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JOURNAL^{***} OF THE IL & COLOUR CHEMISTS' ASSOCIATION

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The effect of part replacement of titanium dioxide by barytes on the properties of alkyd paints

K. V. S. N. Raju and M. Yaseen

Styrene copolymerisation of isomerised tobacco seed (*Nicotiana tobacum*) oil and its alkyd

M. S. Saxena, P. K. Jain and A. K. Vasishtha

Organosilanes as adhesion promoters for organic coatings. Part 1: silanes on the metal surface

P. Walker

Colour measurement by K. McLaren and R. P. Best has unfortunately been withdrawn from this issue due to prior arrangements having been made for its publication elsewhere.



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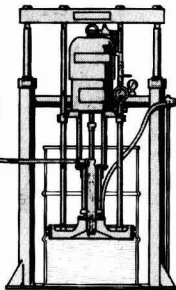
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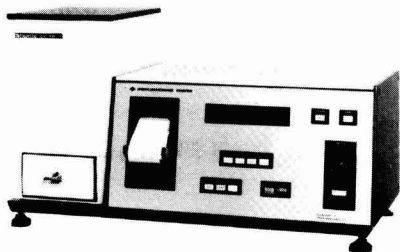
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Following the innovation at the 1981 Conference there will be a discourse session included in the fourth technical session.

For further information contact: **The Director & Secretary, Oil & Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex HA0 2SF, England (Tel: 01-908 1086; Telex: 922670 OCCA G).**

Transactions and Communications

The effect of part replacement of titanium dioxide by barytes on the properties of alkyd paints

By K. V. S. N. Raju and M. Yaseen

Regional Research Laboratory, Hyderabad-500 009, India

Summary

The effect of the part replacement of titanium dioxide pigment by barytes extender on the properties of alkyd-based paints has been studied. For a quantitative assessment of the changes in the properties of paints the following parameters were investigated: tensile strength, percentage elongation, water absorption and water vapour permeation; these were in addition to the more conventional tests for retention of gloss, scratch hardness, flexibility and resistance to impact of a falling weight. The tests

were carried out periodically on coatings exposed outdoors and indoors for a period of a little over six months. They showed that paints pigmented solely with titanium dioxide can tolerate its replacement with a barytes extender to the extent of 10 per cent PVC (pigment volume concentration) of extender in a paint having 30 per cent PVC without any adverse influence on the properties of the paints.

Keywords

Types and classes of coatings and allied products

alkyd coating

Raw materials for coatings

prime pigments and dyes

titanium dioxide

extender pigments

barytes

extender pigment

L'effet, sur les caractéristiques de peintures alkydes, du remplacement partiel de dioxyde de titane par barytine

Résumé

On a étudié l'effet, sur les caractéristiques de peintures à base des résines alkydes, du remplacement partiel de dioxyde de titane par barytine. En plus des essais classiques, c'est-à-dire, rétention de brillant, résistance à la rayure, résistance au choc (chute de poids), souplesse, on a effectué des essais tels que résistance à la traction, élongation par pourcentage, absorption d'eau, et perméabilité à la vapeur d'eau, afin d'établir un moyen d'appréciation quantitative des changements de propriétés des

peintures. Ces essais, effectués périodiquement sur des peintures exposées à la fois à l'intérieure et à l'extérieure pendant un intervalle de six mois à peu près, démontraient dans le cas des peintures à base de dioxyde de titane où la concentration pigmentaire par volume est de 30%, qu'il est possible de remplacer jusqu'à 10% de la concentration pigmentaire par volume du dioxyde de titane par barytine sans aucune influence défavorable sur les caractéristiques des peintures.

Der Einfluss auf die Eigenschaften der Alkydharzlacke der teilweisen Ersetzung des Titandioxids durch Baryt

Zusammenfassung

Der Einfluss auf die Eigenschaften der auf Alkydharzen gegründeten Beschichtungen der teilweisen Ersetzung des Titandioxids durch Baryt wurden untersucht. Abgesehen von den konventionellen Versuchen wie Glanzhaltung, Kratzfestigkeit, Flexibilität, Gewichtfallfestigkeit, wurden solche Versuche wie Zugfestigkeit, prozentuelle Ausdehnung, Wasseraufnahme und Wasserdampfdurchlässigkeit durchgeführt, um eine quantitative Auswertung der Veränderungen der Beschichtungseigenschaften

festzustellen. Auf Beschichtungen, die innen und aussen während etwa sechs Monate ausgelegt wurden, wurden diese Versuche periodisch durchgeführt, und zeigten, dass im Falle der, auf Titandioxid gegründeten Beschichtungen deren Pigmentvolumenkonzentration 30% ist, ist es möglich, ohne ungünstigen Einfluss auf die Filmeigenschaften, bis 10% der Pigmentvolumenkonzentration des Titandioxids durch Baryt zu ersetzen.

Introduction

Refs 1-22

Inert pigments having a low refractive index and being relatively inexpensive are used as extenders or fillers in

paint formulations. They not only help reduce costs, they also influence properties such as: (i) flow and rheological movement under stress, (ii) under-coat interaction, (iii) gloss, (iv) levelling after application, (v) mechanical and impact resistance, (vi) hiding, (vii) reflectance and brightness, (viii) dispersibility, (ix) colour and shade, (x)

flatness, (xi) sedimentation and cake formation, (xii) prevention of bacterial growth etc. These properties can be controlled very well or improved by the choice of the right types of extenders. However, these extenders may have adverse effects if used beyond optimum limits.

The term extender or filler given to such pigments dates back to when their sole function was considered to facilitate the production of cheaper paints. Studies in recent decades have indicated their importance in the manufacture of paints with respect to specific end-uses¹. The incorporation of calcium carbonate and diatomaceous silica^{2,3} or a blend of micronised talc and dolomite^{4,5} in TiO₂ pigmented paints results in good colour uniformity, opacity and washability. Similarly, the addition of finely ground extenders to paints produces better dispersion uniformity, structuring, colour tone, hiding, gloss and reflectance⁶⁻⁹. Nazrenko *et al.*¹⁰, found that excessive loading of micronised extenders in TiO₂ pigmented paints has adverse effects on the properties of the paints. Lyng¹¹ points out that one should be careful in exchanging a prescribed extender for another in a paint formulation.

Cremer¹² found that the replacement of TiO₂ by finely divided BaSO₄ (blanc fixe) up to a TiO₂/blanc fixe ratio of 7:3 did not affect the properties of air drying alkyd finishes. The part replacement of prime pigments by extenders in paint formulations is tolerated to a certain limit. The right choice of system, procedures of grinding and dispersion, pigment volume concentration and solvents produces paints having properties better than those without extenders¹³⁻¹⁶. In TiO₂ or iron oxide-based primers the presence of extenders improves their corrosion resistance and adhesion, apart from other properties¹⁷⁻²¹. In a recent publication, Sathyanarayana *et al.*²² have reported that properties like adhesion, gloss, scratch hardness and tensile strength are not affected significantly in the case of un-weathered coatings of paints in which TiO₂ is partly replaced by zinc oxide, talc, china clay and barytes to the extent of 30 per cent by weight.

