthioxanthone (M.Pt. 104-106°C)	—S—	$(CH_3)_2CH$—
2-Acetylthioxanthone	—S—	CH_3CO—
2-Chloroxanthone	—O—	Cl—
Fluorenone	single bond	H—
Dibenzosuberone	—CH_2CH_2—	H—
6,11-Dihydrodibenzo(b,e)-thiepin-11-one	—CH_2S—	H—
2-Chloro-6,11-dihydrodibenzo (b,e)-thiepin-11-one	—CH_2S—	Cl—

*Novel compounds
(*a*) Ward Blenkinsop designations

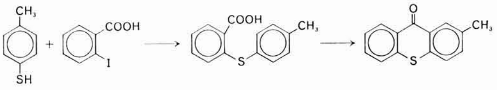
Thioxanthone[4,5]



2-Chlorothioxanthone[6]

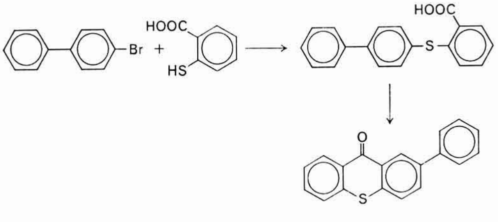


2-Methylthioxanthone[7]

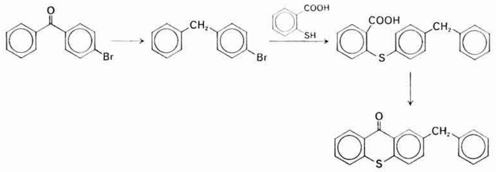


The 2-ethyl, 2-isopropyl, 2-tert-butyl and 2-cyclohexylthioxanthones may be prepared by a similar sequence using the appropriate p-alkylthiophenols.

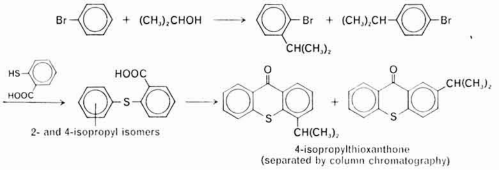
2-Phenylthioxanthone[4]



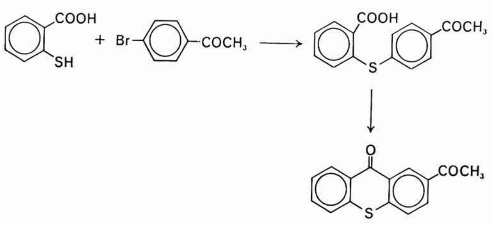
2-Benzylthioxanthone[4]



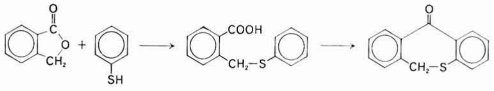
4-Isopropylthioxanthone[4]



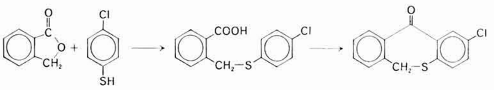
2-Acetylthioxanthone[4,8]



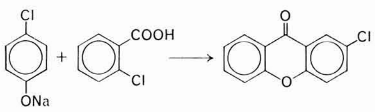
6-11-Dihydrodibenzo (b,e)-thiepin-11-one[9,10]



2-Chloro-6,11-Dihydrodibenzo (b,e)-thiepen-11-one[9,10]



2-Chloroxanthone[11]



Fluorenone and dibenzosuberone were obtained from the Aldrich Chemical Co., Gillingham, Dorset.

Colour

A visual assessment of colour (Table 2) was made using chromatographically purified specimens of the compounds.

Table 2

Compound (solids except where otherwise noted)	Colour
Thioxanthone	Yellow
2-Chlorothioxanthone	Yellow (paler than methyl-thioxanthone)
2-Methylthioxanthone	Yellow
2-Ethylthioxanthone	Very pale yellow
2-Isopropylthioxanthone	Very pale yellow
2-tert-Butylthioxanthone	Extremely pale yellow–ivory
2-Phenylthioxanthone	Pale yellow
2-Benzylthioxanthone	Very pale yellow
2-Cyclohexylthioxanthone	Very pale yellow
4-Isopropylthioxanthone	Very pale yellow
2-Acetylthioxanthone	Yellow
2-Chloroxanthone	White
Fluorenone	Yellow
Dibenzosuberone (liquid)	Extremely pale yellow
6,11-Dihydrodibenzo(b,e)-thiepin-11-one	Very pale brown
2-Chloro-6,11-Dihydrodibenzo-(b,e)-thiepin-11-one	Very pale yellow

Solubility

Solubilities shown in Table 3 refer to percentage w/v of compound.

Table 3

Compound	Solvent						
	1,2-dichloro ethane	1,2-dichloro propane	Toluene	Acet-one	Ethan-ol	Meth-anol	Ethyl acetate
Thioxanthone	1.1	<0.5	<0.5	<0.5	<0.5	<0.5	0.5
2-Chlorothio-xanthone	7.1	3.1	3.3	1.4	<1.0	<1.0	—
2-Methylthio-xanthone	16.6	7.7	5.5	3.0	<1.0	<1.0	2.9
2-Ethylthio-xanthone	10.0	5.5	5.0	2.3	<0.5	<0.5	2.8
2-Isopropyl-thioxanthone	>50.0	>50.0	>50.0	30.0	3.6	3.3	30.0
2-tert-Butyl-thioxanthone	>50.0	>50.0	>50.0	>40.0	8.0	5.0	>50.0
2-Phenylthio-xanthone	8.3	3.8	3.6	1.3	<1.0	<1.0	1.4
2-Benzylthio-xanthone	25.0	11.4	6.9	2.6	<1.0	<1.0	2.8
2-Cyclohexyl-thioxanthone	36.0	36.0	36.0	4.2	1.1	<1.0	7.2

Table 3—Continued

4-Isopropylthioxanthone	15.0	12.5	12.5	4.2	1.0	<1.0	5.0
2-Acetylthioxanthone	1.1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2-Chloroxanthone	3.3	2.1	2.0	1.0	<0.5	<0.5	1.2
Fluorenone	54.5	38.5	35.7	41.7	6.7	6.7	27.8
Dibenzosuberone		Miscible in all Proportions				Immiscible between 31.0-50.0	Miscible in all proportions
6,11-Dihydrodibenzo (b,e)-thiepin-11-one	5.0	5.0	5.0	5.0	2.0	2.5	5.0
2-Chloro-6,11-dihydrodibenzo-(b,e)-thiepin-11-one	5.0	5.0	5.0	3.3	<0.5	<0.5	3.3

Transmission spectra and UV-curing performance

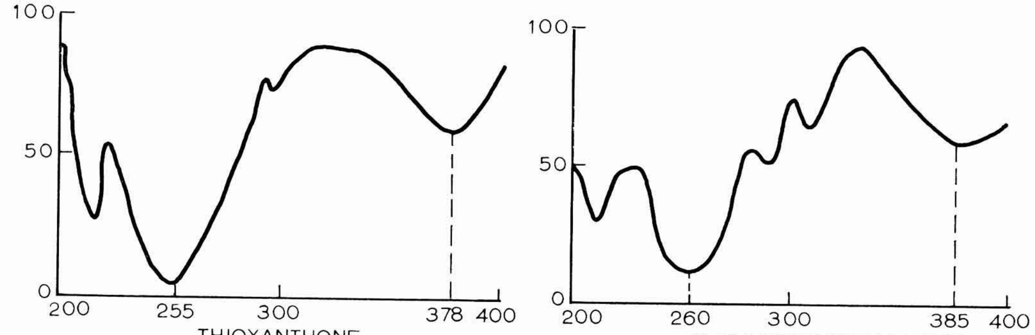
Refs. 12-17

The UV transmission spectra for the compounds are shown below; in each case:

the X-axis shows the wavelength 200-400 nm

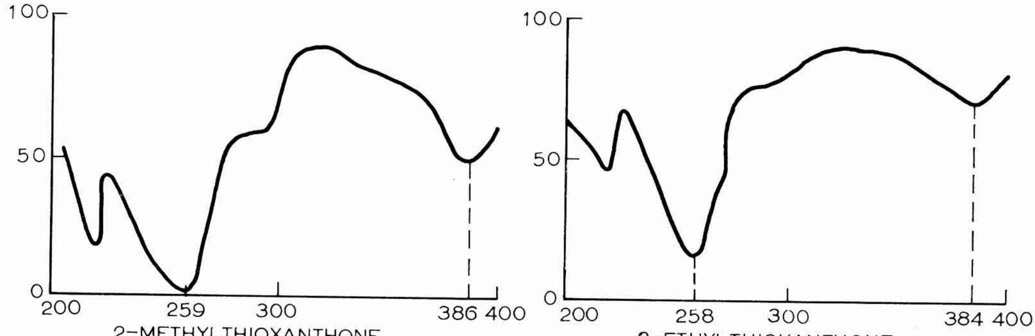
the Y-axis shows the transmission 0-100 per cent.

All measurements were carried out in methanol (spectroscopic grade) using a 1 cm cell. Percentages shown refer to the concentration of ketone in methanol.



THIOXANTHONE (0·0006%)

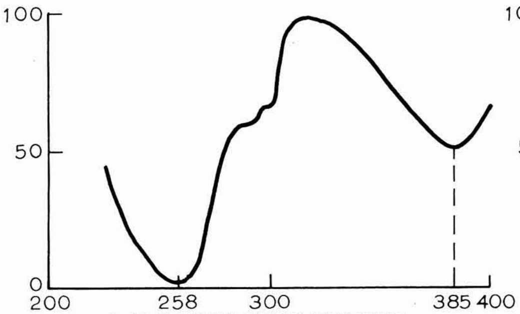
2-CHLOROTHIOXANTHONE (0·00097 %)



2-METHYLTHIOXANTHONE (0·0012 %)

2-ETHYLTHIOXANTHONE (0·000435 %)

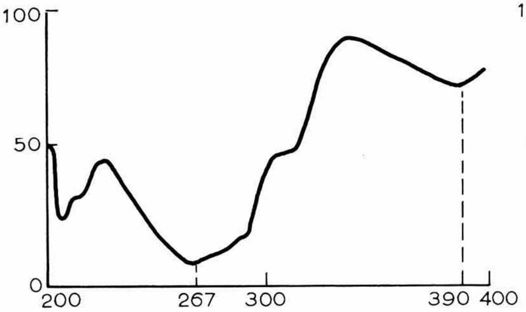
Fig. 2. (Part A)



2-ISOPROPYLTHIOXANTHONE (0·001%)

