

J O C C A





In this issue:

- Effect of titanium dioxide pigments on the cure of thermosetting films
- The adhesive properties of shellac modified with toluene di-isocyanate (TDI)

Wet Adhesion



Journal of Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex, HA0 2SF, England.



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Effect of titanium dioxide pigments on the cure of thermosetting films

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Summary

The effect of grade of titanium dioxide on the cure of thermosetting high solids paints, using hexa methoxymethyl melamine resin catalysed by acids and also of powder coatings crosslinked with epoxy resins or triglycidyl isocyanurate has been studied. In addition the cure of the high solids system has been compared with that of alkyd/UF paints cured at room temperature by the addition of acid catalyst.

The use of dielectric analysis to give an accurate and reliable assessment of the degree of cure is described. This method is more reproducible than film hardness, as measured by pendulum tests and furthermore it has been found that film hardness can reach a maximum value before full cure has been attained.

The pigment parameters that can affect cure are presence of zinc, as a crystal stabilising agent, and the alumina and silica present as coatings on the titanium dioxide pigment. Each can affect the cure to varying degrees, either retarding or improving it, depending upon the resin system. Reasons for some of these effects are given but others are more obscure.

Introduction

The theme of this year's conference is "New substrates, new materials, new problems". With regard to titanium dioxide pigments and new substrates, we have not yet, (and the "yet" must be stressed) found any instances where variation in paint performance on a substrate can be directly attributed to differences in grade of pigment. However, new materials have certainly posed new problems, not previously encountered with "traditional" paint systems, regarding the selection of the optimum grade of titanium dioxide pigment. Two such materials are high solids stoving paints and powder coatings. In both instances, the grade of pigment can significantly affect the degree of cure, the problem being exacerbated by the tendency to use lower stoving temperatures in order to conserve energy.

The reduction in film cure can result in a reduction in physical properties such as impact resistance and adhesion, in addition to film hardness; and also poorer weathering properties and resistance to chemicals and solvents. Whilst this paper highlights the problems and shows how they can be overcome, we are not yet able to give a complete explanation of why different pigments behave as they do.

In the case of high solids, the reduction in cure has been attributed to partial neutralisation of the catalyst by the pigment coating but, with other "traditional" catalysed systems using the same catalyst e.g. wood finishes based on alkyd/amino resins, the variation in performance is almost completely opposite; i.e., pigments which give the greatest degree of cure in stoved acid catalysed high solids paints generally give the softest films in air dried acid cured wood finishes.

Similarly, with powder coatings, the order of film hard-

ness with different grades of titanium dioxide pigments can be reversed by change of resin, even of the same type.

Although satisfactory explanations have not yet been deduced, it was considered that these variations in performance were of sufficient importance to bring to the attention of pigment users, and also possibly elicit the complete answers from our friends in the resin manufacturing industry.

The paper is divided into three parts. Part 1 describes earlier work with high solids systems in which the problems first became apparent, using tests such as film hardness and solvent resistance. The accuracy and reproducibility of such methods leave much to be desired and Part 2 describes the use of Dielectric Analysis to give a more reliable measurement of the degree of cure.

Part 3 gives some of the anomalous results obtained with thermosetting powders using "industry accepted tests" such as gel time and reverse impact and describes how Dielectric Analysis can be used to measure cure and also how the method of use can be modified to monitor the curing reaction.

Part 1 – Earlier work

(a) High Solids

The increasing pressure to reduce environmental pollution due to solvent emission resulted, in the mid/late 1970's, in a range of "high solids" resins being offered by most of the European resin manufacturers. These consisted of alkyds, polyesters and thermosetting acrylics generally crosslinked with hexa methoxymethyl melamine (HMMM) resins. Due to the relatively low reactivity of these amino resins, an acid catalyst was necessary to give the desired cure at an acceptable temperature. The most common catalyst was para toluene sulphonic acid (ptsa) or blocked derivatives thereof. Polyurethanes consisting of either polyesters or acrylics crosslinked with isocyanates were also offered.

Whilst none of these resin systems attained the desired level of 80 per cent non-volatile at application viscosity, the majority gave a significant increase over conventional resins (68-76 per cent cf 40-55 per cent).

While evaluating these high-solid resins to ascertain which titanium dioxide pigments gave the best results, it was found that the hardness of the stoved films varied significantly with the grade of pigment used. When the results were analysed it was found that the variation appeared to be dependent upon the inorganic coating on the pigment surface.

Tables 1(a), 1(b), 1(c) give hardness values using a Persoz pendulum (BS 3900 Part E5:1973) in which the time taken for the amplitude of swing to fall from 12° to 4° from the vertical is noted. Nine resin systems were used with

	Ta	ble I	
Persoz	Pendulum	Hardness	(seconds)

Resin	Alkyd (1)	Alkyd (2)	Polyester (3)	Polyester (4)	Polyester (5)	Polyester (6)	Acrylic (7)	Acrylic (8)	Acrylic (9)
P:B	0.8:1.0	1.0:1.0	1.0:1.0	0.8:1.0	0.9:1.0	1.0:1.0	0.8:1.0	1.0:1.0	0.9:1.0
HMMM content	20%	30%	30%	30%	36%	25%	30%	27%	29%
%ptsa (on HMMM)	1%	1%	1%	1%	1.8%	1%	1.2%	2.2%	2.1%
Temp	130°C	130°C	150°C	130°C	150°C	150°C	150°C	130°C	130°C
(a) Ca	talyst ptsa								
Pigment Type									
А	240	225	260	260	315	210	245	230	230
В	215	205	260	190	310	210	260	230	220
С	200	195	275	205	300	175	230	210	200
D	160	150	260	140	230	50	200	225	200
(b) Ca	talyst ptsa b	locked with	n morpholine	– quantity o	calculated or	active prop	ortion		
Α	250	245	275	270	310	225	250	220	235
В	210	220	280	250	320	200	250	230	230
С	200	205	270	240	300	135	240	180	200
D	180	170	250	120	250	50	180	180	160
(c) Ca	talyst Propri	etary Com	oound, block	ing agent ur	ndisclosed -	quantity calc	ulated on a	ctive propor	tion
A	265	215	285	275	280	225	250	170	250
В	240	205	270	220	240	150	240	130	250
С	255	140	270	225	225	110	230	130	230
D	240	95	260	50	170	50	200	95	135

three acid catalysts. The pigments could be categorised into four types according to the alumina/silica coating levels.

Pigment Type	Al ₂ O ₃	SiO ₂	Crystal stabilizing Agent
А	2.4	1.2	Zn
В	4.5	2.0	Al
С	3.0	0.1	Al
D	6.0	0.1	Al

The formulations, quantity of catalyst (calculated on weight of HMMM) and stoving temperatures were those recommended by the resin manufacturers.

For the purpose of this paper, pigments have been categorised as follows:

Туре	Coating	Crystal stabilizing agen			
Α	Silica/Alumina(<5%)	Zn			
В	Silica/Alumina(<6.5%)	Al			
С	Alumina (<4%)	Al			
D	Alumina (>4%)	Al			
E	Experimental	Zn or Al			
F	Uncoated	Zn or Al			
G	Silica/Alumina(>7%)	Al			
н	Zirconia/Alumina	Al			

With only one exception (Polyester 3) pigment D, which has the highest alumina coating, gave the softest film, whilst Pigment A, which has a mixed alumina/silica coating, gave the hardest. Pigment C, which contains only half the alumina present in Pigment D was generally intermediate between A and D. Pigment B, which has a similar alumina/silica ratio to Pigment A, but about double the quantity gave similar hardness values in the majority of instances, but was marginally softer in others (estimated experimental error ± 10 per cent).

Bertrand and Gosselin¹ using Polyester 5, pigmented with Pigments A, C and D showed that film hardness decreased with increasing P:B ratio (Figure 1a) and increased with quantity of catalyst (Figure 1b) and stoving temperature (Figure 1c). Thus if Pigment D was used in place of Pigment A, either the stoving temperature had to be increased by 40°C or the level of catalyst doubled from 1 to 2 per cent to achieve the same film hardness.

No such differences in film hardness had ever been previously noted using alkyds, polyesters or acrylics of "conventional" solids, crosslinked with amino resins but with which addition of catalyst was not necessary. This of course could be due to the stoving schedules normally employed being sufficiently severe to mask any differences due to pigment coating. To check this point three series of paints were made using alkyd/UF, alkyd/MF and thermosetting acrylic/MF systems of "conventional" solids pigmented with the same range of pigments used in the high solids evaluation. An uncoated grade of TiO₂ (Pigment F) was also included. Films were cured at temperatures 30°C

ptsa 1.9%



Figure 1a Variation in film hardness of high solids paints with pigment content and type of pigment

Figure 1b. Variation in film hardness of high solids paints with quantity of catalyst using three types of pigment

Figure 1c. Variation in film hardness of high solids paints with curing temperature using three types of pigment

below and above those recommended by the resin manufacturers.

Film hardness was measured using a König pendulum with which the time taken for the amplitude of swing to fall from 6° to 3° from the vertical is noted.

The results are contained in Table 2 and show that, with the two alkyd systems, although film hardness increased with stoving temperature there were no differences due to grade of pigment, whilst with the thermoset acrylic resin there were no differences due to either grade of pigment or stoving temperature (experimental error ± 10 per cent).

The evidence therefore suggested a catalyst/pigment coating interaction and Bertrand² showed that when an acid catalyst e.g. PTSA, was used, pigments containing a high proportion of alumina reduced the film hardness, (Figures 2a and 2c) whereas in systems catalysed by bases, e.g. tetra butyl ammonium iodide(TBAI) the hardness was reduced by pigments containing silica – Figures 2b and 2d.

It was postulated that the differences in film hardness were due to a catalyst/pigment coating interaction, dependent upon the relative basicity/acidity of the coatings. Alumina gives a basic surface which deactivates acid catalysts whereas silica imparts a degree of acidity which partially neutralises base catalysts.

He also showed that the reduction in cure resulted in poorer gloss retention and chalk resistance on exterior exposure. With the three resin systems of "conventional" solids the effect on weathering of varying the curing temperature by 30°C was similar with all pigments i.e. for each resin system the same curing temperature was the best

1986(2)

for all pigments, showing that the grade of pigment had not affected the degree of cure.

For conciseness, only the results with pigments A and D are given i.e. the grades which gave the extreme hardness results with the high solids systems (figures 3a - 3f).

With the castor alkyd/UF and TSA/MF, reducing the temperature resulted in only a slight lowering of gloss retention but had a greater effect on chalk resistance. Increasing the temperature had a slightly greater adverse effect, particularly on chalk resistance. Thus, with these two resin systems the curing schedules recommended by the resin manufacturers were optimum for weathering properties.

With the coco alkyd/MF system, better weathering, particularly chalk resistance, was obtained by reducing the curing temperature from 150°C to 120°C.

(b) Films acid-cured at room temperature

Although the evidence indicates that the variation in film hardness in high solids systems is due to a catalyst/pigment coating interaction, the deactivation of acid catalysts by alumina, present on the pigment surface, is certainly not universally true. One example is acid-cured alkyd/UF wood finishes which use relatively reactive UF resins plasticised by alkyds and cured at room temperature by addition of acid catalysts. Figures 4a – 4c are typical of a large number of resin combinations tested in Tioxide laboratories over the past 20-25 years.