The study of the part replacement of prime pigments by extenders has concentrated more on the evaluation of appearance than on other properties. Moreover, not much information is available about the changes that take place with regard to the physical and mechanical properties of coatings having different loadings of extenders, especially in the course of weathering. The data presented here highlight these aspects.

Materials

The major types of materials used in this study are described below:

1. Medium oil length alkyd containing 55 per cent linseed oil, glycerol and phthalic anhydride, prepared by standard monoglyceride process – acid value 13.5, hydroxyl value 38.0 and average molecular weight 2428.
2. Commercial pigment grade TiO₂ and barytes²².
3. White spirit/xylene mixture in the ratio²² 1:1.
4. Cobalt and lead naphthenates as driers.

The details of paint formulations are given in Table 1.

Table 1
Paint formulations

Binder: 55% linseed glycerol phthalate, acid value = 13.5, OH value = 38.0			
Formulation No.	Pigment (TiO ₂) % PVC	Extender (barytes) % PVC	Total % PVC
I	30	0	30
II	25	5	30
III	20	10	30
IV	15	15	30
V	10	20	30
VI	5	25	30
VII	0	30	30
VIII		unpigmented binder	

The ingredients were ground to Hegman gauge 7-8 in a Red Devil (Kady Mill) and adjusted with solvents to 60 sec flow through Ford cup No. 4. Driers were added to the formulations 24 hours before use.

Tests conducted on painted panels

1. Retention of gloss
2. Scratch hardness
3. Resistance to impact
4. Flexibility

Tests conducted on free films

1. Tensile strength at break
2. Percentage elongation under stress
3. Water absorption
 - (a) in film exposed to atmosphere saturated with water vapour
 - (b) in film immersed in water
4. Permeation of water vapour through film under concentration gradient 100 to 50 per cent relative humidity.

Test Procedures

Refs. 23-27

The methods used for the surface preparation and painting of mild steel panels (150 × 100 × 1.5 mm) were the standard procedures prescribed for gloss and impact resistance tests²³. Similarly, tin plated thin sheets were painted for conducting scratch hardness tests using a powered automatic tester (Research Equipment Ltd, UK), and flexibility tests using a conical mandrel (Sheen Instruments, UK).

The free films of paints for conducting tests on un-weathered coatings and coatings exposed outdoors and indoors for definite periods, were prepared by using a substrate of tin which was later amalgamated with mercury²⁴. Water absorption in films was determined using standard procedures²⁵. Rate of permeation of water vapour through free films was found by the wet cup method described elsewhere²⁶. The tensile properties of free films under

stress were determined using the table model 1026 tensile tester (Instron Limited, UK) and data were analysed for the best values at break²⁷.

Test panels for weathering

Refs. 25, 28, 29

The total number of painted panels required for conducting the various tests on un-weathered or outdoor or indoor weathered films was prepared in one go. After 48 hours air drying, one set of panels was tested for gloss, scratch hardness, impact resistance and flexibility. A major part of the remaining sets was exposed facing south at a 45° angle on racks 4 feet above the floor on the terrace of the laboratory building. The rest of the sets were exposed flat on racks in a laboratory at ambient temperature and humidity.

As with the painted panels, tests on weathered free films were carried out using coatings of each paint on tin foil backed with glass plates exposed outdoors and indoors after 48 hours air drying^{25,28,39}. One set of coatings was also exposed in a carbon arc weather-o-meter for accelerated weathering. The exposed surfaces were cleaned with care and the supported coating then placed in a mercury bath to amalgamate the tin substrate. In the case of coatings weathered over long periods, a special technique was used so that the free films were not damaged during handling in the various stages of testing.

Most coatings attained optimum properties during exposure periods of 2-4 months, hence the tests were terminated after a period a little over 6 months.

Results and discussion

Refs. 22, 25, 26, 30

For all the paint formulations, PVC was maintained at 30 per cent by volume, irrespective of the pigment extender ratio. It was kept at a relatively lower level than the CPVC²² (critical pigment volume concentration) so that the coatings retained some flexibility, which in turn facilitated the handling of free films after long exposure. The replacement of TiO₂ with barytes was done by volume per cent not by weight, so that the total PVC of the system remained unchanged. The data obtained from the tests conducted on painted panels and on free films before and after weathering are reported in tables 2-10.

Gloss retention and scratch hardness

The gloss values of all the painted panels were measured with a multi-angle glossmeter (Gardner Instruments Inc., USA) at a 45° angle having a standard gloss value of 52 at that angle. After 48 hours air drying, paints that had TiO₂ pigment replaced by barytes at a 10 per cent PVC level were found to have a slightly better gloss value, i.e. 56 compared to 51, than paints with TiO₂ only. However, this value decreases to 35 and 8 in the case of paints having TiO₂ replaced by 50 and 100 per cent barytes. During exterior weathering the decrease in gloss was fairly fast as after a month it declined to 26 from 51 and to 37 from 56 for paints containing 30 per cent PVC TiO₂ and 20 per cent PVC TiO₂ + 10 per cent PVC barytes, respectively. In interior weathering the gloss values of the same paints dropped to 32 and 37 after six months (Table 2 figures 1 and 2). These results showed that in 30 per cent PVC paints the replacement of TiO₂ by 10 per cent PVC

Table 2
Effect of weathering on gloss retention of paints
(gloss values of paints at 45° angle, standard value: 52)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
	Panels air dried for 48 hours							
	51	55	56	35				8
Panels weathered outdoors								
No. of days								
3	43	36	50	30				4
10	45	34	49	29				3
20	32	36	42	23				
30	26	31	37	17				2
50	16	18	22	14				
70	10	11	14	9				
90	6	6	8	5				1
120	4	4	6	4				
Panels weathered indoors								
15	50	56	57	39				3
60	45	50	52	30				
120	37	42	43	25				3
180	32	35	37	22				2

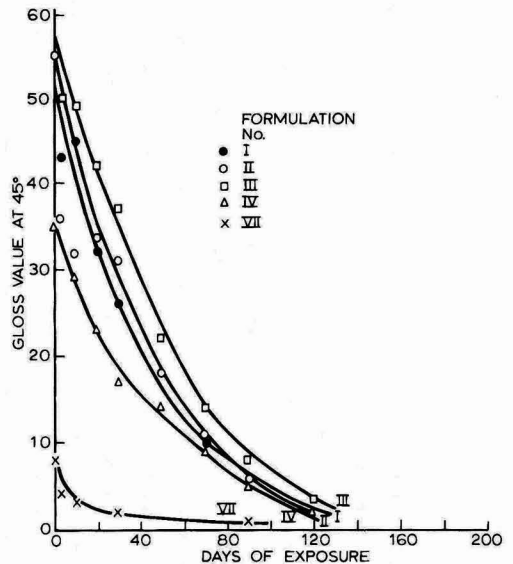


Figure 1. Effect of outdoor weathering on gloss retention of paints

of barytes improves their gloss retention both during exterior and interior weathering.