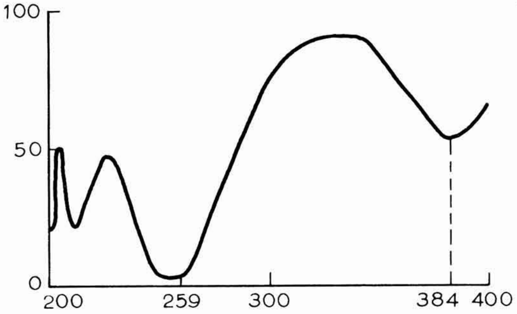
2-tert-BUTYLTHIOXANTHONE (0·0019 %)

Fig 2. (Part A—*continued*)



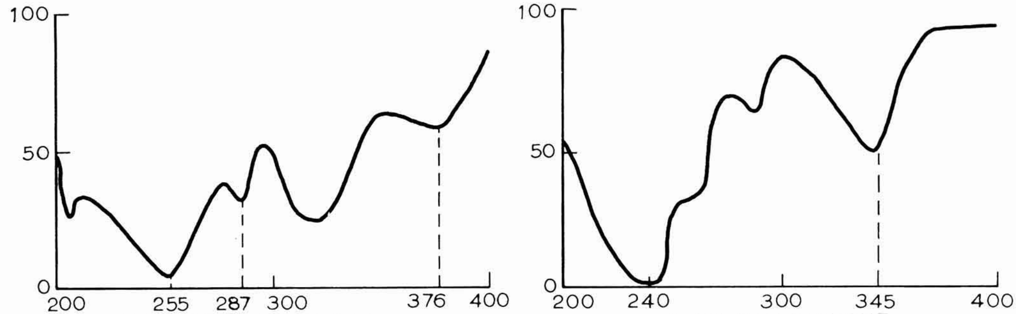
2-PHENYLTHIOXANTHONE (0·000755%)

2-BENZYLTHIOXANTHONE (0·00105%)



2-CYCLOHEXYLTHIOXANTHONE (0·00115 %)

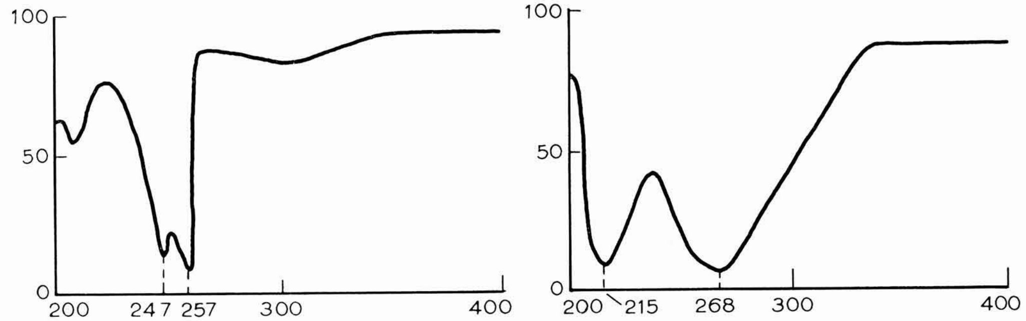
4-ISOPROPYLTHIOXANTHONE (0·0068%)



2-ACETYLTHIOXANTHONE (0·001%)

2-CHLOROXANTHONE (0·00112 %)

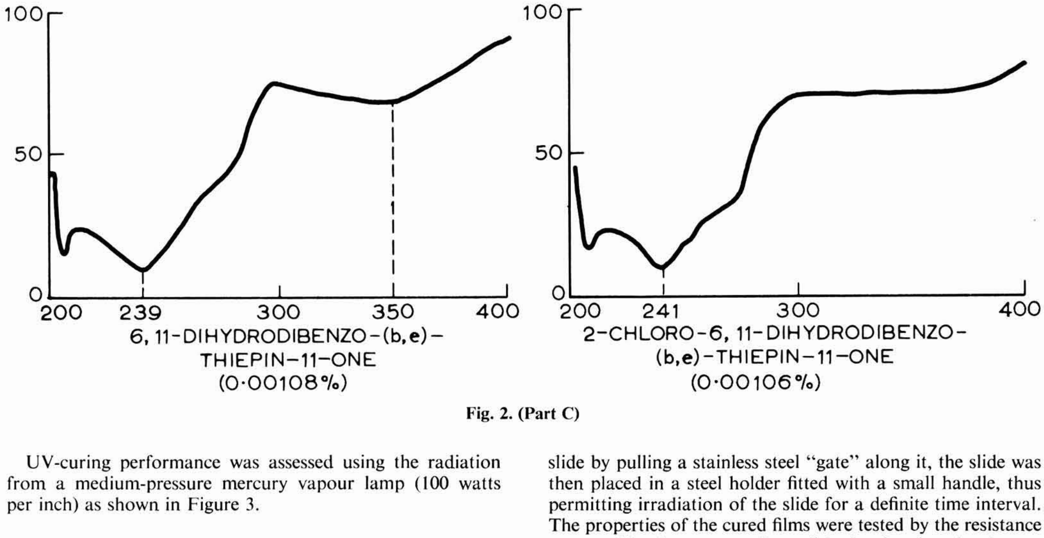
Fig. 2. (Part B)



FLUORENONE
(0·000225%)

DIBENZOSUBERONE
(0·00225%)

Fig. 2. (Part B—*continued*)



6, 11-DIHYDRODIBENZO-(b,e)-THIEPIN-11-ONE
(0·00108%)

2-CHLORO-6, 11-DIHYDRODIBENZO-(b,e)-THIEPIN-11-ONE
(0·00106%)

Fig. 2. (Part C)

UV-curing performance was assessed using the radiation from a medium-pressure mercury vapour lamp (100 watts per inch) as shown in Figure 3.

Fig. 3.

The white-pigmented test formulation was based on the acrylic urethane resin DV530[12] containing 40 per cent w/w titanium dioxide, R-TC2 grade[13]. Each initiator was tested at a 2 per cent level in this formulation, together with 2 per cent N,N-dimethylaminoethanol as the photoactivating amine. Some of the resultant mixture was transferred to one end of a microscope slide and a 25 micron thick film formed on the slide by pulling a stainless steel "gate" along it, the slide was then placed in a steel holder fitted with a small handle, thus permitting irradiation of the slide for a definite time interval. The properties of the cured films were tested by the resistance to scratching by a range of pencil leads of various hardnesses (2B (softest) to 6H (hardest)).

In most cases, cure-speed is the characteristic of prime importance.

From Table 4, it can be seen that 2-chloro-, 2-methyl-, 2-ethyl-, 2-isopropyl-, 2-tert-butyl-, 2-benzyl-, 2-cyclohexyl-, and 4-isopropyl-thioxanthones are best in this respect. Moreover, the 2-ethyl-, 2-tert-butyl and 4-isopropyl-compounds give films free of a yellowcast. Other compounds which give white films have a very slow cure-rate. In addition, all the alkylthioxanthones which give a good cure-rate have good solubilities, implying ease of incorporation into a formulation.

However, in practice, economic considerations are also of great importance. It has been found from an investigation of available synthetic routes, 2-chlorothioxanthone and 2-isopropylthioxanthone optimise performance, colour, solubility and cost. 2-Chlorothioxanthone has a disadvantage with regard to solubility, but it can be dispersed adequately in a formulation in the same manner as a pigment, i.e., by milling. At no time have stability problems been encountered

Table 4

Compound	Irradiation time (seconds)	Pencil hardness	Appearance of film
Thioxanthone	45	>6H	very pale yellow
2-Chlorothioxanthone	30	>6H	very pale yellow/high gloss
2-Methylthioxanthone	30	>6H	distinctly yellow
2-Ethylthioxanthone	30	>6H	white/high gloss
2-Isopropylthioxanthone	30	>6H	very pale yellow/high gloss
2-tert-Butylthioxanthone	30	>6H	white/high gloss
2-Phenylthioxanthone	45	>6H	pale yellow/high gloss
2-Benzylthioxanthone	30	>6H	very pale yellow/high gloss
2-Cyclohexylthioxanthone	30	>6H	very pale yellow/high gloss
4-Isopropylthioxanthone	30	>6H	white/high gloss
2-Acetylthioxanthone	60	>2B <HB	pale yellow/high rivelled
2-Chloroxanthone	240	>2B <HB	white/highly rivelled
Fluorenone	60	>6H	white/high gloss
Dibenzosuberone	60	>2B <HB	white/highly rivelled
6,11-Dihydrodibenzo(b,e)-thiepin-11-one	120	>2B <HB	white/highly rivelled
2-Chloro-6,11-dihydrodibenzo (b,e)-thiepin-11-one	120	>2B <HB	very pale yellow/highly rivelled

during work on formulations containing 2-chlorothioxanthone or 2-isopropylthioxanthone, nor do they give rise to odorous by-products on irradiation.

Further work was carried out in order to determine whether improvements could be made to the cured film by varying the amine photoactivator.

Twenty-five microns thick films of DV530 resin[12] containing 40 per cent w/w of titanium dioxide R-TC2[13] together with photoinitiator and photoactivator were irradiated for 0.15 seconds in a single lamp (200 watts per inch) "Minicure" oven[14]. The properties of the cured films were tested by finger touch and by pencil hardness.

Thus, when ethyl 4-dimethylaminobenzoate is used as activator with 2-chlorothioxanthone, only one sixth of the amount of the latter is required to give a cured film of similar hardness to that obtained when N,N-dimethylaminoethanol is used. Also, with ethyl 4-dimethylaminobenzoate, a white film is obtained.

Ethyl 2-dimethylaminobenzoate[15] may also be used as a very effective photoactivator. It has the additional advantage that it is a liquid, and has appreciable solvent properties for 2-chloro and 2-isopropylthioxanthones. Using the pigment/resin described above, a combination of 2-chlorothioxanthone with ethyl 2-dimethylaminobenzoate was compared with one containing benzil, another aromatic ketonic photoinitiator (Table 6).

Solutions of 2-chloro- and 2-isopropylthioxanthone in ethyl 2-dimethylaminobenzoate were also compared (Table 7).

Physiological aspects

Determinations of oral LD_{50} (for mice) and dermal LD_{50} (for rats) have been carried out on the most promising photoinitiators and photoactivators described above[4] (Table 8).