Table 2
 König Pendulum Hardness (seconds) – Effect of stoving temperature

Pigment		Alkyd/UF			Alkyd/MF		TSA/MF				
Туре	90°C	120°C	150°C	120°C	150°C	180°C	120°C	150°C	180°C		
А	33	85	104	53	64	74	118	118	114		
В	31	84	101	50	68	75	118	110	112		
С	30	80	105	51	65	73	115	117	112		
D	31	86	110	55	69	76	118	116	116		
F				55	65	80	117	120	111		



Figure 2a. Influence of type of pigment on hardness/curing temperature curve for high solids paint catalysed by acid



Figure 2b. Influence of type of pigment on hardness/curing temperature curve for high solids paint catalysed by base



Figure 2c. Influence of type of pigment on hardness/acid catalyst curve for high solids paint



Figure 2d. Influence of type of pigment on hardness/base catalyst curve for high solids paint







Figure 3b. Influence of curing temperature on gloss retention and chalk resistance of films exposed at Florida. Pigment: D; Resin: Castor alkyd/UF







Figure 3d. Influence of curing temperature on gloss retention and chalk resistance of films exposed at Florida. Pigment: D; Resin: Coco alkyd/MF



Figure 3e. Influence of curing temperature on gloss retention and chalk resistance of films exposed at Florida. Pigment: A : Resin: TSA/MF



Figure 3f. Influence of curing temperature on gloss retention and chalk resistance of films exposed at Florida. Pigment: D; Resin: TSA/MF



Figure 4a. Influence of type of pigment on hardness development of acid cured, alkyd/UF wood finish cured at room temperature with normal quantity of acid catalyst

Figure 4b. Influence of type of pigment on hardness development of acid cured, alkyd/UF wood finish forced dried at 80°C for 30 minutes using normal quantity of acid catalyst

Figure 4c. Influence of type of pigment on hardness development of acid cured alkyd/UF wood finish cured at room temperature with reduced quantity of acid catalyst

Pigment Type	Al ₂ O ₃	SiO ₂	Crystal stabilising Agent	Organic additive
A1	2.4	1.2	Zn	Polyol
A2	2.4	1.2	Zn	Amine
B	4.5	2.0	Al	Polyol
C1	3.0	0.1	Al	Polyol
C2	4.0	0.1	Al	Polyol
G	4.3	9.2	Al	_

Using the normal quantity of catalyst i.e. 6 per cent ptsa calculated on UF solids, the only pigment to show any differences from the others when cured at room temperature is the heavily coated Pigment G (Figure 4a), but no differences are obtained when curing is assisted by heating at 80° C for 30 min (Figure 4b). However, if the amount of catalyst is reduced to 2 per cent then, although the film hardness is reduced with all pigments, there is a wide variation between grades of pigment (Figure 4c) (a similar variation is also obtained if the paints containing 6 per cent catalyst are allowed to stand for 7-14 days, after addition of catalyst, before application).

These variations had generally been explained as follows:

- Pigments A1 and A2 are zinc stabilised and the zinc could react with, and partially neutralise the acid catalyst and therefore give a softer film than the C type pigments.
- Pigment A2 has an amine organic treatment and this could react with the acid catalyst and thus give a softer film than Pigment A1 which is treated with an organic polyol.
- 3. Pigment G has a heavy inorganic coating which gives a very porous surface which will physically adsorb the acid catalyst (Surface area by nitrogen adsorption 30 m²/g cf 13m²/g for pigments A and C). Heating the films at 80°C for 30 minutes forces the catalyst out of the pores and hence catalyses the curing reaction and results in a film hardness similar to the other grades (Figure 4b).
- 4. The slightly softer film with Pigment B of Type C could also be due to slight physical adsorption of the catalyst by the slightly heavier coating (surface area $17m^2/g$).

The degrees of differences between pigments will generally be between those shown in Figures 4a and 4c. In some instances the differences between pigments of the same type e.g. C1 and C2, A1 and A2 are sufficiently small to be ignored. Where differences are obtained they are invariably in the order shown in Figure 4c. Factors, other than grade of pigment, which can affect hardness are type of alkyd e.g. type of oil, oil length, acid value, hydroxyl value and degree of esterification, reactivity of amino resin, alkyd:UF ratio, type and quantity of acid catalyst, solvent blend, non-volatile content and ambient temperature.

The above explanation does not take into account the relative basicity/acidity of the pigment surface which appears to be the dominant factor in the cure of high solids systems. If the hardness values obtained using Pigments C1, C2, B and A1 (all polyol treated) are considered

(Figure 4c) then the order of decreasing hardness is also the order of decreasing alumina/silica ratio

This is the absolute reverse of the high solids paints.

Part 2 - Use of dielectric analysis

As the catalyst/pigment coating interaction appeared to be completely opposite in the two systems, further investigations were carried out.

In the previous work, pendulum tests had been used to measure film hardness, but this describes only the state of the surface and, in addition, the accuracy and reproducibility leaves much to be desired (e.g. temperature, humidity and film thickness are some of the factors known to affect the values obtained). Solvent resistance had also been used to assess the degree of cure and, although the results were generally in agreement with the hardness measurements, the test is very subjective and the reproducibility and accuracy questionable. In order to obtain a more reliable assessment of cure, dielectric analysis using a technique developed by Simpson³ was used.

The principle involves applying an AC voltage of known frequency to the coating, thus producing a dynamic electrical stress. Dipoles, present due to polar groupings in the polymer, respond to the stress by attempting to align themselves with the electric field. Since these polar groups are attached to other molecules their mobility is restricted. The delayed response of the dipoles can be considered as a "lag" in vector terms and is quantified by a factor known as the dielectric loss tangent, tanð. As the degree of crosslinking increases, the restraint on the mobility of the dipoles increases and the amount of energy dissipated, tanð, increases.

If the coating under examination is subjected to a steady rise in temperature and tanð measured, it is found that tanð peaks at a temperature referred to as T_{α} . This peak is due to relaxation processes within the film, and is related to the glass transition temperature Tg. As the degree of cross-linking is increased the temperature at which the peak occurs increases.

A schematic diagram of the dielectric spectrometer is shown in Figure 5.

A Wayne Kerr Autobalance Universal Bridge B642 operating at a fixed frequency of 1592 Hz is used to impose a small voltage on the sample clamped between two electrodes. The resulting current is measured to provide values of conductance (G) and capacitance (C).

By definition

$$tan\delta = \frac{G}{C 2\pi f}$$
where f = applied frequency (1592Hz)

Although G and C are dependent upon film thickness, this cancels out and tanb is independent of film thickness.

The conductance and capacitance terminals of the bridge can be connected to an electronic ratio/amplifier unit enabling tan δ to be automatically determined. This value is



Figure 5. Schematic diagram of dielectric spectrometer used for T_{α} determination



Figure 6. Influence of quantity of catalyst on dielectric profile of high solids paint films cured at 130°C for 30 minutes

fed into one arm (y axis) of a x-y-t recorder. The sample, in its holder, is placed in a linear temperature programmable oven which allows the air temperature heating rates to be varied from 1°C to 40°C per minute. A thermocouple is placed in the base plate of the specimen holder to record specimen temperature. This value is fed into the other arm of the recorder (x axis) enabling profiles of tanð versus temperature to be obtained and hence T_α.

(a) High Solids Stoving Enamels

Typical plots of tan δ /temperature for films prepared using a high solids Polyester/HMMM system cured at 130°C and catalysed with different quantities of ptsa are shown in Figure 6. Hardness values (König pendulum) for films applied and stoved at the same time are given in perenthesis. As would be expected, the temperature T_a at which tan δ peaked, and the hardness values, increased with quantity of catalyst.

In the earlier work, the pigment parameters which appeared to affect the degree of film hardness were alumina/silica ratio, quantity of coating, use of zinc as crystal stabilising agent and type of organic treatment. Pigments having the desired permutations and combinations necessary to examine each factor, singly, in turn, are not commercially available on the Tioxide range of pigments and accordingly pigments were coated in the laboratory with the desired quantities of alumina and silica using base pigments manufactured on a production scale.

A series of paints was made using Polyester 10/HMMM(4:1) pigmented at P:B 1:1 using the experimental pigments. Commercial pigments A1 and A2, which had given the hardest films and pigment D, which had given the softest film, in previous work, were also included. Quantities of catalyst, added at the time of manufacture, were 0.5 per cent, 1.0 per cent and 2.0 per cent calculated on weight of HMMM. Curing was carried out at 130°C for 30 minutes.

Pigment	ZnO	Al_2O_3	SiO ₂	Organic treatment
E0		1.3		Polyol (uncoated grade)
E1		5.0	0.1	Polyol
E2		4.0	1.2	Polyol
E3		2.7	2.4	Polyol
E5	1.0	3.7	1.1	Polyol
E8	1.0	0.7	3.0	Polyol
E9	1.0	0.3	0.1	Polyol (uncoated grade)
A1	1.0	2.4	1.2	Polyol
A2	1.0	2.4	1.2	Amine
D		6.0	0.1	Polyol

Variations in T_α with quantity of catalyst for each pigment, are shown in Figures 7a and 7b and the hardness values (König pendulum) in Figures 8a and 8b. Both values increase with increasing catalyst for each pigment but the T_α curves are more consistent (fewer "cross-overs") than those for hardness. This indicates a greater degree of accuracy in T_α measurement than hardness.

 T_{α} and hardness values for one of the films catalysed at 2 per cent were also determined on a daily basis and it can be seen that T_{α} was much more consistent.

Age of films (days)						
	Initial	1	2	5	6	7
T _α (°C)	92.5	91.5	91.5	91.5	92.0	91.5
Hardness (sec)	183	195	193	187	183	191

The hardness values for all ten pigments at each catalyst

level are plotted against T_{α} in Figure 9. For the lowest and highest T_{α} values, hardness increases only slowly with increasing T_{α} , whereas at intermediate values both increase at a similar rate.

Correlation between T_{α} and hardness is reasonable and it is considered that the "scatter" is more likely to be due to inaccuracy in hardness measurement rather than in T_{α} determination.

 T_{α} is plotted against alumina content at each catalyst level in Figures 10a and 10b. For the non zinc containing pigments (Figure 10a) there is excellent correlation, T_{α} i.e. degree of cure, decreasing with increasing alumina content at the two lower levels of catalyst. At 2.0 per cent catalyst there is sufficient catalyst to both interact with the basic alumina coating and also to catalyse the crosslinking reaction between the HMMM and polyester. With the zinc stabilised pigments (Figure 10b) no such correlation exists. with E5, which has the highest alumina coating, giving the highest T_{α} of the coated grades. However, plotting T_{α} against silica content (Figure 11a) shows excellent correlation at 1.0 and 2.0 per cent catalyst and only one spurious result at 0.5 per cent catalyst. No similar correlation between T_{α} and silica exists with the zinc free pigments (Figure 11b) unless the results for the uncoated pigment E0 are ignored.