The scratch hardness tests conducted on panels air dried for 48 hours did not exhibit major differences in scratch hardness values for paints having part replacement of TiO₂ by barytes. For example, the scratch hardness values for paints containing 30 per cent PVC TiO₂ and those in which TiO₂ was replaced by 10 and 15 per cent

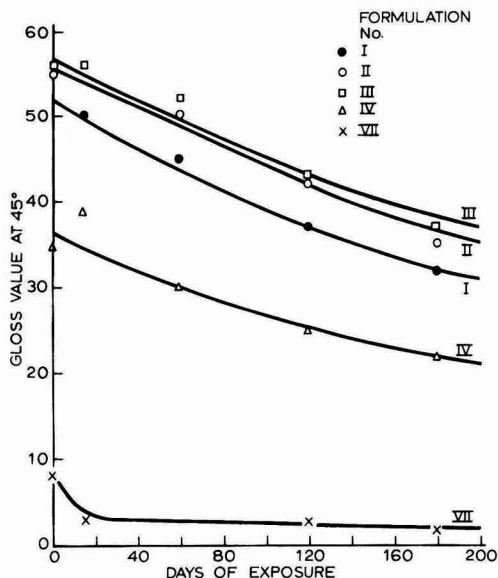


Figure 2. Effect of indoor weathering on gloss retention of paints

PVC barytes were the same, i.e. 0.3 kg. However, after five days outdoor weathering the values for these paints were 1.6, 1.4 and 0.8 kg, showing considerable differences due to replacement. After six months the same paints had scratch hardnesses of 1.3, 1.2 and 0.9 kg respectively. Similar behaviour in the scratch hardness values of these paints was observed in the course of indoor weathering (Table 3). The data on scratch hardness of these paints showed that the paint in which TiO₂ pigment was replaced by 10 per cent PVC barytes withstood the exterior and interior weathering better than paints of the other formulations tried. Data on the change in scratch hardness of

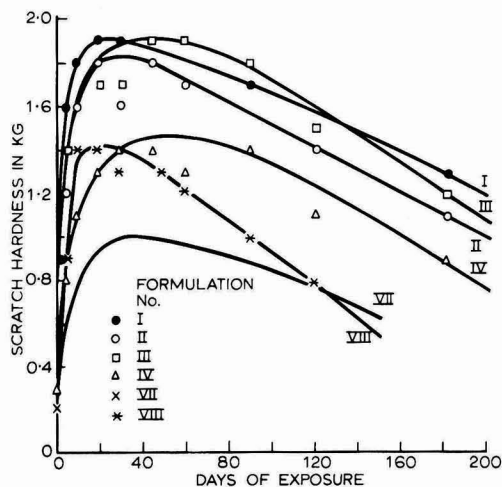


Figure 3. Effect of outdoor weathering on scratch hardness of paints

Table 3
Effect of weathering on scratch hardness of paints (scratch hardness of paints in Kg)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Panels air dried for 48 hours								
	0.3	0.25	0.3	0.3			0.2	0.5
Panels weathered outdoors								
No. of days								
3	0.9	0.7	0.8	0.5			0.4	0.8
5	1.6	1.2	1.4	0.8			0.6	0.9
10	1.8	1.6	1.6	1.1			0.7	1.4
20	1.9	1.8	1.7	1.3			1.0	1.4
30	1.9	1.6	1.7	1.4			1.0	1.3
45	1.8	1.8	1.9	1.4				1.3
60	1.6	1.7	1.9	1.3				1.2
90	1.9	1.8	1.8	1.4			0.8	1.0
120	1.5	1.4	1.6	1.1				0.8
180	1.3	1.1	1.2	0.9				
Panels weathered indoors								
No. of days								
3	0.6	0.5	0.5	0.45			0.3	0.7
5	0.8	0.7	0.7	0.6			0.4	0.8
10	1.1	1.0	1.1	0.9			0.6	1.0
20	1.6	1.4	1.5	1.2			0.7	1.25
30	1.9	1.5	1.7	1.2			0.8	1.4
45	2.1	1.8	1.9	1.4			1.0	1.5
60	2.1	2.0	2.0	1.6			1.0	1.5
90	2.3	2.1	2.0	1.6			1.2	1.5
120	2.3	2.0	2.1	1.5			1.2	1.3
180	2.1	1.8	2.0	1.4			1.0	1.2

weathered binders are also listed in Table 3 for relative assessment (figures 3 and 4).

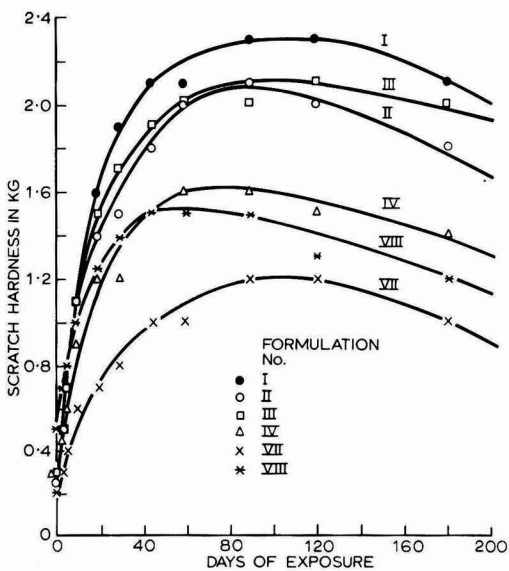


Figure 4. Effect of indoor weathering on scratch hardness of paints

Table 4
Effect of weathering on impact resistance of paints
(impact test by falling weight)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Panels air dried for 48 hours								
	SF ¹	SF	SF	SF			SF	P
Panels weathered outdoors								
No. of days								
5	F	F	F	F			F	P
10	CF	CF	CF	CF			F	P
20	CF	CF	CF	CF			CF	SF
30	CF	CF	CF	CF			CF	CF
45	CF	CF	CF	CF			CF	CF
Panels weathered indoors								
15	SF	SF	P	SF			SF	P
65	F	F	F	F				SF
90	F	F	F	F			F	F

P = pass, SF = slight failure, CF = complete failure, F = failed

Impact resistance and flexibility

Painted mild steel panels were air dried for 48 hours and then tested for resistance to the impact of a 10.5 lb falling weight (Sheen Instruments, UK). The coatings showed some signs of failure. After ten days outdoor weathering all of the coatings failed completely in the test except the binder (Table 4). The panels exposed indoors when tested after 60 days also showed complete failure. This test did not reveal any significant information on the effect of replacement. Similarly, the painted panels tested for flexibility by a conical mandrel passed the test after up to 90 days outdoor exposure (Table 5). As very little useful data was obtained from either of these tests during weathering, further tests were abandoned.