Table 5

Percentage 2-Chlorothioxanthone	Photoactivator/ % w/w	Feel	Hardness	Appearance
0.2	Ethyl 4-Dimethylaminobenzoate/2.0	Dry	>6H	White gloss finish
1.2	N,N-Dimethylaminoethanol/2.0	Dry	>6H	Pale yellow gloss finish
1.2	N,N-Dimethylaniline/2.0	Dry	>1H <2H	Pale yellow gloss finish
1.2	Ethyl benzoate/2.0	Dry	>2B <HB	White gloss finish

Table 6

Photoinitiator/ % w/w	Photoactivator/ % w/w	Exposure (seconds)	Pencil hardness	Film appearance
2-Chlorothioxanthone/ 0.2	Ethyl 2-dimethylaminobenzoate/2.0 (EOB) (*a*)	0.15	>6H	White/high gloss
Benzil/0.5	Ethyl 2-dimethylaminobenzoate/2.0 (EOB) (*a*)	0.225	>4H <6H	Pale yellow/ glossy

(*a*) Ward Blenkinsop designation

Table 7

Photoinitiator/ photoactivator	Concentration (%) in TiO_2-pigmented DV530 resin	Exposure (seconds)	Pencil hardness	Film appearance
13% w/w 2-isopropylthioxanthone in ethyl 2-dimethylaminobenzoate (668) (*a*)	2.5	0.15	>6H	White/high gloss
9% w/w 2-chlorothioxanthone in ethyl 2-dimethylaminobenzoate (saturated)	2.5	0.15	>6H	Almost white/ high gloss

(*a*) Ward Blenkinsop designation

Table 8

Compound	Oral LD_{50} (mice; gm. per kgm. body-weight)	Dermal LD_{50} (rats; gm. per kgm. body-weight)
2-Chlorothioxanthone	>1.0	>0.5
2-Isopropylthioxanthone	>1.0	>0.5
Ethyl 4-dimethylaminobenzoate	>4.0	*
Ethyl 2-dimethylaminobenzoate	>1.0	>0.5

*Not determined; the compound has been used for many years as a topical sunscreening agent for cosmetic formulations, and no adverse physiological effects have been noted during manufacture or use.

In addition, 2-chlorothioxanthone and a solution of isopropylthioxanthone in ethyl 2-dimethylaminobenzoate have been subjected to mutagenicity testing[16] by the Ames method[17] and within the context of this method do not appear to have carcinogenic potential. This may be contrasted with the aromatic ketonic photoinitiator benzil, which has been found to be mutagenic[18].

Conclusions

It has been found that in this screening investigation, the photoinitiators 2-chlorothioxanthone and 2-isopropylthioxanthone, when used in conjuction with photoactivators ethyl 4-dimethylaminobenzoate or ethyl 2-dimethylaminobenzoate, are the safest and most cost-effective UV-curing systems for use with titanium dioxide-pigmented resin coating formulations.

Acknowledgments

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The place of business in our society*

By Sir John Methven

Director-General, Confederation of British Industry, 21 Tothill Street, London SW1H 9LP.

I am deeply honoured to have been asked to deliver this lecture as part of the 60th Anniversary Celebrations of your most distinguished Association. I have to confess that until recently I had not given much thought to the matter of surface coatings and had not appreciated the scale and importance of your industries—even though my wife is herself in the business. Nor had I appreciated the complexity of the technology involved. However, I had to go no further than the Contents page of the April issue of your Association's *Journal* to receive a sharp corrective. I was immediately struck by the title of the first article: "Exciplex interactions in photoinitiation of polymerisation by fluorenone-amine systems". Reading that—the title, I mean—I did not dare attempt the article itself—I immediately felt suitably humbled. And I hope you will not expect a scholarship and erudition to match it from me tonight.

Ladies and Gentlemen, your President was kind enough to allow me to choose the subject of the lecture way back in July last year. I have to say that if I were choosing now I should find it easier to wax fluent and with great spontaneity on "The Role of Mr Denis Healey in the Repression of British Business", to some extent reversed in two votes in the House of Commons recently.

Instead, my subject is The Place of Business in our Society, and I suppose there are those who would make of it a matter of high philosophy. But, Mr President, I see it in very practical terms and that is how I shall treat it. What I shall be talking about is, in a very real sense, an important aspect of my own job as Director-General of the Confederation of British Industry, because it is our task to manage a major part of the interface between business and the rest of society.

The first point I should like to make is fundamental to all my thinking on this subject, and that is that the wording of the title of this lecture was very carefully chosen. You will note that it is The Place of Business *in* our Society. *In* our society, I emphasise. Not business *and* society. And I say *in* society because I believe business is not something separate from society, but is rather an integral part of it. Going out and earning a living for the family and for the tribe has been, from the earliest times, an essential function of even the most primitive societies and, though we may now go out and win orders instead of spearing our dinner, there is no fundamental change in the social significance of what we do. Business is not just a minor part of society. It is rather something without which there would be *no* society. Without business we should quite simply starve to death.

Now that might seem like the most banal truism, but it is nevertheless something that is not recognised by enough people in the United Kingdom. Far too many people in our country, some of them very influential, almost seem to regard business as the enemy of society, though happily such people are decreasing in number now. It is an attitude which, if allowed to persist, could do very serious damage to our competitive position in the world and, therefore, to our society's prosperity. It is an attitude which, quite wrongly, seems to be based on the unfortunate equating of business with undeserved riches.

Tonight, I should like to show that far from profit being a dirty word, the making of profits—and good profits at that—is both the most important responsibility of business to society, and the best measure of the extent to which it is performing that duty.

In talking about profit, I should stress that I am no philosopher, nor yet an economist or theoretician. I am a businessman who has spent his working life in the front line of business—most of the time trying to make a profit. I do know what is involved in making market forecasts, working out costs and setting prices that it is thought customers will be prepared to pay. And I have not done it by drawing curves on charts—one for demand and the other for supply and then using complex mathematics to work out the optimum points on the curves. My experience has been out there in the marketplace and on the shopfloor and the result of getting things right was not an elegant theoretical solution, but rather the earning of real money. And I know that in the tough commercial world of today the profit earned has depended on how well I and my colleagues have done our jobs.

I know from this personal experience that producing a profit does, in the vast majority of cases, depend on the allocation of resources—perhaps not always absolutely to the best use, but at least always to the best use imperfect human beings are capable of devising at any given time. And I know that profit *does* provide an incentive, and I know that it is a very clear and effective measure of success.

After all, the opposite of profit is loss, and if a company consistently makes a loss, it must be because it is performing, albeit unwittingly, an anti-social act of some kind. It is either producing goods that society does not want or is devoting more resources to making the product than society judges to be worthwhile. And if the losses a company makes go on to the point at which it collapses, then society suffers severely in a variety of ways. Shareholders lose their investment, suppliers lose business, employees lose their jobs and customers lose their source of supply—which can be particularly serious if, for instance, they can no longer get spares for machines they have bought. And society suffers in another important way too. It loses the money that flows into the economy from a successful company, and in particular it loses the taxation that comes from a profitable company. So it isn't profits that are anti-social, *it is consistently making losses that is anti-social.*

That is the point perhaps most commonly overlooked by the antagonists of business. They fail to appreciate that profitable business in the only source of wealth, and that without it there can be no health service, no education, no culture, no quality of life in our society. There are, of course, rogues in business

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*Commemorative Foundation Lecture presented at the 60th Anniversary Celebrations of the Association, at the Painters' Hall, London EC4 on 11 May 1978.

as in any other walk of society — and it did not need my three years at the Office of Fair Trading to teach me that. But what cannot be done is to pass a law or recruit the necessary law enforcement officers to police every possible anti-social act. The consequences of that would be a bureaucratic nightmare that would ensure a ghastly waste of human resources and would cripple reputable and disreputable business alike. Any attempt to dictate to society what it should have, ignoring market forces, results in the sort of deprived, unfree society they have behind the Iron Curtain.

This is in no way a plea for absolute laissez-faire. Society clearly does need the protection of a substantial framework of law controlling business operations. And indeed the reputable businessman needs it too, not only to protect him from the operations of less scrupulous competitors, but also to help him to act in accordance with his own conscience. For example, a businessman may be very conscious of the fact that he is polluting the environment with an effluent from his factory, and he may be keen to do something about it. Unfortunately, the necessary measures to clean up the effluent may be expensive and could raise his overall costs to the point where he cannot compete with other manufacturers who do not have the same conscience. In a case like that, it helps the businessman with a conscience if the law compels everyone to do the same.

But in so far as it is necessary to control the activities of business in the interests of protecting society, there is no doubt that self-control by business through the means of voluntary codes of practice is preferable. I was a strong advocate of this when I was at the Office of Fair Trading, and I am convinced that the sort of codes now operated by the Association of British Travel Agents, The Advertising Standards Authority and organisations like the shoe manufacturers and retailers are much to be preferred. Codes of practice have many advantages over laws. They can be enforced in the spirit as well as in the letter, they can be rapidly changed and they can be operated very cheaply. But most importantly they tend not to be inflexible and inhibiting and so do not unnecessarily handicap the reputable company. And, finally, in a business environment of excessive bureaucracy and legislation, they do not add unnecessarily to the already onerous burden imposed on businesses.

I have already said that one of the prerequisites of maximising profits is the proper use of human as well as material resources. Some misguided companies undoubtedly still do not handle their employee relations as they should, though far less frequently than unions and left-wingers claim. But I would argue that for any company that wants to stay in business, exploitation of any kind does not maximise profits. In today's society, whether or not workers are protected by unions, it pays employers to act in a socially responsible manner, because only in that way will they get the best out of their workforce. I am absolutely certain that the vast majority of British employers fully accept that they have a responsibility in this direction — and not just for reasons of enlightened self-interest. Indeed, there were times during the CBI's National Conference last November when some of the speakers would not have been out of place on a TUC platform, so eloquently did they plead their workers' cause.

What I would say, however, is that not all employers have fully grasped the scale of the effort they have to put into their relations with their employees, or the scale of the benefits that can result from such an effort. And one of the points the CBI is stressing very strongly to its member companies is the vital importance of good communications with employees and the advantages of effective participation.

What we need and are working for is effective employee involvement and participation, starting from the bottom of an organisation upwards — giving a workforce a say on the things that they really want a say about. At the same time we must continue to fight off theoretical and totally impractical solutions to the so-called "industrial democracy" problem — such as that recommended by the majority of the Bullock Committee. We must all on the one hand encourage practical employee involvement, and on the other recognise and successfully resist a further bid for trade union power, which is already too great in the UK.

Another segment of society to which business owes a responsibility is its customers, and I am very encouraged that so many United Kingdom companies are now more conscious of this than they were ten years or so ago. The rise of "consumerism" has had something to do with this, but even before the "consumerist" movement started, the marketing concept was being better grasped by trade and industry. I firmly believe, on the basis of my experience, particularly at the Office of Fair Trading, that good marketing and consumerism have a lot in common. Quite simply, giving the customer a good deal is good business, as has been proved over and over again by companies like Marks & Spencer. Nevertheless, I think many companies could go a good deal further in this direction. For example, no company in this country has, to my knowledge, tried what has proved so successful for some American retailers, and that is actually involving customers in management decision-making about products and services. To put it quite simply, the UK must live by its exports: and no-one in this hard world of ours in any way feels that they now owe the UK a living.

Industry does, of course, have a responsibility to the community that lives in and around its factories and offices. And, here again, respect for the local environment and good community relations are not only desirable in themselves but are also good business. If a company wants to avoid difficulties in recruitment or difficulties in getting planning permission then it must pay it to cultivate good local relations.