Comparing E9 with E0 and E5 with E2 (Figures 7a, 7b) i.e. zinc containing, and non zinc pigments with similar



Figure 7b. Influence of pigment coating (zinc stabilised pigments) on $T\alpha$ of films of high solids paints containing different quantities of catalyst





Figure 7a. Influence of pigment coating (zinc free pigments) on $T\alpha$ of films of high solids paints containing different quantities of catalyst

Figure 8. Influence of pigment coating on hardness of films of high solids paints containing different quantities of catalyst. a (upper zinc free pigments, b (lower) zinc stabilised pigments



Figure 9. Correlation between T_{α} and hardness for films of high solids paints



Figure 10. Correlation between $T\alpha$ of high solids paint films and alumina content of pigment coating. a (upper) zinc free pigments, b (lower) zinc stabilised pigments

coatings, the zinc containing pigments E9 and E5 have lower T_α values than their zinc free equivalents and in



Figure 11. Correlation between $T\alpha$ of high solids paint films and silica content of pigment coatings. a (upper) zinc stabilised pigments, b (lower) zinc free pigments

general the zinc containing pigments gave lower T_{α} values than the zinc free pigments.

Summarising, with high solids systems, the presence of zinc gives poorer cure. With zinc free pigments, curing is reduced by the presence of alumina in the coating, whilst with the zinc stabilised grades the presence of silica is detrimental to cure. Explanation of these contrary results is difficult. Obviously more than one mechanism is operative.

The adverse effect of increasing quantities of alumina with the zinc free pigments (E0, E1, E2, E3) is probably due to neutralisation of the acid catalyst, but this does not explain why a similar trend is not obtained with the zinc stabilised grades.

Similarly, zinc stabilised pigments will give poorer cure due to a reaction between zinc and the acid catalyst.

Reasons for the adverse effect of silica with the zinc stabilised grades are less obvious. One possibility is the interaction of the "acidic" silica with the amino resin, but no such interaction has been noted with resins of conventional solids.

(b) Acid cured alkyd/UF wood finishes

The curing of acid cured alkyd/UF wood finishes can be monitored by carrying out dielectric analysis of the films on a daily basis.

Typical plots of tan δ /temperature for different times of cure are shown in Figure 12 and illustrate how the temperature (T_a), at which tan δ peaks, increases as the film

cures. It is interesting that even after two weeks curing at room temperature, T_{α} is still below the value obtained by force drying at 80°C for 30 minutes.

A series of paints was made using both experimental and commercial pigments based on an alkyd/UF/MF resin combination. In order to accentuate differences between pigments, the amount of acid catalyst was reduced to 60% of the normal quantity. Films were applied, one hour after addition of catalyst, at a nominal wet film thickness of 125µm onto tinplate for T_α determinations and onto glass for hardness measurements. In addition to airdrying at room temperature, films were also forced dried at 80°C for 30 minutes, as frequently used in practice, and also stoved at 120°C for 30 minutes as it was considered that this would give close to maximum cure.

To ensure that sufficient time had elapsed for any catalyst/pigment interaction to take place, the tests were repeated 5 weeks after addition of catalyst. (Such an extended pot life time would not be required in practice). All the paints had increased in viscosity but it was still possible to obtain satisfactory films using $125\mu m$ shims and a glass doctor blade.

Increases in T_{α} and of film hardness (König pendulum) with curing time for the experimental pigments are shown in Figures 13a and 13b, and for the commercial pigments in Figures 14a and 14b for films applied 1 hour after addition of catalyst; and in Figures 15a, 15b, 16a and 16b for films applied after 5 weeks.

For both sets of pigments, tested both initially and after 5 weeks storage, the orders of hardness and of T_{α} were generally the same.



Figure 12. Dielectric profile of films of an acid cured alkyd/UF wood finish cured for different times



Figure 13a. Influence of type of pigment on $T\alpha$ /cure time curves for acid cured alkyd/UF wood finish – Experimental pigments. Films applied 1 hour after addition of catalyst



Figure 13b. Influence of type of pigment on hardness/cure time curves for acid cured alkyd/UF wood finish – Experimental pigments. Films applied 1 hour after addition of catalyst

The hardness values for all ten films, applied 1 hour after addition of catalysts, after different curing times, including films forced dried at 80°C and stoved at 120°C are plotted against the respective T_{α} values in Figure 17. In the early stages of cure, film hardness increased only slowly with increasing T_{α} followed by a period when both increased at a similar rate. Finally, film hardness increased only slightly



Figure 14a. Influence of type of pigment on T_a/cure time curves for acid cured alkyd/UF wood finish – Commercial pigments. Films applied 1 hour after addition of catalyst



Figure 14b. Influence of type of pigment on hardness/cure time curves for acid cured alkyd/UF wood finish – Commercial pigments. Films applied 1 hour after addition of catalyst

for larger increases in T_{α} . This is in agreement with the T_a/hardness curve obtained with the high solids system – Figure 9.

Films applied one hour after addition of catalyst

Comparing experimental pigments E5 and E2 (Figures 15a,



Figure 15a. Influence of type of pigments on Ta/cure time curves for cured alkyd/UF wood finish – Experimental pigments. Films applied 5 weeks after addition of catalyst



Figure 15b. Influence of type of pigment on hardness/cure time curves for acid cured alkyd/UF wood finish – Experimental pigments. Films applied 5 weeks after addition of catalyst.

15b) which have similar coatings but differ in crystal stabilising agent, the zinc stabilised pigment E5 gave poorer cure than the alumina stabilised grade E2, indicating an interaction between zinc and the acid catalyst.

Comparing pigments E1, E2 and E3 (Figures 15a, 15b) prepared from the same base pigment (alumina stabilised),



Figure 16a. Influence of type of pigment on Tα/cure time curves for acid cured alkyd/UF wood finish – Commercial pigments. Films applied 5 weeks after addition of catalyst



Figure 16b. Influence of type of pigment on hardness/cure time curves for acid cured alkyd/UF wood finish – Commercial pigments. Films applied 5 weeks after addition of catalyst

both hardness and T_{α} increased as the alumina/silica ratio was increased from 1:1 to 4:1 but no differences were obtained between 4:1 and 5:0.1 (Total weight of coating was similar in all cases -ca 5%). Similarly, comparing E5 and E8, which used the same zinc stabilised base pigment,



Figure 17. Correlation between $T\alpha$ and hardness of acid cured alkyd/UF wood finishes applied 1 hour after addition of catalyst

increasing the alumina/silica ratio increased both film hardness and T_{α} (Note total weight of coating varied with these two pigments).

With both sets of pigments (E1, E2, E3 and E5, E8) the variation in cure could be interpreted as better cure due to presence of alumina or poorer cure due to presence of silica but in neither case was the any evidence of neutralisation of catalyst by alumina.

With the commercial pigments, (Figures 16a, 16b) the alumina coated, zinc free pigments (Type C) gave better cure and harder films than the mixed alumina/silica coated, zinc stabilised Type A pigments, which is in agreement with the experimental pigments and also with long term experience. (On this occasion no differences were obtained between the amine treated A2 pigment and polyol treated A1).

As with the experimental pigments, the detrimental effect of zinc is obvious and the poorer performance of Pigment B compared with the two C type pigments suggests that the presence of silica may result in poorer cure.

Films applied five weeks after addition of catalyst

In all cases the rate of curing and hardness development at room temperature decreased compared with the initial results. With the experimental pigments, those coated with the lowest alumina/silica ratio decreased the least (compare E3, E1, E2; and E8 with E5) which suggests that a catalyst/alumina interaction occurs on long term storage. Even so, the higher alumina pigments were still, at best, only equal to the higher alumina pigments. With the commercial pigments no similar trend was discernible with the relative order of performance of both T_{α} and hardness unchanged, although at a lower value. With both sets of pigments, those containing zinc still gave the poorest performance.

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experimental and commercial pigments did not show any definite correlation for alumina content except that for zinc free pigments there appeared to be an optimum at about 4 per cent. However, for the zinc stabilised pigments there was an indication that the degree of cure decreased with increasing silica, being more pronounced initially than after 5 weeks storage. (This detrimental effect of silica in combination with presence of zinc was also obtained with the high solids paints).

Pigments containing between 1.3 per cent and 3.0 per cent silica will be necessary before more definite conclusions can be drawn. For zinc free pigments the degree of scatter of results was much greater and although a line can be drawn suggesting that silica is detrimental to cure when tested initially, after five weeks the "best fit" could be either with a slight gradient (silica detrimental) or horizontal (no effect), figures 18a, 18b, 19a and 19b.

Films cured at elevated temperatures

Force drying at 80°C or stoving at 120°C resulted in all pigments giving similar cure which suggests that either the more severe curing conditions are sufficient to eliminate differences between pigments, or the mechanism of cure at elevated tempeatures is different from that at room temperature. When tested after 5 weeks storage the T_{α} and hardness values were similar to, or only marginally lower than, the initial results i.e. whatever interaction occurred during long term storage had only a clight effect on cure at elevated temperatures. This again indicates different mechanisms of cure at elevated and room temperatures.

To summarise, for alkyd/UF wood finishes, acid cured at room temperatures, the best results are obtained by using zinc free pigments. A moderately high alumina/silica ratio coating will give the best initial results and, despite showing more loss of cure, after storage will still be at least equal to pigments of lower alumina/silica ratio. Higher values (above 4 per cent alumina) appear to be detrimental. Silica also has an adverse effect on curing, particularly with zinc stabilised grades and when tested immediately after addition of catalyst.

Comparing stoved, acid catalysed, high solids systems and air dried acid cured alkyd/UF paints,

- 1. Zinc will adversely affect cure in both cases.
- Presence of silica in the pigment coating is detrimental with both systems with zinc stabilised pigments.
- With high solids systems, increasing the amount of alumina results in poorer cure with zinc free pigments but in air dried acid cured alkyd/UF paints there appears to be an optimum intermediate level.
- 4. In high solids systems the quantity of alumina in the coating is the dominant factor whereas in acid cured alkyd/UF paints the presence of zinc has the greatest effect.

Part 3 – Powder coatings

One of the first main types of thermosetting powder coatings consisted of epoxy resins crosslinked with dicyandiamide or derivatives thereof. High temperatures of the order of 200° C or greater were used for curing. Variations in gel time (carried out at curing temperatures) were only slight and the resistance of cured films to tests such as reverse impact, solvent or chemical immersion, and



Figure 18. Correlation between $T\alpha$ and silica content of pigment coating for zinc stabilised pigments in acid cured alkyd/UF wood finish after different times of cure. a (upper) films applied 1 hour after addition of catalyst, b (lower) films applied 5 weeks after addition of catalyst



Figure 19. Correlation between $T\alpha$ and silica content of pigment coatings for zinc free pigments in acid cured alkyd/UF wood finish after different times of cure. a (upper) films applied 1 hour after addition of catalyst, b (lower) films applied 5 weeks after addition of catalyst

Table 3a Epoxy/Polyester 11 (1/1) P:B 0.6:1.0

Pigment	Gel time (secs)	Reverse Impact*	ZnO	SiO ₂	Al ₂ O ₃	Organic treatment
C1	215	Р		0.1	3.0	Polyol
C2	215	Р		0.1	4.0	Polyol
B1	240	Р		2.0	4.5	Polyol
B2	255	Р		1.3	3.5	Silicone/Amine
B3	255	Р		0.9	2.2	Polyol
B 4	255	Р		1.1	2.2	Silicone/Amine
A2	345	F	0.9	1.2	2.4	Amine
A1	370	F	0.9	1.2	2.4	Polyol
A3	370	F	1.0	1.6	2.1 .	Silicone/Amine
G1	360	F		9.2	4.3	
G2	365	F		6.3	4.3	

Ta	ble	36		
Epoxy/Polyester	12	(1/1)	P:B	0.6:1.0

Pigment	Gel time (secs)	Reverse Impact*	ZnO	SiO ₂	Al_2O_3	Organic treatment
A1	125	F	0.9	1.2	2.4	Polyol
A2	130	F	0.9	1.2	2.4	Amine
B5	210	F		0.7	3.2	Polyol
C2	225	F		0.1	4.0	Polyol
B1	260	F		2.0	4.5	Polyol

salt spray did not appear to be dependent upon the grade of titanium dioxide used. (This non-dependence on pigment grade could be due to either the resin system or the high curing temperatures).