Tensile properties

Free films of some paints prepared after 48 hours air drying were found to be a little fragile so the duration of drying was raised from 48 to 96 hours before amalgamation of the tin substrate was carried out. The coatings on the tin foil were left for 24 hours in a mercury bath. The undersides of the free films, after removal from the mercury bath, were left to dry and gently cleaned after 24 hours. By the time tests on tensile properties were conducted, the free films had undergone air drying for a week. However, the coatings of paints and binder on tin foil backed by glass plates were exposed outdoors and indoors after just 48 hours air drying.

The data on the tensile strength of the unexposed films of these paints showed a gradual decrease in their strength with increase in barytes content. It was found that most paints attained optimum strength after outdoor exposure of two months and retained this strength a further two months. During indoor exposure, however, the coatings continued to gain strength even after six months. These results showed that 30 per cent PVC TiO₂ paints could tolerate replacement of TiO₂ by barytes by up to 10 per

Table 5
Effect of weathering on flexibility of paints
(flexibility test by conical mandrel)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Panels air dried for 48 hours								
	P	P	P	P			P	P
Panels weathered outdoors								
No. of days								
5	P	P	P	P			P	P
10	P	P	P	P			P	P
20	P	P	P	P			P	P
30	P	P	P	P			P	P
50	P	P	P	P			P	P
70	P	P	P	P			P	P
90	P	P	P	P			P	P
Panels weathered indoors								
15	P	P	P	P			P	P
65	P	P	P	P			P	P
90	P	P	P	P			P	P

P = pass

Table 6
Effect of weathering on tensile strength
(tensile strength of paint films in Kg/cm²)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Coatings air dried for one week								
	50	42	41	37	33	37	30	39
Coatings weathered outdoors								
No. of days								
2	59	50	54	44	37	38	35	40
5	78	63	67	49	48	48	47	52
10	78	64	67	55	59	66	57	59
20	86	72	80	61	66	79	65	66
30	93	79	84	64	68	105	79	75
45	99	86	95	76	83	86	81	101
60	115	92	103	87	87	86	80	120
90	121	91	104	82	60	52.5	60	135
120	107	79	93	82	40	50	35	155
190	65	53	64	61	40	45		170
Coatings weathered indoors								
5	47	45	40	33	37	40	27	38
10	53	53	46	39	46	47	35	47
20	62	59	51	45	50	54	44	48
45	77	66	60	48	52	65	44	55
75	72	66	65	55	58	64	50	63
120	76	67	75	56	65	73	60	76
190	87	73	86	66	72	82	60	90

cent PVC (Table 6). The test data on the properties of the binder indicated a gradual increase in its strength during the test period (figures 5 and 6).

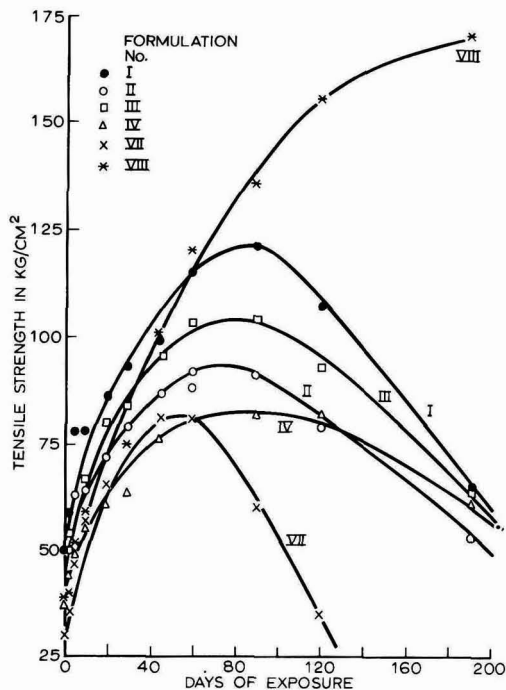


Figure 5. Effect of outdoor weathering on tensile strength of paint films

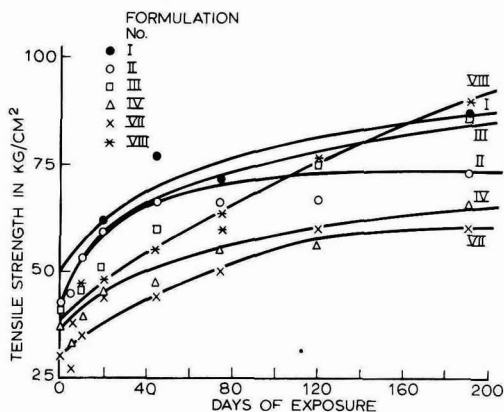


Figure 6. Effect of indoor weathering on tensile strength of paint films

The flexibility test results showed the percentage elongation of the binder, i.e. 124 was reduced to 38 when pigmented with 30 per cent PVC of TiO_2 . Moreover, the replacement of TiO_2 by barytes did not influence the flexibility of the air dried paint films. Their tensile strengths, on the other hand, decreased a little with increasing barytes content. However, the weathering test results did indicate an effect due to the replacement of TiO_2 by barytes on the flexibility of these paints. For example, the films of paints in which TiO_2 was replaced by

Table 7
Effect of weathering on flexibility of paints
(percentage elongation of paint film at break)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Coatings air dried for one week								
	38	35	34	37	36	36	35	124
Coatings weathered outdoors								
No. of days	32	42	30	34	36	27	33	114
2	30	46	36	36	24	26	30	110
5	26	34	32	28	20	20	24	104
10	25	28	30	25	16	14	18	102
20	21	20	22	18	14	12	13	82
30	16	12	14	10	8	6	6	57
45	10	8	10	6	5	4	4	36
60	6	5	6	4	2	2	2	21
90	4	2	2	2	shattered			11
120								4
190								4
Coatings weathered indoors								
5	40	36	36	38	30	28	40	129
10	40	32	34	36	26	28	34	132
20	38	32	32	32	24	24	28	140
45	38	32	32	28	23	22	20	120
75	36	28	28	24	20	20	18	105
120	34	28	28	22	19	16	18	88
190	28	26	22	20	17	14		69

50 per cent or more of barytes became relatively brittle after 45 days outdoor exposure. Similar behaviour was observed in coatings weathered indoors for over six months. The flexibility of the binder also decreased and its films became brittle after prolonged outdoor weathering (Table 7). The flexibility data also supported the limit of replacement of TiO_2 by barytes as being up to 10 per cent PVC.