It goes almost without saying, of course, that business has a responsibility to provide an adequate return to the people who invest money in it. For one thing, if it does not, sources of finance will simply not be there when they are needed. But that very important fact apart, it is time some of the detractors of business realised that the dividends and interest businesses pay out do not simply flow into the bulging coffers of some almost mythical race of idle rich. For the most part, they are going into pension funds and into private savings, or are performing the socially vital role of providing an income for countless people who have saved and invested for their old age, and who as a result are less dependent on state funds. But we do not have to apologise even for the dividends that go to people who *are* well off, because it is from such people that the investment funds flow back into business. This is a particularly important source of investment for small businesses which have suffered most in recent years from the shortage of investment funds — especially those who want to start up new small businesses, which we badly need in the UK. And it is the small business sector that could, with the right stimulus, provide many of the jobs we need to cut down on the current alarming level of unemployment. Taxing investment income at the penal rate of 98 per cent is hardly likely to help matters.

Business has a further channel of responsibility to society. It is business that provides so much of our employment in society; and indeed it is quite clear from all the recent market research that has been done, that the public hold trade and industry primarily responsible for providing employment — particularly amongst young people.

Now, if we in business are to discharge that difficult responsibility, we must ensure two things. First, we must ensure that Government creates the conditions in which business operating in a market economy can be both efficient and profitable — the climate for business must be both stable and benign: and I seem to spend most of my time trying to achieve that. And, secondly, we must ensure that the links between the education world and that of business are much closer and happier than they have been in the past. If business is to be successful, then we have to draw into trade and industry the brightest of our young people, boys and girls — instead of their going off to the academic world and central and local Government, as they have in the past. And we have to make sure that they *enjoy* their jobs in trade and industry as I have always done.

This means giving support to schemes like "Understanding British Industry", a project to provide teaching materials about industry for the 13-16 year-olds, but more particularly by individual businessmen and firms interesting themselves on a local basis in individual schools, colleges and polytechnics and coming half-way to meet those who are teaching in those places. We have to change our society so that it becomes as honourable a thing to work in business in the UK, as it has always been in countries as diverse as Germany on the one hand and Japan on the other.

So far, I have talked exclusively about the responsibility of business to the rest of society, but what is not often mentioned is the responsibility of the rest of society to business. And here I think that business currently has quite a good deal to complain about. The relations of society with business are conducted for the most part through the Government and through the trades unions, and I do not feel it is any exaggeration to say that by their actions in recent years they have placed UK industry under severe handicap in its efforts to compete internationally. We are over-taxed, over-governed, shackled by bureaucracy and we suffer from a serious imbalance of power in relation to the trade unions, so much so that it is being made increasingly difficult for us to discharge our responsibilities to society. In particular, making an adequate level of profit which, as I have said, is our prime responsibility, has become well-nigh impossible with the profits of trade and industry in the UK now being only approximately one-third of what they were in the 1960's.

What the Government and the unions have to realise is that they are in danger of sabotaging the only source of the nation's wealth, for example, both the Chancellor and the Prime Minister have said that it is important to provide the short of stimulus that only lower taxation can provide, but any hopes we might have had that they had at least seen economic sense were cruelly dashed by the Budget. Far from improving the lot of managers and skilled workers, it would have resulted in a severe worsening of their financial position. Something has been done to put this right by the two Government defeats in the Finance Bill on Monday and Wednesday.

There are two thoughts that run through all I have been saying tonight. The first is that business is not something separate from society; it is an integral and important part of it. It is indeed the only source of society's wealth.

The second thought is that the interests of good business and society are, and should be seen as, identical. Good management and good business are good for society. And good profits from industry are vital to a healthy society and a good life-style. Business seeks no conflict with society, with Government or with the unions. The aim of the CBI, for example, is to promote the interests of *everyone* who works in trade and industry, and not just those of the employers. We are as anxious as anyone else to improve the lot of all our people, and we will do it too — if only the Government will permit help to create the conditions in which it is once again worthwhile to work hard, to earn promotion, to take risks and to invest. All we ask is that Government should stop biting the hand that feeds it, and killing the goose that lays the golden eggs — and we, all of us in business, must go on, vigorously, and on all occasions, persuading them.

[*Received 11 May 1978*

Next month's issue

The Honorary Editor has accepted the following papers for publication, and they are expected to appear in the August issue of the *Journal:*

Effect of temperature on water absorption and permeation properties of coatings *by M. Yaseen and W. Funke*

Experimental aspects of adhesion testing *by J. Sickfeld*

Recent progress in the physical chemistry of organic pigments with special reference to phthalocyanines *by R. Sappok*

Hygric criteria for exterior paints for the protection of porous building materials *by D. Y. Perera*

Reviews

Anstrichschäden

By H. Klopfer

Bauverlag GmbH Wiesbaden & Berlin
Pp. 232. Price: DM 85.00.

This book is devoted to a description of the breakdown of paint films under various conditions.

Chapter 1 characterises coatings as polymer films and describes briefly the basic materials used in the manufacture of coatings and the process of film formation.

Chapter 2 deals in some 70 pages with the influences known to cause breakdown. These are grouped into six subsections: weathering, exposure to chemicals, ageing, internal stresses, permeability to water and gases, the effect of pores or "holidays" in the film. Some of these are treated in some considerable scientific detail and the author's engineering bias is clearly discernible. The reviewer does not share the view expressed in the foreword that the book will be equally acceptable to experts in the paint industry, paint users in trade and industry, and builders and architects. It should also be noted, that the fundamental importance of the substrate in relation to the breakdown of coatings is often ignored and not accorded the importance it deserves.

Chapter 3 classifies the breakdowns encountered into seven groups: colour and gloss changes, growth of organisms and other deposits of all kinds, swelling, weathering and erosion, blisters of all types, surface irregularities, cracking, peeling and flaking.

Chapter 4 provides 95 excellent colour photographs illustrating the breakdowns group by group. Taken together with 59 black and white illustrations and 28 electron micrographs spread throughout the text, these form a collection of first class pictures which should appeal to a large circle of readers. However, the price of about £22 might be an obstacle to wider distribution.

H. R. HAMBURG

Acrylic powder coatings

R. H. Chandler Ltd

November, 1977. Pp 11 + 59.

This is No 30 in the valuable series of bibliographies in paint technology. In the customary style it gives a succint account of the state of the art followed by a review of the recent literature and abstracts of relevant patents. It confirms that acrylic powders are the "Cinderella" of the powder coatings field (at least in Europe). However, there are hopes that if there is a breakthrough by powders as the finish for cars, the acrylic with their success in the solvent-borne field, will be the type selected.

The literature survey is considered in terms of the structure of the acrylic polymer, curing mechanisms and film properties of acrylic powder coatings. Most of the crosslinking mechanisms used for solvent paints have been examined for powders, but there are two interesting new types. These are the bisoxazolinobenzene systems for acidic acrylic resins and self-crosslinking acrylic polymers, into which aminimide groups have been introduced. Several of the reports on film properties involve comparison with other systems. There are 61 literature references and 193 patent abstracts, with name and number indexes.

L. A. O'NEILL

Section Proceedings

London

Metal decorating

A one day symposium entitled "Metal decorating in the paint and printing industries" was held by the London Section, in association with Thames Polytechnic at Thames Polytechnic, Wellington Street, SE18 on Wednesday 8 March 1978.

The morning session, chaired by Mr H. Worsdall, opened with a lecture by Mr D. McFarlane of the British Steel Corporation. Talking about the "Surface characteristics of tin mill products", Mr McFarlane first described the various mill manufacturing processes which produced either blackplate, tin-free steel, hot dip and electrolytic tinplates. In more detail, he then described the composition, advantages, disadvantages and end uses of each of these substrates in the metal decorating industry.

Mr W. Campbell of Ault and Wiberg Ltd presented the next lecture titled "Lacquering trends". Confining his lecture to the internal lacquers for can bodies and ends, Mr Campbell described the properties of the lacquers currently used for these purposes and showed how their properties were used to meet specific resistance requirements, such as sulphur staining resistance, non-tainting of beer etc. The impact of the newer can technologies such as "drawn and wallironed" and "draw, redraw cans" on lacquer requirements were discussed.

In conclusion, Mr Campbell outlined the likely use of the alternative technologies in organic solvent borne lacquers in the metal decorating industry.

Following lunch, a film entitled "Harvest for tomorrow" was shown, by courtesy of the International Tin Research Institute. The film demonstrated how tinplate containers could be used to provide food products to the housewife out of season.

The first lecture of the afternoon session, chaired by Mr A. Newbould, was given by Mr B. Hitchens, Coates Bros. Ltd on "The trends in exterior coating technology".

In describing the constraints on the industry, Mr Hitchens mentioned substrate, can technology, energy, pollution, conservation and health and safety aspects. He then went on to discuss in detail the various parameters, current and possible, and the effect they were likely to have on the future developments by coatings suppliers.

The trend towards reducing the number and thickness of coatings would continue, as would the need to reduce stoving times and temperatures. All these requirements would be met by the development of resin systems with the necessary properties.

Inks would become increasingly more of the non-varnish type and UV cure would be more widely used.

The use of alternative technologies to solvent borne coatings were discussed. The use of UV cured finishes were covered by another lecturer; it was considered that water borne coatings would find uses in aluminium closures, overvarnishes for tinplate and in 'DWI' cans more easily than in other areas, because the general poor wetting properties of water on metal and poor flow of emulsion resins would limit their wider usage.

High solids coatings (>80%) would give better plate wetting flow and better economics than water borne coatings. To achieve the high solids required, low molecular weight, low flexibility resins only could be used. Their use would be limited to areas where high degrees of flexibility would not be necessary.

The final lecture of the afternoon was presented by Mr E. A. Pachebat of International Paint-Industrial Coatings who described "Some practical aspects of the UV curing of coatings".

UV cured inks were first introduced in the UK in 1970 and in Europe in 1971. UV cured coatings for tin-free steel ends, clear varnishes and white enamels for three and two piece bodies were currently available.

The advantages and disadvantages of UV curing were detailed. Mr Pachebat then discussed a number of typical formulations for UV cured metal decorating coatings. The composition of acrylic and cationic cured epoxies were described and their advantages and disadvantages discussed.

The toxicity aspects of the acrylic monomers that are suitable for metal decorating end uses were mentioned. The cost of carrying out full TOSCA tests on a single material, as required by the USA, was estimated to cost $1 million and take two years.

Mr Pachebat also described the practical difficulties associated with the application of very high solids (>90%) coatings on roller coaters. Hydraulic pressures forced rollers apart and it was not possible to apply the low film weights required. Either the roller coaters had to be modified with extra rollers or differential drives to the rollers or off-set gravure was used.