In the mid/late 1970's, polyesters were introduced, crosslinked with either epoxy resin or triglycidyl isocyanurate (TGIC), the latter primarly intended for exterior applications. Curing temperatures were generally reduced to the order of 180°C/190°C and later, due to increasing pressure to reduce energy costs, temperatures as low as 160°C were used. With both polyester systems, and particularly at lower curing temperatures, it was found that the grade of pigment used could significantly affect gel times and film properties such as resistance to reverse impact and solvents.

The parameter that had the greatest influence was the presence or absence of zinc as the crystal stabilising agent, and to a lesser extent the pigment coating. The effect was very resin dependent, completely opposite results being obtained with different resins of nominally the same type. Typical results for two epoxy/polyester systems are given in tables 3a and 3b and for two polyester/TGIC systems in tables 4a and 4b. In each case the pigments have been placed in order of increasing gel time.

Gelation Time

The gelation times were determined using a "custom built" gel tester which is essentially an accurately controlled electrically heated steel block, in which there is a

depression to contain the powder sample. The temperature is pre-set to the required value and 0.25g powder placed in the depression. Stirring is carried out using a wooden cocktail stick and the time noted when a thread of molten powder could not be withdrawn from the main molten mass without it breaking.

Reverse impact test (BS3900:Part E3:1973)

Powders were applied by electrostatic spray to zinc phosphated steel panels (Bonderite 224) and cured at the specified schedule. Film thicknesses were measured using a Permascope ES and film thicknesses of 50μ m±5µm selected. The films were subjected to a reverse impact of 4.75 kg applied through 570 mm via a hemispherical steel probe of 7 mm radius. Any signs of cracking were noted as failure.

Reference to tables 3 and 4 shows the obvious variable effect of zinc. With the epoxy/polyester systems, the zinc containing A type pigments gave the longest gel times with polyester 11 (Table 3a) but the shortest with polyester 12 (Table 3b) and similarly with the two TGIC cured polyesters, (tables 4a and 4b), a reverse effect was obtained according to which polyester was used. Addition of colloidal zinc oxide had a similar effect to presence of zinc in the TiO₂ crystal, increasing the gel times with polyester 13 but decreasing it with polyester 14. However, in neither case was the effect of adding colloidal zinc oxide as great as when the zinc was contained in the titanium dioxide pigment.

Table 4a -Polyester 13/TGIC 93/7 P:B 0.5:1.0

Pigment	Gel time (secs)	Reverse Impact*	ZnO	SiO ₂	Al ₂ O ₃	ZrO ₂	Organic treatment
	220	Р					
H1	225	Р		0.1	4.0	1.5	Polyol
C2	245	Р		0.1	4.0		Polyol
B1	330	F		2.0	4.5		Polyol
B2	330	F		1.3	3.5		Silicone/Amine
G2	365	F		6.3	4.3		
G1	370	F		9.2	4.3		
A4	420	F	0.9	1.2	2.4		
A2	425	F	0.9	1.2	2.4		Amine
Al	450	F	0.9	1.2	2.4		Polyol
A3	450	F	1.0	1.6	2.1		Silicone/Amine
11+5%ZnO	335	F					
B1+5%ZnO	580	F					

Gel time temp: 160°C

Curing schedule: 13 min at 160°C

Note: If cured for 10 minutes at 180°C all powders, including those containing 5% zinc oxide (on weight of TiO₂), passed the reverse impact test. The gel times at 180°C varied from 90 secs to 230 secs with the pigments being in approximately the same order.

	Table	4(b)		
Polvester	14/TGIC	93/7	P:B	0.6:1.0

Pigment	Gel time (secs)	Reverse Impact*	ZnO	SiO ₂	Al ₂ O ₃	ZrO ₂	Organic treatment
	190	Р					
A3	160	Р	1.0	1.6	2.1		Silicone/Amine
Al	185	Р	0.9	1.2	2.4		Polyol
A5	185	Р	0.9	0.8	3.2		Polyol
A2	190	Р	0.9	1.2	2.4		Amine
A4	190	Р	0.9	1.2	2.4		
B2	265	F		1.3	3.5		Silicone/Amine
HI	295	F		0.1	4.0	1.5	Polyol
C2	300	F		0.1	4.0		Polyol
B1	320	F		2.0	4.5		Polyol
G1	350	F		9.2	4.3		753.
G2	355	F		6.3	4.3		
A5+1%ZnO	170	Р					
A5+5%ZnO	160	Р					
H1+1%ZnO	260	F					
H1+5%ZnO	240	F					
Gel time Curing se	temp: 190°C chedule: 13 m	in at 190°C					

The effect of pigment coating is more obscure. With polyester 11 (Table 3a) and polyester 13 (Table 4a) the silica/alumina coated B type pigments gave slightly longer gel times than the C type pigments, which have essentially alumina coatings, but no similar trend was discernible with polyesters 12 and 14 (tables 3b and 4b).

In all cases the heavily coated G type pigments gave longer gel times than the other zinc free pigments but this could be due to either the presence of silica or to the very high quantity of coating.

With polyesters 12, 13 and 14, there was reasonable correlation between gel time and resistance to reverse impact, the powders with the shortest gel times giving the best impact results. It is interesting that with polyester 13 (Table 4a), increasing the curing temperature from 160° to 180° C resulted in all the powders passing the impact test, whereas at 160° C only two pigmented powders were successful.

When using the commercial pigments, the effect on gel time of varying the alumina/silica coatings on the pigments was obscured due to differences in total quantity of coating and also having different organic treatments. Accordingly, the experimental pigments used in the high solids investigation, and which were all treated with a polyol, have been used in powder coatings. To date, tests on only one resin combination, an epoxy/polyester, have been completed.

Gel times and reverse impact resistance were determined as described above and also film hardness (König

Table 5 Epoxy/Polyester 15 (1.3/1.0) P:B 0.6:1.0

		Fi	lms cured at	180°C	Films cure	ed at 200°C			
Pigment	Gel time secs	Τα °C	Reverse Impact	Hardness König secs	Τα °C	Reverse Impact*	ZnO	SiO ₂	Al ₂ C
None	146	161	Р	210	158	Р			
E0	159	156	F	179	158	Р		0.0	1.3
E1	178	157	F	184	159	Р		0.1	5.0
E2	185	158.5	F	189	161	Р		1.2	4.0
E3	222	158	F	189	161.5	Р		2.4	2.7
E4	205	158.5	F	186	161	Р		1.3	1.5
E9	332	152.5	F	184	157	Р	1.0	0.1	0.3
E5	409	150	F	177	153	F	1.0	1.1	3.7
E6	418	148	F	189	151.5	F	1.0	1.2	2.7
E7	346	149	F	189	153.5	Р	1.0	2.3	1.5
E8	254	155.5	F	189	160	Р	1.0	3.0	0.7

pendulum). In addition T_{α} values were determined for films cured for 15 minutes at 180°C and also 15 minutes at 200°C.

Gel times can be regarded as a measure of the time taken to reach a given degree of polymerisation whereas T_{α} gives an indication of the degree of cure in the stoved film. Results are given in Table 5.

There was reasonable correlation between T_{α} and gel time (Figure 20), T_{α} increasing with decreasing gel time up to a maximum value of T_{α} , this maximum value increasing if the curing temperature was increased i.e. degree of cure, of films stoved for 15 minutes increased with rate of cure (gel time⁻¹) up to a maximum, which depended upon



Figure 20. Correlation between $T\alpha$ and gel times for epoxy/polyester powder coating

temperature of stoving.

This increase in maximum value of T_{α} infers that temperature of stoving is more important than time, to ensure complete cure. All films, except the unpigmented one, failed the reverse impact test when cured for 15 minutes at 180°C but only two (those with the longest gel times) failed when cured for 15 minutes at 200°C.

There was no minimum value of T_{α} (degree of cure) above which all films passed the reverse impact test; the powders pigmented with zinc free grades (E0-E4), when cured at 180°C gave T_{α} values of 156-158.5°C and failed, whereas E7 and E9 gave values of 153.5 and 157°C for films cured at 200°C and these passed.

With regard to effect of type of pigment on gel time and T_{α} , the most obvious differences were due to the presence of zinc which resulted in longer gel times and lower values of T_{α} . With the zinc free pigments E0-E4, the type of coating had no effect on degree of cure (T_{α}) and only a very slight effect on gel time. With the zinc stabilised grades the effect of varying the alumina/silica was much greater, particularly on gel time. Comparing E6, E7 and E8, all having a total coating of approximately 3.8 per cent, increasing the silica (or decreasing the alumina) had a beneficial effect i.e. shorter gel time and greater T_{α} .

Comparing E8 and E9 shows that depositing a silica coating onto the uncoated E9 pigment gives an improvement.

As previously stated, differences in gel time for the zinc free pigments were only slight, but comparing E1, E2 and E3 which had approximately 5.1 per cent total coating, varying the alumina/silica ratio had the opposite effect to that with the zinc stabilised grades; decreasing the silica (increasing the alumina) reduced the gel time. Also the uncoated base pigment E0 had the shortest gel time.

The König pendulum test showed only very slight differences, which were within experimental error, and show that film hardness, as measured by pendulum tests, does not give a reliable indication of film cure.

Similar tests with other resin combinations, particularly



Figure 21. Schematic diagram of electrode arrangement for monitoring the cure of powder coatings

those where the presence of zinc is beneficial, will be necessary before any definite conclusions can be drawn.

Alternative use of dielectric analysis with Powder Coatings

In addition to determining T_{α} for cured films as previously described, dielectric analysis can also be used to determine tanô as a function of cure time. A schematic diagram of the arrangement is shown in Figure 21. A known amount of powder is contained in a small aluminium pan which serves as one electrode. An insulating film is placed between the powder and upper electrode to prevent short circuiting between the electrodes due to porosity of the powder (or film). To prevent any air gaps forming between the powder/insulating film due to film shrinkage during curing, a strong spring is placed beneath the base plate so that pressure is constantly applied in an upward direction.

Figure 22 is a typical tan δ /time curve for powder coatings. Simpson³ postulated that t₁ correlates with the melting point, t₂ with onset of curing and t₃ with gel point.

Values for t_1 , t_2 and t_3 were determined using the Epoxy/Polyester 15 combination with the experimental pigments. Heating rates were 20°C and 40°C per minute. 0.3g of powder was used, first compressed into a pellet to exclude as much air as possible. At the heating rate of 20°C/min, the points t_1 , t_2 and t_3 were well defined, as illustrated in Figure 22 but, at the higher heating rate, a sharp point corresponding to t_1 was not obtained.