Water absorption

The measurement of the amount of water absorbed by the films was conducted using two different methods. In the first method the well dried free film was placed flat over a perforated support just a little above the water surface in a chamber maintained at $30 \pm 1^\circ\text{C}$. This method ensured the relative humidity around the film was at about 100 per cent. The film was kept under these conditions for 96 hours as it was found earlier that it takes 48 hours to attain water absorption equilibrium in similar films³⁰. In the second method the film was left immersed in water for 96 hours. In both cases the weights of the films dried over anhydrous calcium chloride – after water absorption – was taken as the initial weights for calculating the percentage of water absorbed²⁵.

The amount of water absorbed by paint films placed over a water surface increased from 2.54 to 3.67 per cent where TiO_2 had been replaced by barytes (Table 8). In the case of the immersion method the amount of water absorbed by the paint films containing solely TiO_2 was only 2.67 per cent. However, the amount of water absorbed increased considerably when the paint film contained barytes. For example, the amount of water absorbed by

Table 8
Effect of weathering on water absorption
(percentage water absorption by paint films
exposed to 100% RH)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Coatings air dried for one week								
	2.54	3.07	3.18	3.05	3.33	3.65	3.67	2.22
Coatings weathered outdoors								
No. of days								
20	2.82	3.39	3.40	3.94	3.62	3.61	4.01	3.02
60	3.11	3.70	3.91	4.24	4.17	3.64	4.02	3.38
190	3.26	4.00	4.09	4.13				3.43
Coatings weathered indoors								
No. of days								
20	2.44	3.34	3.27	2.96	3.13	3.79	3.67	2.36
190	2.61		3.96	3.80	3.56			2.91

the paint film containing 30 per cent PVC barytes was 14.04 per cent, while the value for the binder film was 2.47 per cent using the same method. (Table 9.) The amounts of water absorbed by the weathered films of all the paints and the binder were more, relatively, when tested using the atmosphere saturated with water vapour method. But the amounts of water absorbed in paint films having TiO₂ replaced by barytes decreased with increasing periods of weathering when the tests were conducted using the immersion method (tables 8 and 9).

The increase in water absorption by the paint films having part TiO₂ replacement indicated that barytes in the paint film may have a tendency to form interaction bonds which become hydrated when in contact with liquid water or its vapour. This phenomenon was more obvious when paint films were immersed in water rather than being exposed to water vapour. However, this observation requires further study before conclusions are drawn. This phenomenon might be used to improve the properties of certain paints as the presence of a certain amount of water in the film acts as a plasticiser and helps the coating to withstand dimensional stresses. Hence, the part replacement of TiO₂ by barytes may improve the performance of TiO₂ containing paints in long term service, especially in hot and humid climatic conditions.

Water vapour permeation

The pigmentation of some binders usually increased the rate of permeation of water vapour through their films using the wet cup method²⁶. However, this varies from pigment to pigment as well as from binder to binder. For example, the rate of permeation through the air dried film of the binder was 3.68, whereas it was 5.68 and 3.00 respectively when the same binder was pigmented with TiO₂ and barytes. There was a gradual decrease in the rate of permeation with increasing barytes content in the paint films. The rate of permeation of water vapour through films of coatings weathered outdoors and indoors also decreased slightly over the period of weathering (Table 10).

The decrease in the rate of permeation with increasing barytes content in the film could be explained in the light

Table 9
Effect of weathering on water absorption
(percentage water absorption by paint films
immersed in water)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Coatings air dried for one week								
	2.67	8.32	9.53	12.62	13.78	13.39	14.04	2.47
Coatings weathered outdoors								
No. of days								
20	3.86	5.87	7.42	8.47	9.33	9.81	10.42	3.75
60	3.77	5.38	6.13	6.87	7.65	6.65		3.81
190	3.61	5.82	6.58	6.35				4.15
Coatings weathered indoors								
No. of days								
20	2.9	8.56	9.78	12.70	13.98	15.02	14.83	2.41
190	2.44		7.82	10.10	12.83	12.85		2.40

Table 10
Effect of weathering on water vapour permeation
(rate of water vapour permeation through paint film
in g/m²/h/1 mil)

Formulation No.	I	II	III	IV	V	VI	VII	VIII
Coatings air dried for one week								
	5.69	4.46	4.05	3.46	3.61	3.42	3.00	3.68
Coatings weathered outdoors								
No. of days								
20	4.60	3.56	2.98	2.60	2.64	2.37	2.41	3.31
60	3.44	2.91	2.35	2.25	2.36	2.18	1.98	3.05
120	2.93	2.41	2.05	2.14	2.22	2.28		2.45
Coatings weathered indoors								
No. of days								
20	4.91	4.01	3.27	3.31	3.04	3.15	2.65	3.69
75	4.19	3.24	2.74	2.64	2.53	2.71	2.49	3.06
190	3.51	2.70	2.51	2.36	2.21	2.40	1.99	2.78

of water absorption results. With the wet cup method the film was in contact with water vapour on both of its sides and it attains a state of dynamic water absorption equilibrium. Under such conditions the extender-resin system may become hydrated and tighten the packing arrangement in the film. Consequently, this may influence the movement of water molecules in the film under a vapour pressure differential. Subsequently the process of diffusion which promotes permeation slows down. It can be inferred from these results that the coatings that had part of their TiO₂ replaced by barytes may perform better than those paints containing only TiO₂ pigment.

Accelerated weathering

Accelerated weathering in a carbon arc weather-o-meter (Marr & Co. UK) for a period of over 100 hours caused

the free films of weathered coatings to become fairly brittle and hence this test was abandoned.

Conclusion

It has been found by earlier workers that some optical and physical properties of paints are improved when part of the TiO₂ pigment of the paint is replaced by extender. In the present study, the quantitative examination of various properties of coatings – air dried and un-weathered or weathered outdoors or indoors – showed that a coating containing 30 per cent PVC of TiO₂ pigment can have part of the TiO₂ pigment replaced by barytes to the extent of 10 per cent PVC without having an adverse influence on the properties of the coating.

The results indicate that the properties of the coating containing only barytes (extender) were much inferior to those of the binder alone, as well as to the coating in which only TiO₂ pigment was used.

This study is to be extended to cover various types of binders, pigments and extenders to achieve a better understanding of the factors influencing the interactions between binder-pigment and binder-extender.