White pigmented coatings exhibited poor flow unless sufficient time was allowed for flow out. In addition, the absorption by the pigment of the UV wavelengths made cure difficult. Catalysts absorbing in the visible were required.

The final session of the afternoon was a wide ranging general discussion between all the lecturers and audience.

Finally, a vote of thanks to all lecturers and chairmen was proposed by Mr J. Tooke-Kirkby and seconded by all present.

A.J.N.

The influence of solar radiation on paint films

A one day symposium entitled "The influence of solar radiation on paint films" was held at Thames Polytechnic, Woolwich in association with Thames Polytechnic. The London Section Chairman Mr D. Bayliss opened the proceedings with a brief word of welcome and introduced the chairman for the morning session, Mr G. M. Scott.

Dr J. Horder, Thames Polytechnic presented the first paper on "Photochemical degradation of polymeric films". Degradation was caused by the presence of traces of unsaturation in the polymeric film, which absorbed UV. Photochemical degradation of ketones by the Norrish reactions was responsible for cleavage of the polymer chain. Exposure of polyethylene had shown a gradual increase in the hydroxyl, carbonyl and vinyl groups present. Carbonyl groups excited to the triplet state by UV could transfer their energy to molecular oxygen forming singlet oxygen which could then react with vinyl groups to give hydroperoxides which acted as photosensitisers. Film thickness and crystal state were also important factors. The effect of various UV stabilisers was illustrated. "The use of polymer films to monitor UV radiation" was the title of the second paper prepared by Dr A. Davis and Mr D. Gordon, PERME Ministry of Defence and in the absence of the authors, read by Dr K. Holder of MQAD.

A method for monitoring the ultraviolet radiation content of solar radiation at exposure sites was described. This involved measurement of the degradation, by the changes in optical density, of two types of thin polymer film, polyphenylene oxide and polysulphone. A world wide programme for evaluating the UV environment at a number of sites was outlined and some of the results discussed. Information was obtained, using this relatively simple technique, on the relative importance of diffuse and direct solar radiation, the effect of angle of exposure on UV levels, and the relationship between UV levels and total solar radiation.

Mr Oakley of Tioxide International presented the third paper on "The influence of pigmentation on polymer degradation". When a pigment was incorporated, other reactions could take place which could be more significant than photodegradation. The effect of UV on titanium dioxide in the presence of water was to produce hydroxyl radicals which actively degraded the polymer. Metal oxides applied to the outer crystal structure showed a decrease in mass loss and decrease in degradation rate. Oxides of silicon, zinc and zirconium had high surface area and absorbed hydroxyl radicals. The effects of pigment volume concentration, film thickness and state of dispersion were illustrated.

Dr R. C. Denny took the chair for the afternoon session.

Mr J. Tooke-Kirby presented a selection of slides with commentary on "Florida exposure" provided by a commercial testing station in Miami, Florida. Illustrations were shown of the facilities available and of the breakdown effects on exposure panels. Mr J. L. Prosser of the Paint Research Association spoke on the "Testing of high durability coatings". Predictions of long-life eg 20-30 years were based on the extrapolation from weight loss data of paint films. Weight loss experiments using micro balances showed a steady degradation. Different grades of titanium dioxide gave different results for rate of weight loss.

The final paper on the "Simulation of the effect of solar radiation on paint films by artificial sources – modern trends" was presented by Mr M. Hales, of MQAD.

Commercially available apparatus of different types all had drawbacks due to an excess of UV light below 300nm and did not give close enough correlation with natural weathering. The xenon arc, although rather high in IR which had to be filtered out, gave fairly close correlation. Loss of gloss on exposure showed little correlation for a series of paints, for some of the paints, in some of the test methods. Coloured paints gave good correlation in some cases.

B.F.G.

Information Received

Extended capability

Pyrene Chemical Services Ltd, a member of the Brent Chemicals International Group, has completely refurbished its Gold Seal test panel manufacturing facility and equipped it with new strip decoiling and panel finishing machinery. Gold Seal metal panels provide a reproducible, high quality surface finish to British Standards specifications, as a base for testing the comparative adhesion, corrosion resistance and anti-chipping characteristics of organic finishes, usually when applied over specific pretreatments.

New warehouse development

A new £0.5m warehouse, built by Synthetic Resins Ltd, has been opened at their Speke production centre, and all resin stocks will now be held in the covered storage areas. The new warehouse is planned to cope with the anticipated requirements for the next ten years, and increases the capacity for storage to 4000 pallet loads.

Far-East agency network

Tintometer Ltd has established its first comprehensive network of agents in the Far East to handle the sales and marketing of its range of scientific, chemical analysis and colour measuring equipment.

The move follows increasingly heavy demand for the UK company's products and particularly for those pertaining to domestic and industrial water supplies and factory effluents.

Swale joins BCI Group

Swale Chemicals has become a member of the expanding Brent Chemicals International Group. Swale are one of Europe's largest suppliers of specialised coatings and inks to the converting industry, and with the increased resources of the BCI Group, will be able to extend their operations into new markets.

New technical centre

Plastic Coating Systems of Surrey have recently opened a new laboratory which includes testing facilities and offices under one roof. The company specialises in the development of surface coating materials, methods and machinery, and the new facilities will ensure that their position as a leader in plastics technology is maintained.

U.K. agency

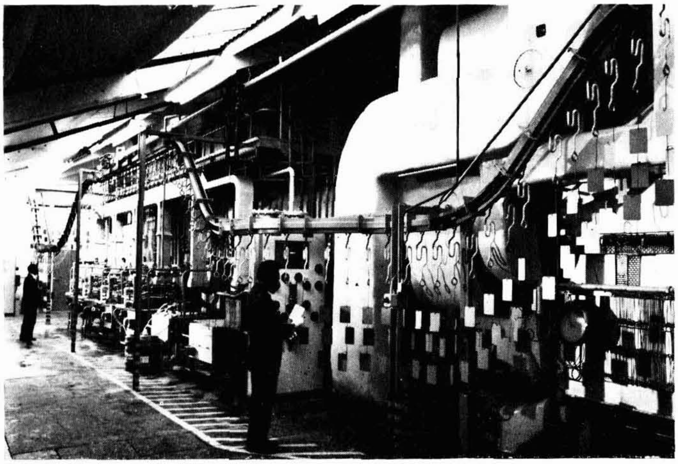
Packaging Aids Ltd have been appointed sole U.K. agents and stockists of the range of custom built packing tables for the paint and allied manufacturing industries from Ekdahls of Sweden.

The Ekdahls benches are equipped with a variety of facilities for dispensing rolls of paper, plastic cell or foam of various widths, as well as cutters and trimmers.

New products

Industrial silicone sealants

The Industrial Division of Loctite (UK) Ltd has announced the introduction of a new range of RTV products specifically designed for industrial use under the name of Loctite Superflex Silicone Sealants. They can be used to make instant-seal joints and gaskets of any shape or size, and are applied direct from the container to the surface, where they immediately start to solidify into tough, permanently flexible rubber-like joints.



A view of the Gold Seal test panel manufacturing facility of Pyrene Chemical Services Ltd, which has just been completely refurbished.

New atomic absorption spectrophotometer

Perkin-Elmer has announced the availability of the new Model 560, an improved version of the Model 460 microprocessor-based double beam atomic absorption spectrophotometer, which includes a new computing ability that makes it possible to do complex calculations and implement sophisticated logic programmes quickly and accurately. Averaging, standard deviation and coefficients of variation and reslope are built in, and the Model 560 also features a new dual-blazed grating which permits optimum photometric response throughout the wavelength range with a single grating.

Mini-metering system

Foscolor Ltd, the specialist pigment dispersion company of the Foseco Minsep Group, have recently launched a new complete mini-metering system for the accurate dosing of pigments, intended for use with Foscolor's liquid Masterbatch. Named the Mastermeter, the mechanically operated system dispenses pigments or additives with an accuracy of 0.05 per cent. The machine has also been designed so that a complete clean-down can be done in a matter of minutes.

Effluent tester

Chemlab Instruments Ltd, agents for Wissenschaftlich Technische Werstatten who produce a wide range of instruments for the measurement of dissolved oxygen and water pollution, has announced the availability of a new combined instrument designed for the checking of water effluents. Known as the Combibox, it will measure and continuously monitor pH, conductivity and temperature, and is supplied housed in a durable box with a well guarded electrode system with long leads designed for immersion in rivers or effluent streams.

New acrylic emulsions

Two new products have recently been added to the Ionac Chemical Company range, which are marketed in the U.K. by British Chemical Products & Colours Ltd. Both are thermoplastic acrylic emulsions exhibiting excellent compatibility with many commonly used aqueous ink and coating vehicles, including acrylic solution polymers, alkali soluble and maleic resins, styrene-shellac, styrene-acrylic and polyethylene emulsions. Designated Polymer X224 and Polymer X225, they are recommended for use in aqueous flexographic overprint varnish systems and in aqueous blister pack overprint varnish applications, respectively. Both products give improved film forming properties, high gloss and good dry and wet rub resistance.

Process pumps

Corrosion and abrasion resistant process pumps of the horizontal centrifugal type are now available in the Fybroc range marketed in the U.K. by Alpha Technical Services Ltd. Constructed in glassfibre-reinforced vinyl ester or epoxy resin, they are ideally suited to handling highly corrosive liquids and slurries. Flow rates of 950 m^3 per hour and heads of up to 95 metres are obtainable, with operating temperatures up to 99°C in most cases.

New dyes for nylon

The Atlantic Chemical Corporation has announced the manufacture of two new dyes for nylon, Atanyl Red 6B and Atanyl Royal Blue B, which were developed as general purpose colours for the dyer where economy and build-up are important. The new dyes are also suitable for printing.

Electro-hydraulic airless spray unit

Highly viscous heavy protective and fibre-loaded finishes which cannot be atomised by most spraying equipment can be sprayed by a powerful new unit recently launched by Gray-Campling Ltd. The Wagner Airless Electro-Hydraulic Unit 6500 EH has a powerful diaphragm pump incorporating a hydraulic piston producing 3675 psi atomisation pressure and enabling larger orifice spray tips to be fitted.

As the 6500 EH is powered by an electric motor it is particularly suitable for applications where no compressed air is available.

Digital pH/temperature meter

Channel Electronics (Sussex) Ltd has announced the introduction of the new 6000 series digital pH/temperature meters to complement the existing range of pH measuring instruments. The battery operated meters are capable of up to 35 hours continuous use or 60 hours intermittent use.

New fluorescence spectrometers

Perkin-Elmer Ltd have announced two new additions to their fluorescence product line, the Model 2000 fluorescence spectrophotometer and the Model 1000M filter fluorimeter. Both instruments use a stroboscopic xenon source to provide a continuum of energy over the range 260-650 nm. This produces very little ozone and also eliminates the need to change sources when a variety of analyses are being performed. Discrete excitation wavelengths are chosen by means of narrow band-pass interference filters.