At the lower heating rate, t_1 varied from 280 seconds to 287 seconds for the different pigments indicating that grade of pigment did not significantly affect the melting point. Similarly t_2 varied from 313 seconds to 325 seconds at 40°C/minute, and from 210 seconds to 218 seconds at 40°C/minute, again indicating that onset of cure was



Figure 22. Dielectric profile for a powder coating as a function of curing time

independent of grade of pigment. However, t_3 i.e. time postulated as when gelation occurred also showed only slight variation, 384 to 398 seconds at 20°C/minute, and 264 to 288 seconds at 40°C/minute, whereas gel times determined using the heated bar apparatus gave values from 159 to 418 seconds. One possible reason for the discrepancy is that with the heated bar gel time tester, the heating rate is very rapid.

Obviously, further investigations are necessary, including different resin combinations. If t_3 does correspond to the point of gelation of the film, then its determination at different heating rates would be useful in determining the optimum curing schedule for articles of different heat masses.

Conclusions

A Different grades of titanium dioxide pigments can affect the cure of various coatings to varying degrees. Three such coatings are:

- 1. High solids stoving paints containing hexa methoxymethyl melamine resins catalysed by acids.
- 2. Cold cured alkyd/UF paints using reactive urea formaldehyde resins also catalysed by acids.
- Powder coatings based on polyesters crosslinked either by epoxy resins or triglycidyl isocyanurate.

The pigment parameters responsible are presence or absence of zinc and the alumina/silica content of the pigment coating. Results are not consistent between the three systems and the effect of alumina and silica is dependent upon whether the pigment contains zinc or not.

(a) Effect of zinc

With the two liquid paint systems the presence of zinc is always deterimental and is the dominant factor in the air dried alkyd/UF paints, whereas with powder coatings it can be either beneficial or detrimental, dependent upon the polyester resin used.

(b) Effect of silica

With zinc stabilised grades, increasing the quantity of silica

reduces the cure of both liquid paint systems, but improves the cure of the powder coatings. With zinc free grades, silica has no effect with high solids paints and a slightly detrimental effect in cold cured alkyd/UF paints and powder coatings.

(c) Effect of alumina

With zinc stabilised pigments, alumina improves the cure with both liquid systems, but gives poorer cure in powder coatings whilst with zinc free pigments it is very deterimental with high solids paints, being the most dominant factor, has a slightly beneficial effect with cold cured alkyd/UF systems and no effect in powder coatings.

It is obvious that with these types of coatings, choice of pigment is very critical but, the effects can be minimised by increasing the curing temperature and/or catalyst content.

B Dielectric analysis is a very useful method of assessing the degree of cure of paint and powder films, being much more reliable than the traditional methods such as pendulum hardness and solvent resistance and in the case of powder coatings, gel times and reverse impact tests. The traditional methods, however, are still very useful, particularly for monitoring quality of production. The alternative method of using dielectric analysis to monitor the cure of powder coatings through melting, onset of cure and gelation is potentially very useful, particularly for determining the curing schedules required for articles of widely different masses. However, further evaluation with other resin systems is necessary.

Acknowledgements

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[Recieved 16 April 1985

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next month' inve

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

Parameters controlling colour acceptance in latex paints by H. G. Stephen

Paint adhesion to galvanised steel surfaces, Part I: Effects of binder and pigments by W. J. van Ooij and R. C. Groot Non-phthalic alkyd resins based on maleopimaric acid by M. C. Shukla and A. K. Vasishtha



 Stoffe und Gewebe aller Art, Farbstoffe und verwandte Erzeugnisse in den U.S. möglich machten.

 Mr C. Hubbard Davis, Founder, Sub-Tropical Testing Service, Inc. (1904-1977)

 PO Box 560876, 8290 S.W. 120th Street, Miami, Florida, U.S.A. 33156

The adhesive properties of shellac modified with toluene di-isocyanate (TDI)

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Indian Lac Research Institute, Namkum, Ranchi 834010, Bihar, India.

Summary

The adhesive properties of shellac modified with toluene diisocyanate (TDI) over wood to wood and wood to mild steel surfaces has been studied. It was found that the incorporation of 40 per cent TDI in the case of wood to wood and 60 per cent in wood to mild steel surfaces (on the weight of shellac) led to an increase in bond strength from 0.053 ton/sq. inch to 0.223 ton/sq. inch and 0.066 ton/sq. inch to 0.275 ton/sq. inch respectively, which is four times more than plain shellac. These bonds are water and chemical resistant.

Introduction

Shellac, a well known natural resin behaves more like polyester as it consists of inter and intra-ester bonds of polyhydroxy carboxylic acids with about five hydroxyl and one carboxyl group on an average molecule (MW 1,000). Hence, it is possible to react shellac with polyisocyanates to form polyurethanes.

It has been reported¹ that TDI reacts only with hydroxyl groups while carboxyl groups of the resin does not take part in the reaction. Lower percentages¹ of TDI with shellac produce alcohol soluble products, but with stoichometric amounts insoluble products are obtained, obviously due to polyfunctionality of the resin. From time to time several surface coating compositions²⁻⁴ based on modified shellac and TDI have been studied and substantial improvement with regards to water, alkali and chemical resistance have been obtained. The present article embodies the results of the adhesive property of shellac treated with TDI over wood to wood (teak wood) and wood to mild steel surfaces which is not yet reported in the literature.

Experimental

Dewaxed lemon shellac solution (w/w) in dioxane (dried) in different proportion was prepared and bond strengths determined in the usual way⁵ from which an optimum composition was found (Table 1). To the optimum composition different percentages of TDI (on the weight of shellac) were added. After ten minutes addition of TDI, the solution was applied over the surfaces and the composites clamped for 24 hours. The adhesive strength of the various compositions was determined after ageing for seven days. The results are given in Table 2.

Results and Discussion

From Table 1 it is evident that the 25:75 shellac/dioxane composition has an optimum strength over wood to wood and wood to mild steel surfaces namely 0.053 and 0.066 ton/sq. inch respectively. Incorporation of TDI in the optimal composition i.e. 20, 30, 40, 50 and 60 per cent resulted an increase in bond strength of more than four times as compared to plain shellac (Table 2) on both surfaces (wood to wood and wood to mild steel). In the case of wood to wood surfaces 40 per cent TDI was found optimal and a further increase of TDI did not bring any improvement. However, for wood to mild steel surfaces 60

Adhesive strength of dewaxed lemon shellac (DLS)

Sample No	DLS/Dioxane	Bond strength wood to wood, w	(ton/sq. inch) rood to mild steel
1	(a) 15 : 85 (w/w)	0.036	0.045
	(b) 20 : 80 (w/w)	0.036	0.063
	(c) 25 : 75 (w/w)	0.053	0.066
	(d) 30 : 70 (w/w)	No homogenous s	solution is formed

Table 2.

Adhesive strength of DLS in presence of TDI

Sample No	e 25 : 75 (w/w)	Bond st wood to wood	rength (ton wood to mild steel	/sq. inch) pot life at room temp. 30°C (minutes)
2	(a) 20 per cent TDI	0.087	0.067	30-32
	(b) 30 per cent TDI	0.163	0.069	25-26
	(c) 40 per cent TDI	0.223	0.075	25-26
	(d) 50 per cent TDI	0.223	0.150	20-22
	(e) 60 per cent TDI	0.206	0.275	15-18

per cent TDI was effective and lower percentage did not show much improvement.

On the basis of the reactivity number of shellac, i.e. 350 (hydroxyl value 280 and acid value 70), the total amount of TDI required for complete reaction would be 52.7g per 100g of shellac resin and more or less the same composition was found in the present case as indicated by the bond strength data. The pot life of the compositions (Table 2), however, is short, but it could be increased by lowering the reaction temperature.

Stanley *et al*⁶ have shown that at cryogenic temperature bond strength increases considerably as compared to the bond strength determined after ageing at room temperature. They also studied the effect of elevated temperatures, i.e. 250 and 400°F, and observed that the value was lower than the value obtained at room temperature. Similar studies were also carried out in the present case (Table 3), where it was found that the value at cryogenic temperature did not increase but rather decreased. However, at higher temperatures similar observations were noticed.

Delamination test

Both the bonds (wood to wood and wood to mild steel), when dipped in water withstood several days without any

Table 3.

Effect of temperature on bond strength

	25 : 75 (w/w)		Bond strengt	h (ton/sq. inch)
Sample No	and 40% TDI	Curing of the bond	wood to wood	wood to mild steel
3		(a) at room temp. (b) overnight at room temp	0.223	0.150
		and then 3 hrs. at 0°C (c) overnight at room temp.	0.083	0.140
		and then 3 hrs. at 71.8°C (d) overnight at room temp.	0.14	0.05
		and then 3 hrs. at 121°C	0.183	0.06

change. These bonds also resisted 5 per cent sulphuric acid, 2 per cent caustic soda, toluene, white spirit and acetone.

Acknowledgement

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letter

Wet Adhesion and Corrosion

Sir,

I am prompted to take up the challenge of my old friend Peter Walker (*JOCCA*, 1985, 67, (**12**), 319) to join this debate, particularly because in his penultimate paragraph he supports the thesis which I put forward to the American Chemical Society in 1971¹. This was that adhesion forces must always be considered in relation to the forces generated in the coating or imposed externally; in many situations a very low adhesion force may suffice to provide a sound bond. The argument is developed further in a chapter, by John Prosser and myself in a recently published book on industrial adhesion².

However, on the specific question of the significance of wet adhesion to underfilm corrosion the balance of the argument is, I believe, with Prof. Funke. This is not to suggest that poor wet adhesion always implies poor corrosion resistance; as with most coating failure a combination of several adverse factors is necessary to generate underfilm corrosion.

Let us consider first the membrane properties of a porefree coating of water resistant material at least 100 μ m thick. First the electrical resistance measured vertically over a 10 cm² area should be well above 10⁹ ohms. Assuming a corrosion cell potential of 1 volt the current through the film will be sufficient to correspond to the dissolution of only 8.4 μ g of ferrous ions per year, a negligible corrosion rate. For the same film area oxygen permeation is likely to be less than 10 mg per year, which would also seem to be a reasonably effective control on

corrosion. Water permeation varies more with film composition but is likely to be at least ten times greater.

With these film properties, for rapid and significant underfilm corrosion to occur there appear to be two prerequisites. First, there must be breaks or porous flaws in the coating, including accidental misses or subsequent mechanical damage, to allow oxygen and water to reach the anodes. Second, and crucially, local adhesion failure must allow a channel of electrolyte to develop between the coating and the substrate so that ions can migrate between cathodic and anodic areas. Such local lack of adhesion may result from inadequate surface preparation, e.g. air or soluble salts trapped in residual rust, from stresses developed during film formation, from retention of watermiscible solvents or from mechanical shock but is most likely to involve attack by water as envisaged by Prof. Funke. The failure induced by water need not be complete: a residual surface layer of the order of 5 nm thick, as described by Mr Walker, would not introduce an effective impedance into the electrical circuit.