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Styrene copolymerisation of isomerised tobacco seed (*Nicotiana tabacum*) oil and its alkyd

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Summary

Refined tobacco seed oil (*Nicotiana tabacum*) was isomerised and subsequently copolymerised with styrene monomer by a solvent method. Improvements in drying time and film properties were observed with copolymerised tobacco seed oil.

Styrenated alkyds were also prepared from the above

copolymerised oil by pre-styrenation and post-styrenation processes and their film properties were compared with those of tobacco seed oil modified alkyds. The alkyds prepared by the post-styrenation process gave better film properties than the alkyds prepared by the pre-styrenation process.

Keywords

Raw materials for coatings

oils

styrenated oil

binders (resins, etc.)

styrenated resin

Processes and methods primarily associated with

manufacturing or synthesis

copolymerization

La copolymérisation d'huile de graines de tabac (*Nicotiana tabacum*) isomérisée avec styrolène, et la résine alkyde dérivée du copolymère

Résumé

L'huile de graines de tabac (*Nicotiana tabacum*) raffinée était isomérisée et puis copolymérisée en solution avec styrolène. On a noté une amélioration, à la fois, de la vitesse de séchage et des caractéristiques de feuillet de l'huile de graines de tabac copolymérisée.

On a préparé d'ailleurs des alkydes styrénisées à partir de cette

huile copolymérisée par les procédés de pré- et de poststyrénation, et l'on a fait comparé leurs caractéristiques de feuillet auprès de celles des résines alkydes modifiées par l'huile de graines de tabac. La résine alkyde préparée par le procédé de poststyrénation rend de meilleures caractéristiques de feuillet que les résines alkydes préparées par le procédé de préstyrénation.

Die Copolymerisation des isomerierten Tabaksamenöls (*Nicotiana tabacum*) mit Styrol, und das auf diesem Copolymer gegründete Alkydharz

Zusammenfassung

Das raffinierte Tabaksamenöl wurde in Lösung isomerisiert und dann mit Styrol copolymerisiert. Eine Verbesserung der Trocknungszeit und der Filmeigenschaften wurden bemerkt.

Styrolisierte Alkydharze wurden auch von diesem copolymerisierten Öl durch die Vor- und Nach-

styrolierenverfahren hergestellt, und ihre Filmeigenschaften mit den der von Tabaksamenöl modifizierten Alkydharzen verglichen. Das von dem Nachstyrolierenverfahren hergestellten Alkydharz lieferte bessere Filmeigenschaften als die von dem Vorstyrolierenverfahren hergestellten Alkydharze.

Introduction

Refs. 1-13

The copolymerisation of oils with styrene has been known to impart fast drying properties to the oil as well as improved resistance to chemicals and water. Alkyds prepared from styrenated oils also show improved film properties. In order to obtain a clear and homogenous styrenated oil, the oil must have conjugated

unsaturation^{1,2}. Tung oil and styrene were successfully copolymerised by Peterson³ and Brunner and Tucker⁴. Dehydrated castor oil was styrenated by Bhow and Payne⁵, Hoogsten *et al.*⁶ and Crofts⁷. Non-conjugated drying and semi-drying oils have been styrenated after combining with oils having conjugated unsaturation. Rinse and Korf⁸ copolymerised linseed oil in combination with tung oil and dehydrated castor oil by mass and solvent polymerisation. Chand *et al.*⁹ copolymerised nigerseed oil in combination with tung oil, DCO and stand oil. Saxena

*et al.*¹⁰ copolymerised tobacco seed oil in combination with the above conjugated oils.

Isomerisation of the non-conjugated oil can also lead to conjugation. Terril¹¹ and Falkenburg *et al.*¹² suggested anthraquinone as a catalyst for isomerisation. Gupta *et al.*¹³ isomerised *Acacia arabica* oil using anthraquinone and successfully copolymerised the oil and its alkyd with styrene. Both the oil and styrenated alkyd were reported to give improved film characteristics.

The present paper reports a study of the styrene copolymerisation of isomerised tobacco seed oil and alkyd resin prepared from this oil.

Experimental

Materials used

Tobacco seed oil

Refined tobacco seed oil having the following characteristics was obtained from M/s Asian Paints, Bombay : acid value 1.1, iodine value (Wij's) 142.0, saponification value 190.0, refractive index (at 30°C) 1.4755, colour (Lovibond, 1" cell) 4Y + 3.5R, viscosity (at 30°C bubble tube) < 0.5 poise, and diene value 0.0.

Anthraquinone

AR grade, BDH, anthraquinone was used as a catalyst for the isomerisation of tobacco seed oil.

Styrene monomer

Extra-pure styrene of M/s Sisco Laboratories Pvt Ltd, Bombay was used for the styrenation of isomerised tobacco seed oil and its alkyds.

Benzoyl peroxide

LR grade, BDH, benzoyl peroxide was used as an initiator for the copolymerisation reaction.

Xylene

LR grade, BDH, sulfur free xylene having a boiling point range of 137-142°C was used as the solvent during copolymerisation.

Phthalic anhydride

Commercial phthalic anhydride of 95 per cent purity was used for the preparation of alkyds.

Glycerol

Commercial glycerol of 95 per cent purity was used in the alkyd preparations.

Calcium oxide

Commercial calcium oxide was used as the catalyst for monoglyceride preparation.

Isomerisation

Refined tobacco seed oil was isomerised using varying concentrations of anthraquinone and temperatures as shown in Table 1. During the isomerisation, a constant

flow of nitrogen was maintained to ensure an inert atmosphere. Samples were taken at intervals and examined for colour, acid number, viscosity and diene number.

Styrenation of tobacco seed oil

Tobacco seed oil, isomerised under optimised conditions, was styrenated with varying ratios of styrene to oil and with 1 and 3 per cent benzoyl peroxide on the weight of oil as the initiator; a solution polymerisation method using xylene as the solvent was employed. The ingredients were refluxed at 140°C for 24 hours. The viscosity, colour, acid number and clarity of the reaction mixture was checked at intervals in order to follow the progress of the reaction. Finally, the xylene was distilled off and the last traces were removed by heating under vacuum.

Preparation of styrenated alkyds

Styrenated alkyds were prepared by pre-styrenation as well as by post-styrenation, as described below.

Pre-styrenation process

Styrenated oil (55 parts) and glycerol (16 parts) were placed in a three-necked flask and heated to 180°C. Calcium oxide (0.2 per cent by weight of oil) was added as a catalyst and the temperature raised to 230°C. The monoglyceride formation was periodically checked using the methyl alcohol test and after the formation of monoglycerides, phthalic anhydride (44 parts) and an additional quantity of glycerol (5 parts) were added slowly. Esterification was continued at 240°C till a clear product was formed. The prepared alkyd resin had an acid number of 8.6.

Post-styrenation process

Tobacco seed oil alkyd of medium oil length was prepared by the monoglyceride process described above using un-styrenated oil. The alkyd had an acid number of 8.1.