Paliotol Yellow 2140 HD

Paliotol Yellow 2140 HD is the first member of a new range of pigments placed on the market by BASF Aktiengesellschaft. The letters HD stand for high opacity and easily dispersible. Paliotol Yellow 2140 HD, an isoindoline pigment, has higher opacity than all organic pigments, and all inorganic pigments with a similar shade.

TR1 flowmeter totaliser

The TR1 flowmeter totaliser is the first of a new range of electronics in DIN sized cases from AOT Flowmeters Ltd. The instrument displays flowrate and flow total from signals generated by turbine type flowmeters

Anti-corrosive pigments

A range of composite pigments for both water and oleoresinous based paints, containing none of the heavy metals and exhibiting maximum corrosion resistance when used in primer coatings is available from R. S. Stokvis & Sons Ltd.

The reference CW22 series of pigments are a calcium boro silicate composite, whilst CW11 and CW111 are a calcium phospho silicate composites. Both of these compositions have a low specific gravity and a high bulking factor, thereby offering distinct advantages in cost performance.

New metering feeder

A new screw feeder which can meter dry, semi-dry, powdered and granular materials to an accuracy of one per cent has been introduced by Simon-Solitec Ltd. Named the Mark II Volumetric Metering Feeder, the machine provides either intermittent or continuous output and has capacities from 1.2 to 800 litres per hour. The consistently high level of accuracy is due to an improved vibratory action which conditions the material, ensuring a homogeneous density and a steady smooth feed to the screw.

Diesel powered airless paint sprayer

A diesel powered airless paint spraying unit, designed for areas where neither electricity nor compressed air are available, has been introduced in the U.K. by Gray-Campling Ltd. Named the Wagner 7000H Diesel, it is fitted with a 4HP engine and trolley mounted for mobility.

Fast filler for foaming fluids

Neumo Ltd have available a new automatic machine capable of fast, clean, accurate filling of various sized containers with foaming and non-foaming fluids. The machine is a compact, single track continuous running unit capable of accepting up to four filling heads each fed by a five litre capacity quick-strip positive displacement pump actuated by a reciprocating air motor.

Paint and pretreatment directory

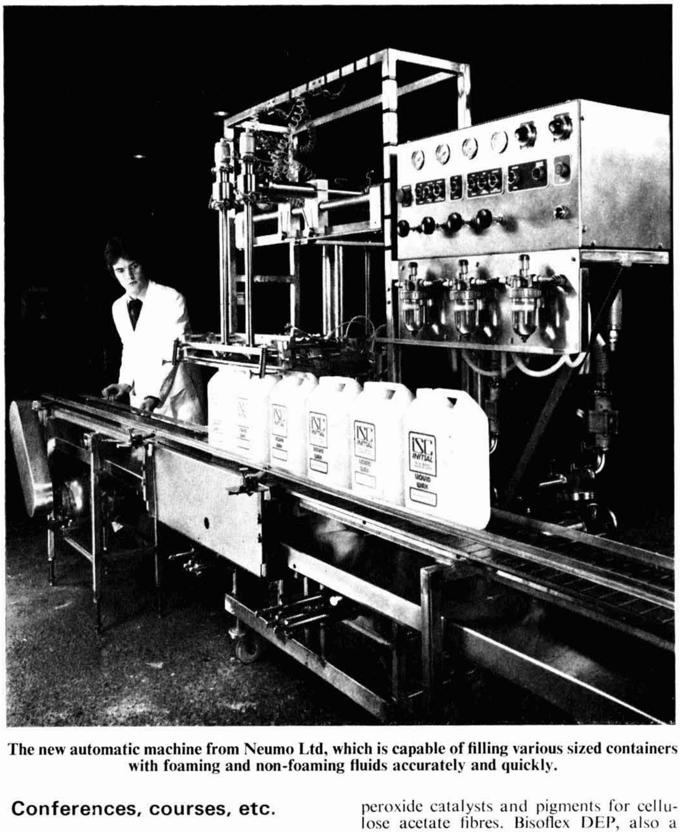
The Paint Research Association has published a Directory of paint and pretreatment products, which contains a comprehensive list of 4200 products classified by function under 84 headings related to intended use.

North Sea corrosion

A conference on 'North Sea corrosion—what have we learnt?' is being organised by the Institution of Corrosion Science and Technology and the Institute of Marine Engineers to be held in Edinburgh from 23-25 October 1978.

Bisoflex plasticiser Technigrams

BP Chemicals has issued two Technigrams giving information on Bisoflex DMP (dimethyl phthalate) and Bisoflex DEP (diethyl phthalate) plasticisers. Bisoflex DMP is a solvent plasticiser for cellulose acetate and a vehicle for dispersions of peroxide catalysts and pigments for cellulose acetate fibres. Bisoflex DEP, also a cellulose acetate plasticiser, finds further applications in the cosmetics industry and as a denaturant for alcohol.



The new automatic machine from Neumo Ltd, which is capable of filling various sized containers with foaming and non-foaming fluids accurately and quickly.

Conferences, courses, etc.

Colour blacks

The Pigments Division of Degussa has published a booklet on colour blacks which describes the company's 16 channel and 17 furnace blacks and the lamp black No. 101 The physical and chemical data is presented in tabular form, and the German/English publication also contains a summary of the fields of application for the products.

Radiation curing conference

The Association for Finishing Industries of SME is organising a conference on radiation curing to be held from 26-28 September in Illinois.

THE INTERNATIONAL FOCAL POINT FOR THE SURFACE COATINGS INDUSTRIES • 3-6 APRIL 1979 LONDON •
OCCA 31

OCCA-31 Exhibition

Alexandra Palace, London, 3-6 April 1979

The international focal point
for the
surface coatings industries

Arrangements for OCCA-31

The Exhibition Committee announces that the thirty-first annual exhibition of raw materials, plant and equipment for the paint, printing ink, colour and allied industries organised by the Association will be held at Alexandra Palace, London N22 from 3-6 April 1979.

Whilst the Committee naturally encourages the showing of new products, it does not stipulate that a new product has to be shown each year, since it fully appreciates that this is not possible. Accordingly, the Committee draws attention to the fact that new technical data on existing products is regarded as acceptable subject matter.

Motif for the Exhibition

The motif, designed by Robert Hamblin, emphasises the unique aspect of the OCCA Exhibitions, which annually provide an international focal point for the surface coatings industries. In 1978, exhibitors were drawn from 16 countries and visitors from over 50 countries. The colours of the motif shown on the advertisement on the back outside cover of this issue will be carried throughout the publicity leading up to the Exhibition. The two main colours of the motif, green and yellow, will be incorporated on the facias of the stands and it is intended to organise complementary floral displays in the Exhibition Hall. The use of these colours will create a pleasing contrast as visitors move from corridor to corridor at the Exhibition, as the green colour will be used from north to south and the yellow colour from east to west.

Invitation to Exhibit

Copies of the Invitation to Exhibit have been despatched to companies and organisations in the UK and overseas which have shown at previous OCCA Exhibitions or have requested information for the first time for the 1979 Exhibition. Completed application forms for stand space must be returned to the Director & Secretary of the Association **not later than 31 October 1978.**

The interest shown in the OCCA-31 Exhibition as a direct result of the highly successful OCCA-30 Exhibition held in April this year has been considerable, and already companies have asked for larger stands than they have had in previous years. Additionally, several companies who have not previously shown at OCCA Exhibitions as well as some who were not able to be present at OCCA-30 have contacted the Association requesting that sites be reserved for them.

The Exhibition Committee was particularly pleased to see the large number of exhibits at OCCA-30 from companies new to the Exhibition, both from the UK and many overseas countries, and this emphasises the continuing importance of this annual Exhibition to the world surface coatings industry and demonstrates its international character.

Visitors to the OCCA-30 Exhibition came from over 50 countries. A view of one of the crowded corridors at the Exhibition is shown above, and below is a view of the Information Centre and one of the seating areas in the foregrouud.

Visitors to OCCA-30 are known to have come from over 50 countries and admissions by season tickets of over 10000 were recorded at the turnstiles at the entrance to the Exhibition. Several Sections of the Association organised parties to the Exhibition, and it is hoped that these will also be arranged for OCCA-31.

Any organisation wishing to receive **details of OCCA-31 should write to the Director & Secretary of the Association at the address on the Contents page to receive a copy of the Invitation to Exhibit.**

Facilities at Alexandra Palace

The Association has full use of the facilities at Alexandra Palace during the period of the Exhibition which include a restaurant, bars, a cafeteria and a private exhibitors' bar. Another facility which is of considerable benefit to those travelling to the Exhibition by car is the ample free car parking space within the Palace grounds.

For visitors travelling to the Exhibition on the underground system, the Association will once again be running a free bus shuttle service to and from the Exhibition, this year from Wood Green Station on the Piccadilly Line. The extension of the Piccadilly Underground Line to the Heathrow Central terminal was opened in late

1977, and this now provides a direct link for travellers arriving at the airport to both central London and to Wood Green Station and the bus shuttle service to the Exhibition.

Information in foreign languages

As in previous years, the Association will be circulating information leaflets in six languages which will contain application forms for those wishing to purchase copies of the *Official Guide* and season admission tickets before the Exhibition. Interpreters will once again be in attendance at the Information Centre at the Exhibition to assist foreign exhibitors and visitors with their queries.

Official Guide

This unique publication contains descriptions of all companies and their exhibits at the Exhibition and is issued to each member of the Association at home and abroad as well as to all visitors to the Exhibition with their admission tickets. It is published several weeks in advance of the dates of the Exhibition to allow visitors the maximum opportunity to plan the itinerary of their visits. In 1978, for the first time, the *Official Guide* contained a chart classifying the products on show into the various types, and it is intended to produce a similar chart for the *Official Guide* to OCCA-31. This chart is invaluable to visitors, especially those who have only a limited amount of time available, as it provides a quick, easy reference of all the companies present who are displaying the products in which they are interested.

Advertising facilities

Advertising space is available in the *Official Guide*, both to companies who will be exhibiting at the Exhibition, and also to other organisations. This important publication is constantly referred to both before the Exhibition and afterwards as a convenient record of the latest developments in the industry. Consequently, organisations wishing to take advertising space in the Guide should book their requirements as soon as possible in order to ensure a prominent position. Details of the advertising rates and media data are available from Mr D. M. Sanders at the address on the Contents page.

London Section

Annual General Meeting

The 40th Annual General Meeting of the London Section was held at the Rubens Hotel, Buckingham Palace Road S.W.1 on Thursday, 13 April 1978.

After opening the meeting, Mr D. Bayliss, Chairman, welcomed members. The minutes of the previous AGM were approved. Two scrutineers were appointed to count the voting papers for the election of committee members. The annual report was presented and proposed by the Secretary Mr B. Gilliam, and seconded by Mr D. Eddowes. It was adopted without dissent. Mr D. Bannington presented the financial report which was proposed and seconded by Mr E. Pachebat and Mr K. Bargrove respectively. The report was unanimously approved.