It would seem that a paint coating can protect a metal substrate either by providing a perfectly continuous membrane or by adhering soundly, even when continuously immersed in water; preferably both functions should be attempted. Chemically active inhibitors are needed only if the coating is deficient in either respect. Indeed, ar inhibitor can only function by supplying ions to the electrolyte, which will only occur after the requirements for

letter

corrosion to take place have been set up. Not surprisingly the formulation guidelines for effective use of corrosion inhibitors, namely high pigmentation leading to film porosity, run directly counter to those for good barrier protection. Perhaps the ideal heavy duty protection system might be a sandwich, consisting of:

- 1. An unpigmented sealer applied directly to the metal, incorporating adhesion promoters to ensure maximum wet adhesion.
- A middle layer containing inhibitor, formulated to make possible ready diffusion to any area under threat of attack; such a porous layer could also impede transfer of stresses to the sealer coat.
- 3. A thick top layer of weatherproof barrier material.

Finally, both Prof. Funke and Mr Walker have failed to stress sufficiently the importance of corrosion-stimulating ions in metal corrosion. Traces of chlorides and sulphates, present at the metal/paint interface accelerate and sustain corrosion to a remarkable extent. Chlorides, presumably by a combination of hygroscopicity and entering into the chemistry of the corrosion process, stimulate filiform corrosion under coatings even at normal room humidities, e.g. on steel filing cabinets. Invariably, the contents of the blisters first formed during chloride induced corrosion are markedly alkaline; this is true even in an acidified salt spray test, using a pH4 solution. The indications are therefore that the adhesion failure occurs at cathodic areas and is associated with hydroxyl ion attack on the paint binder (or the paint/metal interface) rather than with undercutting corrosion of the metal. It is suggested that blistering in sea water or dilute HCl and filiform corrosion are different manifestations of the same chemical and physical processes. Under the very wet conditions of a salt spray test rather thick thread-like corrosion trails have been observed to grow at right angles to deliberate scratch damage (the anodes) whereas under much drier conditions filiform trails are thinner and stagger randomly, presumably seeking out spots of minimum adhesion for cathodic alkali to raise the growing corrosion tunnel. If these suppositions are correct the correlation reported by Mr Van Ooij, JOCCA 1985,

between blistering in acidic solutions (presumably dilute HCl) and susceptibility to filiform corrosion would be expected.

25 Spring Gardens	
Dorking	
Surrey RH4 1EE	

Yours faithfully T. R. Bullett.

13 December 1985

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Dear Sir

The current interest in "Wet Adhesion" and corrosion resistance prompts me to write to you concerning a cycling salt spray test which can offer a valuable contribution to this important controversy.

In 1979 (JOCCA 62 131-135) you published my paper describing the development of a cycling salt spray at British Rail Research Division, Derby. My investigation clearly implied "Adhesion is Protection" and I coined a word to emphasize this simple philosophy.

Regrettably, this word has been commercialised and consequently, general opinion feels this will affect the acceptance of the cycling salt spray as a British Standard.

I would be very grateful if by this letter, the many people who contact me or are interested would refer to the test by its original name *viz:* B. R. 77 CYCLING SALT SPRAY TEST.

7 St Mary's Road Dunsville Doncaster DN7 4DQ 7 St Mary's Road F. D. Timmins, C Chem, FRSC. 13 December 1985

occa meeting

Newcastle Section

New developments in pigmentation of surface coatings.

The second meeting of the 1985/6 session was sponsored by BASF and Mr I. Cameron gave a talk on "New levelopments in pigmentation of surface coatings".

He prefaced the talk with a review of the background against which paint and raw material manufacturers have worked in recent years, in particular the change in attitudes towards environmental protection and health and safety. These are reflected in the various lobbies and pressure groups against lead, chromes, isocyanates etc. and the ensuing governmental control regulations which have required companies to change long-established paint formulations, usually with increased cost and reduced performance. The pigment industry has also needed to improve its safety standards e.g. efficient disposal and elimination of carcinogenic intermediates.

Labelling regulations have proliferated and lead chrome pigments, despite substantial evidence to the contrary, may yet be labelled as possible carcinogens. Lead content aspects are complicated by uncertainty regarding the best method of determining "soluble lead" in paints. There have been four methods specified in revisions of ISO 6713 since 1980 but the UK government continues to specify the original in its lead regulations. Despite their excellent pigmentary properties, and continued reduction in "soluble lead" contents, there has been a wholesale switch to non-

occa meeting/

lead types in Decorative and many Industrial/Automotive systems. Future Vehicle Refinish systems will naturally follow the Automotive originals.

Mr Cameron discussed the desirable properties of chrome substitutes. The high density/high hiding/low oil absorption of chromes makes them ideal for highly saturated yellows and reds at, or below, 15 per cent PVC to give excellent hiding, gloss and gloss retention. Organic substitutes have never achieved this, despite having excellent colour fastness, and paint costs on organics are two to five times higher than on chromes. Development of higher hiding organics has only partially eliminated price/performance differentials.

Modern inorganic complex pigments e.g. titanium/ nickel/chrome yellows, bismuth vanadate, have the necessary density and hiding to provide alternative answers, and blends with low hiding/high colour strength organics are often better value than higher hiding power organics. Mr Cameron then compared the various yellow, orange and red organics available as chrome substitutes—the simple, least expensive monoazos, the rather better pyrazoquinazolones, the more versatile and expensive polycyclics (quinophthalones, isoindolines, perylenes) and the latest co-finished blends of calcined nickel/chrome titanium pigments and organic polycyclic yellows.

Finally, Mr Cameron surveyed efforts to replace the proven carcinogenic zinc chrome anticorrosive pigments. He listed many other materials now available, concluding with a review of several zinc phosphate, zinc borate and zinc organic pigments offered by BASF.

After a short question period, the vote of thanks for a very absorbing talk was given by the Chairman, Mr G. D. Carr.

J. Bravey

Thames Valley Section

Coatings for Building Products

The second technical meeting of the Thames Valley Section on 31 October 1985 was generously sponsored by UCB (Chem) Ltd. Prior to the meeting a buffet was enjoyed by 43 members and guests followed by a paper presented by Dr A Vandermissen.

The speaker, in his introduction, outlined the history and involvement of his company in the field of coatings on fibro-cement and concrete products.

A detailed account was given regarding the need to provide protection against the formation of white calcium salt efflorescence, formed during the curing of the cement, together with the provision of maximum repellency insuring durability of the fibro-cement slates and corrugated sheets.

Monomers available for polymerisation and individual

selection depending on specific physical characteristics and properties demanded in the final coating were referred to; a range of polymer dispersions now exists with varying minimum film forming temperatures, wide range of particle size and corresponding flexible/hardness values.

Final reference was made to other consituents involved in the coating formulation. This indicated a need for careful selection of the following:

- Coalescent Solvents
- Thickeners
- Wetting aids/dispersants
- Pigments

The evening closed with a lively question time and a vote of thanks was proposed by Mr J A Gant.

J. A. Gant

Transvaal Section

Production of pentaerythritol

A meeting of the Transvaal Section on 16 October 1985. was addressed by Dr Charles Alexander, of NCP, on the subject, "Production of Pentaerythritol".

The address covered the following aspects:

- 1. International scene.
- 2. The history of "Penta" manufacture in South Africa.
- 3. Production
 - -chemistry
 - -raw materials
 - -process
 - -control
- 4. Summary.

At the end of the lecture a number of questions were raised from the floor.

A vote of thanks was proposed by Mr Neville Holland and the Chairman thanked NCP for sponsoring the meeting.

Hyperdispersants

On 6 November 1985, the Transvaal Section met at ICI House in Johannesburg where the meeting was addressed by Mr Peter Smith on the subject "Hyperdispersants".

The term Hyperdispersant has been devised to describe a novel class of dispersing aids.

When used in the production of solvent based paints. they facilitate the dispersion process and stabilise the dispersion produced.

They act as viscosity depressants in the mill base, and hence permit a much higher concentration of pigment or

occa meeting

extender at the dispersion stage, whilst significantly reducing energy requirements.

Productivity is increased dramatically when compared to conventional milling. Ball milling in solvent alone with Hyperdispersants present can achieve poductivity factors of between 2 and 3, i.e. two to three times the amount of paint can be produced from the same mill base volume. A 30 per cent increase in throughput can be achieved when using continuous milling techniques.

Improvements in the colour strength development of pigment can be substantial, with obvious cost savings to the paintmaker.

The choice of Hyperdispersant for the various pigments and extenders relates to their surface characteristics, but is mainly influenced by their Polar or Non Polar nature. Synergistic combinations of the Hyperdispersants are particularly effective with the Non Polar pigment types.

Hyperdispersant technology permits major innovations in the field of dispersion relating to paint production.

It is now possible to disperse pigments or extenders at very high concentrations (about 30-55 per cent for organic pigments, and 70-90 per cent for inorganic pigments) in solvent alone.

A single pigment concentrate can be prepared in a solvent common to various resin systems. This provides the paintmaker with a flexibility of production previously unobtainable by conventional methods.

The benefits described allow the paintmaker to use his resources of labour, energy, equipment and formulating skill much more flexibly and effectively.

The vote of thanks was proposed by Mr Peter Gate and the Chairman thanked ICI for their sponsorship of the meeting.

R. E. Cromarty

Midland Section

Filtration

The first technical lecture of the Midland Section was held on 17 October 1985, at the Clarendon Suite, Stirling Road, Edgbaston, Birmingham.

Members and guests heard Mr P. Tipping of Jenag Equipment Ltd., give a talk entitled "Filtration".

The speaker said he preferred to use the term straining and not filtration when talking about removing dirt particles from paints and inks. Then, with the aid of slides the speaker described the working of the Jenag Filter. A monofilament mesh is held in a vertical position between two metal plates. The paint or ink to be strained is pulled through the mesh by a pump, then some of it is returned back through the mesh giving a self-cleaning action so preventing it becoming clogged. Because the mesh is held in the vertical position the dirt falls to the bottom of the filter and can be removed. These filters are suitable for all types and consistency of paints or inks including high viscosity pastes. Depending on the size of mesh used they will remove all dirt particles down to about 20 microns. Because these machines are not very efficient below 20 microns the speaker would not recommend them for use with automotive paints.

A short question followed Mr Tipping's talk and the meeting finally closed with a vote of thanks proposed by Mr K. Dobell.

B. E. Myatt

Natal Section

Typical paint failures

On Tuesday 22 October 1985, a joint lecture with the S. A. Corrosion Institute was held at Shell House, Durban, when Mr M. Eveleigh of Plascon Evans Paints, Natal, gave a lecture on "Typical Paint Failures—their Causes and Cures".

The speaker divided this subject into three main headings viz:

1. Incorrect Specification

This aspect is dependent on structural design and is often subjected to cost cutting. Environmental conditions are important and a thorough investigation of the requirements of the coatings is imperative.

2. Incorrect Preparation

It is also affected by cost cutting and in many instances failure to comply with correct preparation techniques is hidden under subsequent paint films.

There is a lot of ignorance of paint specifications and they are often not read. Lack of supervision is prevalent and it is therefore advisable to appoint independent inspectors. Often there is insufficient time to complete a job and the painting being the last operation is rushed.

3. Incorrect Execution

Covers areas of paint handling, stirring, straining and storage. Also mixing and thinning ratios must be adhered to. Faulty paint can slip through a manufacturer's quality control and in many instances only the end user can identify the problem.

Equipment must be kept clean and in good condition but above all the manufacturer's recommendation of usage should always be read.