The above alkyd (100 parts) and styrene (24 parts) were dissolved in xylene (124 parts) in a three-necked flask. Benzoyl peroxide initiator (3 per cent by weight of oil in the alkyd) was then added. The ingredients were refluxed at 140°C for 24 hours. After styrenation, xylene was distilled off and the last traces of it removed under vacuum. The alkyd had an acid value of 9.5.

The styrenated alkyd films (air dried as well as baked) were compared with the un-styrenated alkyd films with respect to scratch hardness, flexibility, and resistance to water, acid and alkali.

Results and discussion

The details of the experiments conducted for the isomerisation of tobacco seed oil are given in Table 1. The details of the experiments conducted to establish the optimum conditions of styrene copolymerisation of isomerised tobacco seed oil are given in Table 2. The film characteristics of the styrenated oil and styrenated alkyds in relation to their un-styrenated counterparts are shown in Table 3.

Isomerisation

The isomerisation of refined tobacco seed oil was carried

Table 1
Isomerisation of refined tobacco seed oil

Serial No.	Oil sample	Lovibond colour (1" cell)	Acid number	Viscosity at 30°C (poise)	Diene number
1.	Un-isomerised oil	4Y+3.5R	1.1	0.5	0.0
2.	Isomerised with 3 per cent anthraquinone at 270°C				
	0.5 h	10Y+3.7R	1.8	2.0	6.5
	1.0 h	13Y+4.0R	2.6	2.0	8.1
	1.5 h	16Y+5.3R	2.7	3.0	7.4
	2.0 h	20Y+5.5R	2.9	7.0	6.4
3.	Isomerised with 4 per cent anthraquinone at 270°C				
	0.5 h	10Y+3.7R	1.8	2.0	7.4
	1.0 h	12Y+4.0R	2.5	2.0	9.3
	1.5 h	15Y+5.0R	2.7	3.0	8.9
	2.0 h	20Y+5.5R	2.9	7.0	8.4
4.	Isomerised with 5 per cent anthraquinone at 270°C				
	0.5 h	10Y+3.8R	1.8	2.0	12.5
	1.0 h	11Y+4.5R	2.4	2.0	14.7
	1.5 h	16Y+5.5R	2.7	3.0	13.7
	2.0 h	19Y+6.0R	2.9	7.0	12.2
5.	Isomerised with 5 per cent anthraquinone at 260°C				
	0.5 h	8Y+3.7R	1.8	2.0	11.7
	1.0 h	9Y+3.5R	2.4	2.0	12.9
	1.5 h	12Y+4.0R	2.6	2.0	13.3
	2.0 h	14Y+5.0R	2.8	3.0	13.9
6.	Isomerised with 5 per cent anthraquinone at 280°C				
	0.5 h	14Y+5.4R	2.0	2.0	11.9
	1.0 h	15Y+6.5R	2.7	3.0	9.9
	1.5 h	17Y+8.0R	3.0	7.0	8.1
	2.0 h	26Y+14R	3.3	25.0	5.3

out in the presence of an anthraquinone catalyst at three levels of catalyst concentration, viz. 3, 4 and 5 per cent, and at 270°C. The isomerisation was virtually complete within the first hour of the reaction in all the three cases. Heating for more than one hour led to significant increases in viscosity accompanied by increases in colour and acid number and a simultaneous decrease in the diene value of the isomerised oil, which indicated the possibility of polymerisation during the course of heating. The best results of isomerisation were obtained with 5 per cent anthraquinone (serial No. 4, Table 1) in which a diene value of 14.7 was obtained in one hour.

Heating at this catalyst concentration at 260°C and at 280°C did not show any increase in diene value of the oil. At 260°C, however, the tendency to polymerisation was not so evident. The rise in viscosity and colour of the oil was also not significant. However, at 280°C the tendency to polymerisation was very significant as is evident from

the viscosity data and low diene values under these conditions. The increase in colour and acid value was also significant.

Reaction parameters for serial No. 4 thus appear optimum for the isomerisation of tobacco seed oil. Hence the oil isomerised at 270°C in the presence of 5 per cent anthraquinone for 1 hour was used for further experiments in this study.

Styrenation

Three different ratios of styrene, oil and xylene, viz. 1:1:2, 2:3:5 and 3:7:10, were used for conducting experiments on styrene copolymerisation. Benzoyl peroxide (3 per cent by weight of oil) was used as the initiator for copolymerisation.

At a styrene, oil and xylene ratio of 1:1:2, 80 per cent of

Table 2
Styrene copolymerisation of tobacco seed oil
by solvent method at 140°C with benzoyl peroxide initiator
(3 per cent by weight of oil)

Sample No.	Styrene/oil/xylene ratio	Reaction time (h)	Styrene reacted (per cent)	Lovibond colour of reaction mixture (1" cell)	Viscosity of reaction mixture by Ford cup No. 4 at 37°C (second)	Clarity
A-0	1:1:2	0	0.0	3.0Y+2.4R	15.0	hazy
A-1	1:1:2	4	80.0	3.0Y+2.5R	15.2	hazy
A-2	1:1:2	8	83.0	3.0Y+2.7R	16.0	hazy
A-3	1:1:2	12	86.0	3.5Y+2.8R	16.8	hazy
A-4	1:1:2	16	90.0	3.5Y+3.0R	18.0	hazy
A-5	1:1:2	20	94.0	4.0Y+3.0R	19.2	hazy
A-6	1:1:2	24	95.0	4.0Y+3.1R	19.6	hazy
B-0	2:3:5	0	0.0	3.0Y+2.4R	15.2	hazy
B-1	2:3:5	4	75.0	3.1Y+2.4R	15.4	hazy
B-2	2:3:5	8	78.0	3.2Y+2.6R	15.5	hazy
B-3	2:3:5	12	80.0	3.5Y+2.8R	15.9	hazy
B-4	2:3:5	16	83.0	3.5Y+3.0R	16.5	hazy
B-5	2:3:5	20	85.0	4.0Y+3.1R	17.6	hazy
B-6	2:3:5	24	87.0	4.0Y+3.3R	19.2	hazy
C-0	3:7:10	0	0.0	3.0Y+2.4R	15.5	clear
C-1	3:7:10	4	62.2	3.0Y+2.5R	15.5	clear
C-2	3:7:10	8	70.0	3.3Y+3.0R	16.9	clear
C-3	3:7:10	12	77.3	3.5Y+3.4R	18.6	clear
C-4	3:7:10	16	82.5	3.5Y+3.8R	20.0	clear
C-5	3:7:10	20	86.5	3.8Y+4.2R	21.2	clear
C-6	3:7:10	24	89.0	4.0Y+4.5R	22.6	clear

the styrene had reacted in the first four hours but it took a further 20 hours to react 95 per cent of the styrene in all during the copolymerisation. The increase in the colour of the reaction mixture during copolymerisation was not significant nor was there any significant increase in the viscosity of the reaction mixture. The product obtained was hazy even after 95 per cent of the styrene had reacted.