Mr Bayliss, proposed the election of the Honorary Officers and these were accepted unanimously. Mr Bayliss was re-elected chairman for his second, and final, year of office.

The Meeting approved the selection by the Committee of Dr T. Banfield as Vice-Chairman.

Votes of thanks to the retiring elected Committee members, (Mr E. A. Pachebat, Mr K. Wiseman, Mr K. Bargrove), to Mr A. Hipwood, retiring Hon. Programmes Officer and Mr J. Tooke-Kirby, Immediate Past Chairman were proposed and warmly applauded.

The meeting closed with thanks to all who attended. After a break for refreshment a lecture and presentation to members and guests was given by Mr O. N. Collier of Johnson Matthey & Co Ltd on "The decoration of pottery and glass".

Starting with little more than an empty table and a number of packaging cases, Mr Collier proceeded to fill the table with a whole variety of pottery, ceramics and glassware in a range of shapes and sizes. With the help of his wife and an assistant, Mr Collier then entertained the audience with an explanation of the various methods used to decorate pottery—crockery and display—and glassware. Direct printing and transfer printing, gold lacquering and other techniques were explained and examples shown and in some cases demonstrated.

At the end of an enthralling hour the audience had the opportunity to practise some of the techniques described in the lecture.

A vote of thanks to the lecturer was proposed by Mrs K. Hedgecock to which the audience warmly responded.

A.J.N.

New powder coatings laboratory

On Tuesday 4 April, Mr B. Conlan, M.P. for Gateshead East, officially opened the new International Paint Powder Coatings Research and Development Centre at Felling-on-Tyne.

Such has been the growth of International Powder Coatings, that since production first started in 1975, additional lines have been installed to increase capacity from 200 tons to 1000 tons per annum on a single shift basis.

The new laboratory has been designed to ensure the expansion of this business still further.

To keep ahead of industry's demands for improved technology on powder coatings, the laboratory block has been designed by International Paint technical staff, after an in-depth evaluation of likely future trends in powder development. The laboratories include a development laboratory, a customer demonstration laboratory and a specially designed section for the manufacture of powder samples consisting of four separate rooms, each accommodating one stage in the manufacturing process. These are premixing, extruding, milling and sieving.

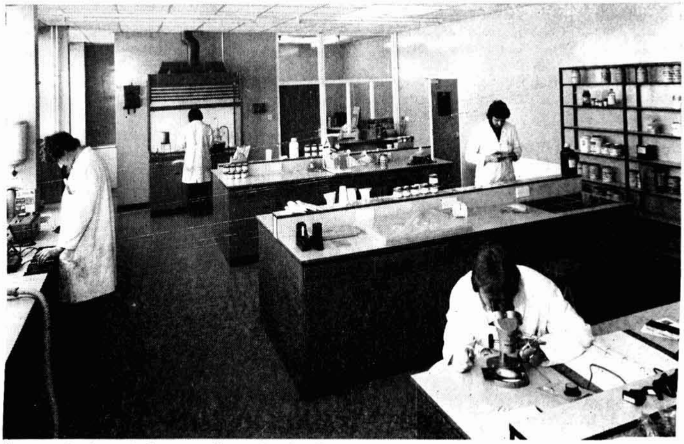
All items of equipment are served by individually designed dust extractors and each room is fed with temperature controlled filtered air. The rooms are tiled from floor to ceiling with central drain points, the electrical fittings are waterproofed and washing down is done with pressure hoses. Facilities are also available for vacuum and compressed air.

This clinical approach highlights the quality standards to which powder coatings are produced.

A full range of testing equipment for powders and applied film was also available.

With an investment of over £750,000 in two and a half years it is obvious that the International Paints and the Powder Coatings Division has faith in the future of this type of coating.

F.H.



A general view of the central powder development laboratory, which illustrates the clinical standards used in the production of powder coatings.

Stratford -on- Avon

OCCA CONFERENCE

STRATFORD HILTON HOTEL • 20–23 JUNE 1979

Shakespeare's birthplace

Photograph by Herald Photographic Studio, Stratford-upon-Avon

CALL FOR PAPERS

The challenge to coatings in a changing world

It is envisaged that the papers to be presented will be concerned not only with the challenge of new materials and the substitution of traditional ones, new methods of construction and the problems of application. but also with the effects of new legislation (both national and international) and the sociological, climatic and leisure aspects of life in the last quarter of the twentieth century.

The Hon. Research and Development Officer now invites offers of papers for presentation at this Conference. Anyone wishing to submit a paper for consideration should notify his intention as soon as possible to: The Director and Secretary, Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, England (Telephone 01-908 1086; telex 922670).

News of Members

Mr E. V. Carter, an Ordinary Member attached to the Midlands Section and a Fellow in the Professional Grade, has been appointed Director of Miox Ltd, distributors of Austrian micaceous iron oxide pigment.

Mr G. J. Dawson, an Ordinary Member attached to the Midlands Section, has been awarded a Master of Science degree for a thesis on the estimation of antimony in biological material.

Mr L. Miller, an Ordinary Member attached to the General Overseas Section and resident in Perth, Australia, has obtained the Certificate in Exporting and the Diploma in Applied Science (Chemistry) from the Education Department of Western Australia.

Dr J. L. Scott, an Ordinary Member attached to the General Overseas Section, has taken up an appointment as Technical Director of South Florida Test Service Inc, in Miami, Florida.

Dr W. Carr, an Ordinary Member attached to the Manchester Section and a Fellow in the Professional Grade, has been awarded the Gold Medal of the Institute of Printing in recognition of his contributions to knowledge in the area of organic pigment technology. Dr Carr has also been the recipient of an Association Commendation Award.

Forthcoming Events

Details are given of Association meetings in the United Kingdom and Ireland up to the end of the month following publication and in other parts of the world up to the end of the second following publication.

Coatings 78 Symposium

The South African Division of the Association is organising a Conference to be held on 2–5 November 1978 in Cape Town with the title 'Coatings 78 – Economy, ecology, performance'. Those wishing to receive further details should contact Mr P. J. Quorn, c/o Inmont SA (Pty) Ltd, Kinghall Avenue, Epping No. 3, 7460 South Africa.

Thames Valley Section

Medieval Evening

To celebrate the 60th Anniversary of the Association, and in response to many requests at previous Buffet Dances, the Thames Valley Section have arranged a Medieval Evening to be held at Great Fosters, Egham, Surrey on 8 September 1978.

On arrival, guests will be greeted and given a glass of mead. A few minutes before dinner, the 'King' will enter followed by his minstrels and retinue. He will then lead the assembled guests into the Tythe Barn where jollities will continue until the evening ends and the 'King' and his troubadours retire.

Informal dress or period costume will be worn, for which prizes will be awarded.

Tickets, at £8.00 each including VAT and further details are available from: M. H. Prigmore, 'Elmwood', Church Road, Penn, Bucks. HP10 8EX.

Manchester Section

Student Seminar

The Manchester Section is to hold a one-day Seminar entitled 'Emulsion paints' on Friday, 15 September at the Woodcourt Hotel, Sale, Cheshire. Four papers will be presented with the following titles:

> 'Acrylics—the route to quality decorative paints' by Mr D. Kerrison of Rohm & Haas Ltd.
>
> 'Titanium dioxide in aqueous finishes' by Mr J. Valpora of Kemira Oy.
>
> 'The colouration of decorative aqueous finishes' by Mr J. M. Hill of Ciba-Geigy Plastics and Additives Company, Pigments Division.
>
> 'The principles of emulsion paint formulation and manufacture' by Mr W. Phillips of Crown Decorative Products Ltd.

Fees for the seminar will be £8.10 for members and £13.50 for non-members (including VAT), and further details may be obtained from Mr G. T. Flood, Pigments Division, Ciba-Geigy Plastics & Additives Company, Roundthorne Estate, Wythenshawe, Manchester M23 9ND.

Register of Members

The following elections to membership have been approved by Council. The Section to which each new Member is attached is given in italics.

Ordinary Members

Almond, Paul Christopher, GradRIC, 8 Dene Crescent, Newcastle-upon-Tyne NE3 1QS. *(Newcastle)*

Beckly, Albert Henry, 14 Frimley Close, Woodingdean, Brighton, Sussex. *(London)*

Borwick, Amory Donal, BA, Mobil Oil SA (Pty) Ltd, PO Box 35, Cape Town 8000, South Africa. *(Cape)*

Buchling, Carl Wilhelm Petrus, PO Box 50, Witbeeck 1729, South Africa. *(Transvaal)*

Clark, Ian Ferguson, 2 Derwent Place, Derwent Avenue, Benoni 1500, South Africa. *(Transvaal)*

Ekeh, Godwin Chukuemeka, 4 Yonge Park, London N4 3NT. *(General Overseas)*

Erlich, George Julius, Erli House, Tillington Place, Petworth, West Sussex GU28 0RA. *(London)*

Fox William Thomas, BTech, Dulux Paint Co. Ltd, PO Box 76, Chungli, Taiwan. *(General Overseas)*

Garrard, George William, 80 Kingsgate Avenue, Kingsgate, Broadstaires, Kent. *(Manchester)*

Gower, Philip Alan, BSc, Research Consultants Ltd, Armagh House, Tobermory, Isle of Mull, Argyll. *(Scottish—Eastern Branch)*

Joshi, Jatashanker Kanji, 11 Wyrley Road, Witton, Birmingham B6 7BS. *(Midlands)*

Lamb, George, AMCST, Blackburn Chemicals Ltd, Whitebirk Industrial Estate, Blackburn, Lancs BB1 5SX. *(Manchester)*

McGhee, Brian, BSc, 43 Oak Avenue, Greenhills, East Kilbride, Glasgow. *(Scottish)*

McQueen, Ian, BSc, 14 Hazel Dene, Bishopbriggs, Glasgow G64 1TZ. *(Scottish)*

San, Seong-Lim, BSc, Dulux Paint Co. Ltd, PO Box 76, Chungli, Taiwan. *(General Overseas)*

Say, Terence Edwin, BSc, CChem MRIC, Bevacoid SA (Pty. Ltd, PO Box 65, New Germany, Natal 3620, South Africa) *(Natal)*

Stephenson, Joseph Richard, 27 High Beech Lane, Chepstow NP6 5BQ, Gwent. *(Bristol)*

Szyndler, Henry Roman John, BSc, 3 Peters Drive, Leicester LE5 2AQ. *(Manchester)*

Visioli, Flavio, V A Falcone, Parco Lamaro 12, Napoli 1, Italy. *(General Overseas)*

Associate Members

Duffy, Patrick, 12 Grangewood, Rochestown Avenue, Dunlaoghaire, Co. Dublin. *(Irish)*

Lacey, Daniel Noel, 36 Monastery Rise, Clondalkin, Co. Dublin. *(Irish)*

O'Sullivan, Michael, Albright & Wilson (Ireland) Ltd, Ibex Works, Dunlaoghaire, Co. Dublin. *(Irish)*

Ruffell, Edward Charles, Lee Filters Ltd, Central Way, Walworth Industrial Estate, Andover, Hants. *(London)*

Shinmaru, Tetsuya, 2-36 Higashi 3 cho, Shukuyacho, Sakai, Osaka 590, Japan. *(General Overseas)*

Registered Students

Bourne, Jonathan Paul, Westwood, 5 The Beeches, West Didsbury, Manchester. *(Manchester)*

Davis, Jeffrey Martin, 229 Bury New Road, Whitefield, Bury, Manchester. *(Manchester)*

Irish, Kevin, 25 Park Parade, Dewsbury, West Yorkshire WF13 2QJ. *(West Riding)*

Nugent Allan, 39 Deveron Road, Westerton, Bearsden, Glasgow G61 1LN. *(Scottish)*

Pity he didn't know about Proxel preservatives!