After a short question time Mr T. Henning gave a vote of thanks to the speaker and Messrs Plascon Evans, Natal, for sponsoring the evening's refreshments.

conference dircurrionr

Discussion is encouraged at OCCA Conferences following the presentation of papers. Those who put the questions were asked to fill in discussion slips with the details of their questions. Edited versions of the discussions that followed some of the papers published in the **December 1985** and **January 1986** issues appear below: questions in ordinary roman type, answers in italic. Only those questions for which discussion slips were received are published.

Novel corrosion inhibitors

Z. Kalewicz

D. SCANTLEBURY: What is your evidence for a true metallic bond between your iron phosphide and zinc?

Z. KALEWICZ: There is no metallic bond between di-iron phosphide and zinc. But the surface of the DIP nucleus is modified by presence of Zn diffusion coating. This implies an interaction between coating and substrate on a physical/chemical level, subject to the usual laws of diffusion and phase diagram restraints. From an electro-chemical standpoint there is now substantial evidence that a thus modified highly conductive nucleus is physically active, serving as an alternate cathodic site for the pigment particle.

W. J. VAN OOIJ: Have you looked at the distribution of zinc corrosion products throughout the primer following salt spray corrosion, and was this distribution different from that of normal zinc-rich primers?

Z. KALEWICZ: We did not look at the distribution of zinc corrosion products throughout the primer film. We found, only, that during all salt spray corrosion tests, there appeared no white rust on the surface of panels.

New materials for the furniture industry

A. J. Sparkes

B. WINDSOR: With reference to your statement on the use of bagasse in board manufacture. Is this material suspect in relation to biological activity—it was once upheld as an asbestos fibre replacement thixotrops in bitumen products but rejected on Health and Safety grounds?

A. J. SPARKES: Yes, there is a potential hazard due to residual sugar/fungal activity and use of fungicides needed, which can be an "additional hazard". There is very little bagasse used in the UK due to raw material supply (sugar cane) being centred in the Caribbean area.

Formulation of solvent-based paints pigmented with titanium dioxide

J. E. McNutt

J. R. TAYLOR: With respect to Figure 9, the angle MLK's size shows the danger of seeding. Has any work been carried out on solvent parameters and the size of the angle MLK, and can this be used for a computer system?

J. E. McNUTT: We are working now to understand the quantitative relationship between these angles and the hazards of seeding. I will have more to report on that subject at a later time. One complication is that seeding is affected not only by differences in composition of the letdown and millbase vehicles, but also by factors such as rate of addition and quality of mixing.

We are also working on computerization of the graphical operations which I have described.

C. SCHOFF: (Comment) Dr Fettis asked if the ternary diagrams could be used to choose letdowns and optimise systems. My comment is: "Such information definitely could be used to optimise millbases and letdowns if the data

from the diagrams were on a computer. The difficult part is to acquire all the data necessary to draw the diagrams properly. Once the data is in the computer, a number of optimising techniques can be used".

J. E. McNUTT: I thank Dr Schoff for his comment and agree with him. Finding volume-weight relationships is usually easy for commercial materials, but sometimes requires some extra work for proprietary resins.

G. C. FETTIS: Does your technique lend itself to computerization? Can it take account of optimization of production e.g. in minimizing the number of intermediates required?

J. E. McNUTT: Yes, the method can be computerized, and this work is underway. I expect to be able to report on that at a later stage. You make an interesting suggestion, and I will pursue it. I imagine that a computerized program will enable optimization of quantities and costs of intermediates.

Reprints of articles and papers in **JOCCA** are now available

If interested tel: 01-908 1086

new/

Ceramics change

From 1 January 1986, the companies previously trading as Anzon Ltd and HMR Group Ltd (comprising Harrison Mayer, CE Ramsden, George Edwards, Colin McNeal and Cerglas), are now trading as Cookson Ceramics & Antimony Ltd.

The change has been brought about as part of the development of the recently structured Ceramics & Antimony Division of Cookson Group plc. The Division is responsible for the entire ceramics supply, technical ceramics, zircon, and antimony activities of the Group outside of the USA.

By combining previously fragmented resources and with the ability to adopt more positive marketing and more efficient operation, the new company is the major arm of a Division which now has an immensely strong base in international markets. In addition to its own operating companies in Europe, the Division is backed by the Cookson Group resources with some 60 subsidiary and related companies in over 200 locations worldwide producing a turnover approaching £1bn per year.

Cookson Ceramics & Antimony Ltd has four manufacturing facilities in the UK—three at Stoke-on-Trent and one at Howden, Wallsend. The head office at Meir in Stoke is also the Divisional headquarters and sales and accounting have now been centralised at this location.

Reader Enquiry Service No. 30

K+E UK blending

Facilities for blending its water-based and solvent-based inks have been installed in the UK by K + E Printing Inks. Previously carried out in Stuttgart, ink blends can now be produced in a few days compared with two weeks.

Using eleven basic colours, K + E can manufacture the 97 colours in its HKS range, as well as an infinite number of shades to match customer requirements. Batch sizes from 20 kilos can usually be blended in a day and delivered within the following 24 hours.

Inks being produced with the new facilities include Flextol for pre-printing liner board, Aquaflex for paper sacks, Flexoplastol HG for printing on polythene, Flexoplastol HGF for PVC and Flexodessin P for paper. The full ranges of Heliodessin and Helioplastol gravure inks for paper, board and polythene are also included. Reader Service Enquiry No. 31

Silicone development

Durham Chemicals distributors have recently been appointed official distributors for the Baysilone range of silicone-based products manufactured by Bayer AG of Leverkusen, West Germany. The product range includes paint additives to improve flow and gloss levels and stop pigment floatation, as well as lubricants and fluid emulsions for the plastics, fibre, glass and ceramics industries, and a full range of resins and antifoams for use in the textile, surface coating, food and chemicals industry.

Supplier wins powder prize

Top prize in the 1985 Venables Awards, offered by the Paintmakers' Association for the best papers on powder coatings, has gone to Tioxide UK Ltd. This is the first time a raw material supplier has gained an Award.

The prize of £400 was shared by the two authors, D. L. Foulger and L. A. Simpson, of a paper on the use of dielectric analysis to monitor powder curing. Dr L. A. Mr David Hooper, the market development manager for Durham Chemicals distributors commented, "This exciting new distribution agreement with Bayer enables us to offer a complete range of silicone-based products to our customers to fully complement our existing comprehensive product range for the textile and surface coating industries".

Reader Enquiry Service No. 32

Polymer Laboratories new sales office in Germany

Polymer Laboratories have announced the opening of their new sales office in Heidelberg, under the direction of Mrs Doreen Lococo.

This has been set up to help liaison with the fast growing number of customers for Polymer Laboratories instruments in West Germany.

Reader Enquiry Service No. 33

Simpson is a member of the Newcastle Section of OCCA.

^{*} The second prize of £200 was awarded to S. M. Drew of International Paint plc. His topic was the application of instrumental colour management to powder manufacture.

The closing date for entries for the 1986 awards is 31 July. Entry forms are available from the Association at Alembic House, 93 Albert Embankment, London SE1 7TY.



Quality and inventory control

Berger Industrial Coatings are using a Husky IS intrinsically safe handheld computer as an aid to quality and inventory control in their manufacturing plant at Chadwell Heath, Essex.

The Husky IS is used as a portable datacapture device in areas of the plant where potentially flammable atmospheres preclude the use of conventional instrumentation or computers, while the data recorded on the handheld unit is used to provide accurate, instantly updated information for inventory control of raw materials.

Berger sees inventory control as a key element in the quality of the finished



The Husky IS

products, but the task of recording workin-progress data for batch production has traditionally been complicated by the number of different raw materials involved in the product range (over 700) and the fact that every dry ingredient has to be preweighed and every production step recorded as it happens.

Berger Business Systems, the data-

new/

processing service division of the group, have written a program which prompts the operative to take 'start' and 'stop' readings from the solvent meters, and which checks to see if that solvent has already been added. If it has, the operative will be reminded of the fact, and asked whether he still wishes to proceed.

In certain situations, the system will not let the operative proceed any further until his supervisor has checked that he is carrying out the correct procedure. This is achieved by 'locking up' the Husky until the supervisor enters a pre-arranged password.

The Husky IS units used by Berger Industrial Coatings are 80K Byte models, which are capable of each holding a full day's data. However, in practice, the units are downloaded two or three times daily to an Ericsson personal computer in a nearby non-hazardous area.

The data from the Husky IS units is checked and collated by the desktop computer and then sent via telephone lines to the company's IBM 3083 central mainframe computer for analysis using the COPICS order-picking and inventorycontrol software package.

The Chadwell Health plant manufactures paints and coatings for car manufacturers such as Ford, Vauxhall and Land Rover as well as for the automotive refinishing business. In addition, it supplies the general industrial marketplace in areas such as business machines, tractors, plastic components and packaging, while another important area is in protective finishes for installations such as oil rigs, the Thames Barrier and military systems. Raw materials represent a very high proportion of the cost of such finishes.

Reader Enquiry Service No. 34

Viscosity measurement

The AE oscillation viscometer, a new system designed for measuring viscosity over a wide range of temperatures and pressures, is now available from George Meller Ltd. It is claimed it can measure viscosity over a 6 decade range to $2 \times 10^{\circ}$ cp in pure fluids, slurries, emulsions, blends and other complex mixtures. The system's wide operating parameters are said to make it ideal for a broad range of applications including petrochemical, pharmaceutical, food, paint, waxes, thermoplastics and portland cement compositions.

The system, which provides extreme accuracy and repeatability, includes a



The AE oscillation viscometer

heating/cooling unit, a pressure vessel with viscosity probe and transducer, and an electronic control unit. In operation, the control unit oscillates the viscosity probe at the primary resonance frequency of the transducer. The increase in power required to maintain this oscillation in a fluid is converted to viscosity units. Shear rate is virtually independent of sample viscosity.

The system illustrated operates under pressures from atmospheric to 5,000 psi and at temperatures from -35° to $+150^{\circ}$ C, although higher temperature and pressure systems can be supplied.

Reader Enquiry Service No. 35

Chindwell Door Finishing Kit

Glasurit Beck has produced a Lasutect Door Finishing Kit for Chindwell doors, the tropical hardwood door supplier.

Chindwell recommends Lasutect for its combination of high build film and moisture vapour permeability which gives long-term protection to its doors. Moisture, if trapped within the timber, can



The Lasutect door finishing kit

lead to joint openings, panel splitting or warping. Lasutect's unique film is said to allow moisture vapour to escape without harming the wood.

Glasurit's Lasutect Door Kit is said to contain sufficient HL base coat and DSL top coat to protect any Chindwell door. Chindwell hardwood doors are assembled from machined components of kiln-dried timber, which has been carefully selected to minimise natural surface defects such as knots. All operations from felling to manufacture are integrated to combine DIY convenience with modern plantation technology.

Lasutect, which now contains high solid binder for improved performance, is a twocan translucent wood protection system that is moisture vapour permeable to allow the wood to breathe. Without this control of moisture, timber expands differentially and cracks. It would also be exposed to the harmful effects of ultraviolet light which will destroy the timber surface.

Reader Enquiry Service No. 36



Pye Unicam plans IR summer school

A five-day summer school on infrared spectrophotometry is being organised by Pye Unicam Ltd of Cambridge.

It will provide instruction in modem instrumentation techniques, including data processing, spectral interpretation and the latest sample handling methods. Participants will, it is said, come away with a comprehensive understanding of the theory and practice of IR spectrophotometry.

The annual school, now in its 25th year. will be held at the company's applications laboratory, Cambridge, from 28 July to? August 1986, and specialists from universities, industry and Pye Unicam will be in attendance to advise on specific sampling, interpretation and application problems.

Reader Enquiry Service No. 3

1986 FSCT Annual Meeting

The 1986 annual meeting of the Federatior of Societies for Coatings Technology will have as its theme, "Compliance and Quality: Recognising the Opportunities".

new/

it was announced by program chairman Percy Pierce, of PPG Industries Inc. The annual meeting will be held in conjunction with the Paint Industries' Show at the World Congress Center, Atlanta, GA, 5-7 November.

The theme focuses on emerging technologies (such as water-based, highsolids, and powder coatings) which are helping the industry meet regulatory requirements. Increased awareness and understanding of potential physiological and toxicological effects of coatings and their raw materials, have spurred development of these technologies, which are also responding to increased expectation of quality products, processes, methods, and attitudes.

Prospective speakers are invited to present original papers on the theme and its various aspects, and are requested to submit abstracts (150 to 200 words) for review to: Percy Pierce, PPG Industries Inc., R & D Center, P.O. Box 9, Allison Park, PA 15101. The deadline for receipt of abstracts is 1 March 1986.

Symposium on Health and Safety

The London Section of the Oil and Colour Chemists' Association are holding a oneday symposium entitled "Health and Safety in the Coatings and User Industries". This will be held at the Royal Institution, London W1, on Thursday, 10 April, 1986, commencing at 10 am with coffee being available from 9.30 am.

The meeting will be of interest to Production and Laboratory Managers because of their responsibility for ensuring safe working conditions for their workers as well as to Health and Safety Officers who are directly involved.

The programme will include information on the toxicity of raw materials, labelling for supply and conveyance, and risks of fire and explosion. Prominently featured will be the implications of the control of Substances Hazardous to Health Regulations with speakers from Paintmakers' Association and the London School of Hygiene and Tropical Medicine.

For further information contact:

Mr K. H. Arbuckle, Berger Group, Freshwater Road, Dagenham, Essex RM8 1RU. 01-590 6030.

occa new/

The Association's Exhibition of raw materials and equipment for the surface coatings industries



Readers will be aware that the new concept exhibition for the surface coatings industries organised by the Association (known as SURFEX 86) will be held at Harrogate in May 1986 and all space in Hall D, reception and gallery areas has been reserved.

The exhibition, which takes place at the Harrogate Conference Centre, Yorkshire, England, will be open on the following dates and times:

Wednesday 14 May 1986 09.30 - 18.00 hrs.

Thursday 15 May 1986 09.30 - 17.00 hrs.

On the first evening the West Riding Section of the Association is arranging a Dinner to take place at the Hotel Majestic, Harrogate, and a considerable number of tickets have already been sold. Details have been printed in the members' monthly bulletin and non-members wishing to apply for tickets (£16 each inclusive of VAT) should apply to Mr. G. C. Alderson, c/o Sandoz Products Ltd., Calverley Lane, Horsforth, Leeds LS18 4RP (Tel: 0532 584 646).

The following hotels in Harrogate have offered special rates for exhibitors and

visitors to SURFEX 86. Those wishing to avail themselves of this facility should state clearly that they are attending/visiting SURFEX 86 when booking accommodation:

Old Swan Hotel Granby Hotel The Cairn Hotel The Gables Hotel Adelphi Hotel Askern Guest House

The Official Guide to SURFEX 86 will be printed in the April issue of JOCCA and will also be freely available at the entrance to the Exhibition. Admission will be free.

Advertising in the April issue will not be confined to exhibitors and other companies/organisations wishing to have details of these facilities should contact Priory House as soon as possible.

The full list of exhibitors is published elsewhere in this Journal.

Prior to the exhibition the Paint Research Association will be organising a seminar in Harrogate at the Cairn Hotel on the 12 and 13 May on the subject "Coatings for plastics".



Annual General Meeting 1986

The 1986 Annual General Meeting of the Association will take place at the Unicorn Hotel, Prince Street, Bristol, and will be preceded by a Reunion Luncheon to which Past Presidents, Past Honorary Officers and Honorary Members of the Association are invited as the Association's guests.

Following the Luncheon an illustrated talk will be given by a speaker from the SS Great Britain Project which will deal with the work of restoration and the problems of corrosion involved in this important project.

This talk has been arranged by the Bristol Section of the Association and it is felt that members of the Association will be most interested in the talk on this project, and those visiting Bristol for the AGM may like to take this opportunity to go and see the *SS Great Britain* on the same day.

The Unicorn Hotel has agreed on a special rate for any member attending the AGM wishing to stay overnight either on Tuesday 17 or Wednesday 18 and those wishing to avail themselves of this facility should write to the Unicorn Hotel, stating that they are attending the Association's Luncheon Lecture in order to obtain the preferential rate.

Forms of application for the Luncheon will be sent to members with the 1985 Annual Report in May *JOCCA*.

occa new/

Provided there are sufficient members wishing to participate it may be possible to arrange a guided tour of the SS Great Britain at 10.00 am on the morning of the AGM (Wednesday, 18 June 1986) and applications for the visit should be made at the same time as application for luncheon tickets. However, members travelling from abroad who wish to participate but would not receive the May issue in time should write to Priory House as soon as possible indicating that they would like to take part in the tour and/or luncheon; details will then be sent to them in May by air mail.

Natal Section

11th National Symposium Call for papers

The Natal Section of OCCA will be hosting the 11th National Technical Symposium on 21-25 October 1986. Previous Symposia have offered both local and overseas speakers the opportunity to address a large technically competent audience including South Africa's leading paint technologists. In addition, papers have usually been published in JOCCA or the local technical journals.

The theme of the Symposium is: 'OCCA '86 COATINGS FOR ALL REASONS'

Authors are invited to submit details of papers for consideration by the Selection Committee not later than the end of March 1986. Information required: Name of author(s), title of paper, and a 300-word synopsis.

All correspondence should be addressed to: The Honorary Secretary, Natal Section, Oil and Colour Chemists' Association, Private Bag X504, Umbogintwini, 4120, South Africa.

West Riding

Golf Trophy

The above Stableford competition was again played at Wetherby Golf Club on Thursday 17 October 1985, the starting time being brought forward an hour to 1 pm because of the fears of bad light possibly hampering the late starters. These fears proved to be groundless for the afternoon turned out to be idyllic for golf—a still, warm, cloudless afternoon, perfect Indian Summer weather. Twenty members and guests competed and their high scores reflected the ideal playing conditions. The winner of the West Riding Trophy was Philip Jones with 35 points, the highest guest score and winner of the sweep was Martin Smith with 42 points.

Following a traditional Yorkshire meal the prizes were presented by Section Chairman, Terry Wright, and thanks to the generosity of many competing companies, all competitors again received a prize, thereby rounding off a perfect day.

T. M. Wright

News of members

Roger Handley

We were pleased to hear from Roger Handley, who until recently was Honorary Publications Secretary of the Manchester Section, about his new venture. Roger is now organising the TY CRAIG HOLIDAY PARK near Borth, Wales. He tells us that the Park is just over a mile from the three miles of sandy beach at Borth and that it is open from 1 March to 31 October, with caravans to let and for sale as well as a three-bedroom chalet. He intends to make the site into a select one offering quality service, and we extend our best wishes to him in his new venture.

OCCA Conference

Call for Papers

OCCA's next Biennial Conference will be held in Eastbourne from 17 - 20 June, 1987 under the general title "Advances and application of science and technology in surface coatings".

It is intended to hold four technical sessions in the mornings and afternoons of 18 and 19 June, 1987, each comprising five papers, giving a total of 20 papers.

The objectives of the Conference will be to review the advances made in the science and technology of surface coatings in both the commercial and academic fields and it is intended to encompass as wide a field as possible in the paint, printing ink and allied industries. Attention will also be directed to future possible trends and implications in these industries.

The Honorary 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, UK. (Tel: 01-908 1086, telex 922670).



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

Ordinary members

- Aston, R., B.Sc. (Auckland)
- Boden, P. T. (Manchester)
- Bush, P., B.A. (Ontario)
- Casey, P. F. (London)
- Cauchois, G. P. (Thames Valley)
- Cooke, S. A., B.Sc. (Ontario)
- Cutrone, L., M.Sc. (Newcastle)
- Dennett, P., B.Sc. (Manchester)
- Drummond, C. S. (Midlands)
- George, P. D., B.Sc. (General Overseas -Zimbabwe)
- Gillard, T. M., M.Sc. (London)
- Greidanus, P. J. (General Overseas -Netherlands)
- Haworth, L., B.Sc. (Auckland)
- Herbert, R. L., B.Sc. Midlands)
- Jackson, R. C. (Manchester)
- Jarvis, J. (Manchester)
- Kelsey, G. C. (London)
- Kemeys, K. L. (Auckland)
- Kent, D. J. (General Overseas Germany)
- King, G. A. (Cape)
- Mitrosparas, M. J. (Transvaal)
- Moore, H. B. (Ontario)
- Parsons, W. (Ontario)
- Pemberton R. W., B.Sc. (Auckland)
- Smit, M. N. (Natal)
- Vandenburg, A. D. D. (Natal)
- Wilkie, D. W., B.Sc. (Wellington)

Associate members

Greaves, K. (Transvaal) Naidoo, P. (Natal) Reeve, R. J. (Natal) Rybowski, D. (Bristol) Tuckerman, B. J. (Wellington) Zwicky, M. (Cape)

Registered students

Anderson, N. P. (Bristol) Jones, T. A. (Transvaal) CLASSIFIED ADVERTISEMENTS

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The Harlow Chemical Company is jointly owned by Hoechst (UK) Ltd and Yule Catto plc.

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D. Fagg, Protective Papers, Riverside Works, Watchet, Somerset TA23 0BW.

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Relevant experience is essential—at least 5 years in the development and fomulation of general industrial surface coating—and personal qualities of initiative and enthusiasm are sought. An application form is obtainable upon written request to:

Personnel Manager, Goodlass Wall & Company Ltd., Goodlass Road, Speke, Liverpool L24 9HJ.

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Personnel Department, Scott Bader Company Limited, Wollaston, Wellingborough, Northants NN9 7RL. Telephone: Wellingborough 663100, ext 214.



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OIL and COLOUR CHEMISTS' ASSOCIATION



OCCA CONFERENCE 1987



17-20 JUNE, 1987

CALL FOR PAPERS

ADVANCES AND APPLICATION OF SCIENCE AND TECHNOLOGY IN SURFACE COATINGS

OCCA's next Biennial Conference will be held in Eastbourne from 17-20 June 1987. The theme for the Association's Conference, which in the past has attracted visitors from many parts of the world, will be "Advances and application of science and technology in surface coatings".

The objectives of the Conference will be to review the advances made in the science and technology of surface coatings in both the commercial and academic fields and it is intended to encompass as wide a field as possible in the paint, printing ink and allied industries. Attention will also be directed to future possible trends and implications in these industries.

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