What a waste – the paint stinks, so do the profits, and that's the end of a valuable customer. But with Proxel preservatives ICI has discovered the formula to save time, energy and money. They're the special preparations which give maximum protection against a wide range of paint spoilage organisms. Proxel preservatives are environmentally safe, low in toxicity, come in easy to handle liquid form and have long term chemical stability.

Compatible with both emulsion paints and latex emulsions, the range includes a variety of formulations. For details of the Proxel preservative that you need, contact your local ICI sales office.

ICI Organics Division

Hexagon House, Blackley, Manchester M9 3DA

OD 151

SHELL
CHEMICALS
FOR INDUSTRY

Resins
Epikote resins and Epikure curing agents, Cardura E10 and VeoVa 10 resin intermediates.

Elastomers
Cariflex TR thermoplastic rubber, Cariflex SBR, BR and IR.

Thermoplastics
Shell Polypropylene, Carlona low density polyethylene, Shell polystyrene,* Styrocell expandable polystyrene, Carina polyvinylchloride.*

Urethane Chemicals
Caradol polyols and Caradate isocyanates.

Base Chemicals
Ethylene, propylene, butadiene, dicyclopentadiene, benzene, toluene, solvent xylenes, ortho xylene, para xylene, cumene, styrene monomer, sulphur, Versatic 10, Dutrex and Shellflex grades, naphthenic acids, premium needle coke.

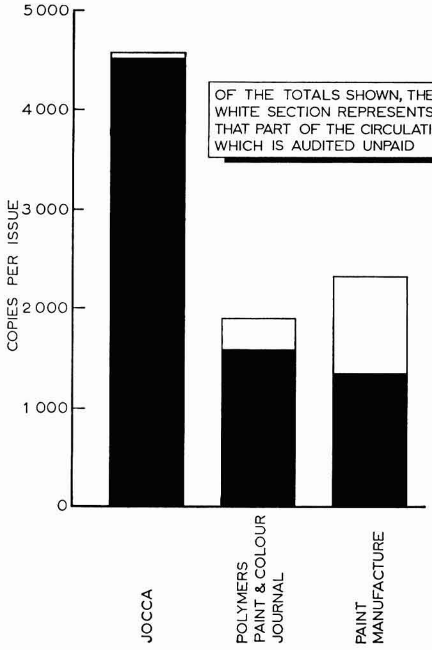
Industrial Chemicals
Chemical solvents, phenol, hydrocarbon solvents, detergent alkylates and alcohols, ethylene oxide, glycols and derivatives, plasticisers and plasticiser alcohols, epichlorhydrin, glycerine.

Speciality Chemicals
Fine chemicals, antioxidants, catalysts, mining and textile chemicals, Apiezon oils, greases and waxes, additives for lubricating oils and fuels.

*available in certain areas.

Shell Chemicals

Comparison of circulations of U.K. publications to the paint, printing ink and allied industries



(Reference Audit Bureau of Circulations Reviews. Jan-Dec 1976)

For full details of advertising in this, and other Association publications, contact D. M. Sanders, Assistant Editor

Journal of the Oil and Colour Chemists' Association (JOCCA)

Priory House, 967 Harrow Road, Wembley, Middx.
HA0 2SF, England **Telephone: 01-908 1086**
Telex: 922670 (OCCA Wembley)

CLASSIFIED ADVERTISEMENTS

Classified Advertisements are charged at the rate of £4.00 per cm. Advertisements for Situations Wanted are charged at £1.00 per line. A box number is charged at 50p. They should be sent to D. M. Sanders, Assistant Editor, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF. JOCCA is published EVERY month and Classified Advertisements can be accepted up to at least the 12th, and in exceptional circumstances the 20th of the month preceding publication. Advertisers who wish to arrange for an extension of the copy deadline should contact the Assistant Editor, D. M. Sanders, at the address given above (telephone 01-908 1086, telex 922670 OCCA Wembley).

APPOINTMENTS VACANT

FORMICA

Development Scientist

The Formica Products Research Centre at Maidenhead is concerned with research and development into new and existing decorative surfacing laminates for the Formica Companies in Europe and Africa.

A vacancy has arisen at the Centre for a Scientist to work on new products and processes from initiation to the factory implementation stage.

Candidates should have a degree in chemistry or polymer science and two to five years industrial experience, preferably in the development of polymers and their application as coatings.

Salary for this position is in the range £3,300 to £4,800.

Staff benefits include a subsidised canteen, free membership of B.U.P.A., four weeks annual holiday and a contributory pension scheme.

Please write giving details of age, qualifications and experience or telephone for an application for to:

Peter Yeomans, Formica Products Research Centre,
68 Lower Cookham Road, Maidenhead, Berks,
SL6 8LA Telephone: Maidenhead (0628) 21441.

CYANAMID

SENIOR PAINT TECHNOLOGIST

The Sterling Varnish Co. Ltd. has a vacancy for a Senior Paint Technologist at its Manchester factory.

Promotion prospects are excellent for the person selected who will be in the 30/45 age group and will report to the Development Manager. He will already have extensive experience in the high performance and industrial segments of the industry and appropriate professional qualifications.

Please reply, giving personal details to:

Development Manager,
S.C.D.,
The Sterling Varnish Co. Ltd.,
Fraser Road,
Trafford Park,
Manchester M17 1DU

The Sterling Varnish Company is a subsidiary of the Reichhold Chemical Incorporated of America.

OCCA-31 Exhibition

3-6 April 1979

Copies of the Invitation to Exhibit are available from the address on the Contents page.

RECONDITIONED EQUIPMENT FOR SALE

Flame-proofed Battery Electric Forklift Trucks!!!

By Conveyancer. 4500lbs capacity. Lifting heights from 9ft to 20ft. Also a full range of conventional Battery Electric Forklift Trucks. Full details and photographs available from:

Speed Electrics, The Wells Road, Nottingham.
Tel: 0602-609782

OCCAA Convention

The 20th Annual Convention of the Oil and Colour Chemists' Association Australia, will be held at the Sheraton Hotel, Perth, Western Australia from 24-26 August 1978. The Convention is being organised by the West Australian Section, under the auspices of the Australian Federal Committee.

Further details are obtainable from:

Mr U. Remund, c/o Berger Paints
WA Pty. Ltd, PO Box 55, Mt. Hawthorn, Western Australia 6106.

CLASSIFIED ADVERTISEMENTS

APPOINTMENTS VACANT

Resin Chemist

Royston, Herts

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Continued expansion has created a challenging new opening for a Resin Chemist to join a team working on the development and production of metallising pastes for the electronic component industry.

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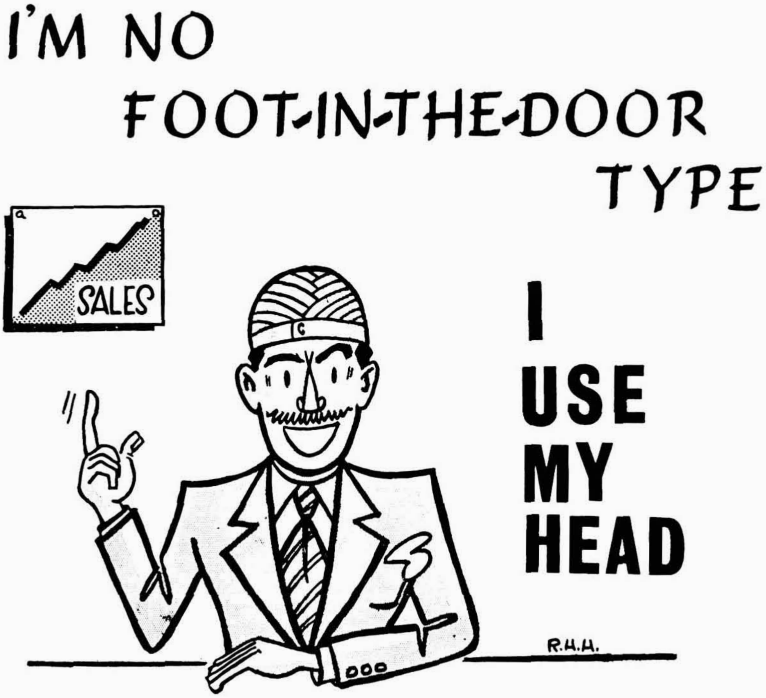

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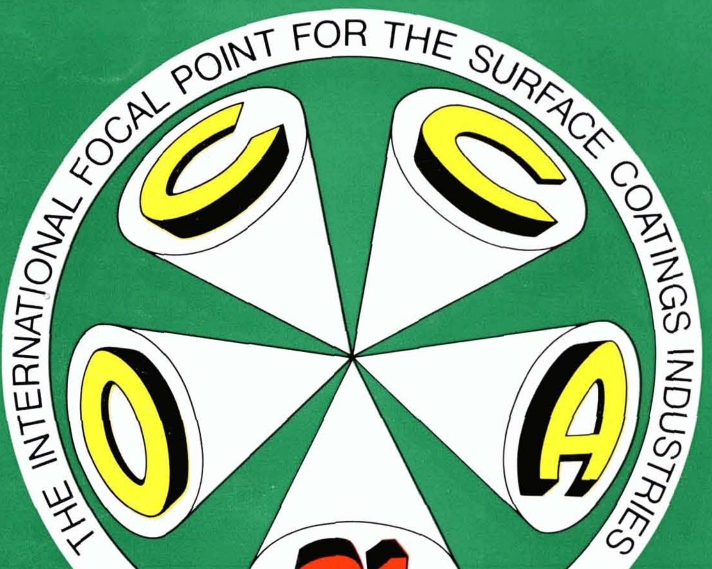

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3-6 APRIL 1979

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