At a styrene, oil and xylene ratio of 2:3:5, the rate of styrenation was slower than in the earlier case where a styrene, oil and xylene ratio of 1:1:2 was used. Seventy-five per cent of the styrene had reacted in the first four hours and after 24 hours only 87 per cent of the styrene had reacted. Here also, no significant differences were observed in the colour and viscosity of the reaction mixture, although both of these characteristics showed gradual increase. The product obtained even after 24 hours was hazy and not suitable for use in coatings.

At a styrene, oil and xylene ratio of 3:7:10, the reaction was slower in the initial stages: only 62.2 per cent of the styrene had reacted in the first four hours. However, 89 per cent of the styrene had reacted after 24 hours. The most significant observation with this set of experiments was that the reaction product had no haziness even in the initial stages of copolymerisation. The viscosity obtained towards the end of the reaction was also higher than in the two previous cases. However, no significant difference was observed in the increase in colour of the reaction mixture compared to the earlier two examples.

From the above results, two main conclusions can be drawn. At higher ratios of styrene to oil, as in set A, the reaction was faster, the product was hazy, and even though 95 per cent of the styrene had polymerised, the

increase in the viscosity was only 4.6 seconds, which indicates a greater degree of homopolymerisation of styrene leading to the formation of a styrene polymer incompatible with the oil. The styrenated product remained hazy even in set B, where the ratio of styrene to oil was decreased to 2:3. However, upon further reduction of the ratio of styrene to oil to 3:7, as in set C, a significant improvement in the product was observed indicating that a copolymer was being formed. Further, even though only 89 per cent of the styrene had reacted after 24 hours (comparable to 95 per cent in the experiments of set A), the increase in the viscosity was 7.1 seconds, indicating more copolymerisation than homopolymerisation.

Hence, the conditions for set C were found to be optimum and therefore all the styrenated samples prepared for the study were made as those in set C-6.

Drying characteristics

The oil and alkyd samples were applied on to metallic panels after thinning with MTO where necessary. Samples I to VI were air dried whereas samples VII to X were dried by baking for 30 minutes at 120°C. Among the air dried samples the fastest drying was sample VI, a post-styrenated alkyd; the second was sample V, a pre-styrenated alkyd. The alkyds dried comparatively much faster than the corresponding oils. The drying time for styrenated tobacco seed oil was quite fast, being quite close to un-styrenated alkyd with respect to surface drying. However, the styrenated oil took a longer time to achieve hard drying and tack-free drying compared to the alkyds. Considerable differences in the drying patterns of isomerised and un-isomerised tobacco seed oils were also observed. The results clearly indicate that the drying time

Table 3
 Film characteristics of styrenated tobacco seed oil
 and alkyds in relation to un-styrenated products

Sample No.	Material	Mode of drying	Drying time (h)			Flexibility ($\frac{1}{4}$ " mandrel)	Stripping test	Scratch hardness	Water resistance (48 h)	Acid resistance (2% H ₂ SO ₄ 24 h)	Alkali resistance (2% NaOH 3 h)
			Surface dry	Hard dry	Tack free						
I	Refined tobacco seed oil	air dried	8	20	tacky after 48 h	no visible damage to the film	scratches free from jagged edges	0.6	2	2	0
II	Isomerised tobacco seed oil	air dried	4	12	42	no visible damage to the film	scratches free from jagged edges	0.6	3	3	0
III	Styrenated tobacco seed oil (C-6)	air dried	2.5	8	40	no visible damage to the film	scratches free from jagged edges	1.8	4	4	2
IV	Tobacco seed oil alkyd (prepared from isomerised oil)	air dried	2	4	18	no visible damage to the film	scratches free from jagged edges	2.6	3	3	2
V	Pre-styrenated tobacco seed oil alkyd	air dried	0.25	2	8	no visible damage to the film	scratches free from jagged edges	3.0	4	4	3
VI	Post-styrenated tobacco seed oil alkyd	air dried	0.25	1.5	6	no visible damage to the film	scratches free from jagged edges	3.2	5	5	4
VII	Styrenated tobacco seed oil (C-6)	baked for 30 min. at 120°C				no visible damage to the film	scratches free from jagged edges	2.2	5	4	3
VIII	Tobacco seed oil alkyd (prepared from isomerised oil)	baked for 30 min. at 120°C				no visible damage to the film	scratches free from jagged edges	2.8	3	3	2
IX	Pre-styrenated tobacco seed oil alkyd	baked for 30 min. at 120°C				no visible damage to the film	scratches free from jagged edges	3.2	5	5	4
X	Post-styrenated tobacco seed oil alkyd	baked for 30 min. at 120°C				no visible damage to the film	scratches free from jagged edges	3.5	5	5	5

5 = Unaffected, 4 = Slight loss in gloss, 3 = Loss in gloss, 2 = Film partially cracked, 1 = Film cracked and partially removed, 0 = Film completely removed

improves considerably after isomerisation and still more after styrenation. The alkyds, however, dry much faster, the fastest drying being the styrenated alkyds.

Flexibility and adhesion

All the samples passed the flexibility test on a quarter inch mandrel, which was expected for oil-based coatings. Similarly all the samples passed the stripping test for adhesion.

Scratch hardness

There were considerable differences in the scratch hardness of the films. The styrenated oil films were significantly harder than the un-styrenated oil films. Between the air dried and baked styrenated oil films (III and VII in

Table 3), the baked films showed greater scratch hardness. The highest scratch hardness was shown by the alkyd films. The styrenated alkyd films had greater scratch hardness than the un-styrenated alkyd films and baking significantly improved the scratch hardness of the films. Between the styrenated samples, post-styrenated alkyd film showed improved hardness on the pre-styrenated alkyd.

Water resistance

The water resistance of the un-styrenated oil and alkyd films was inferior to the styrenated oil and alkyd films. Baking in place of air drying slightly improved the water resistance of the films. The water resistance of post-styrenated alkyd films was superior to that of all the other samples examined.

Acid resistance

The acid resistance of the films under study showed exactly the same pattern as observed in the case of water resistance, the un-styrenated oil and alkyd films showing poor acid resistance and the styrenated oil and alkyd films showing improved resistance. In the case of acid resistance, no significant advantage appeared to be gained by baking instead of air drying. The post-styrenated alkyd films showed improved acid resistance as compared to pre-styrenated alkyd or styrenated oil films.

Alkali resistance

The alkali resistance of un-styrenated oil films was extremely poor as the films of samples I and II were completely destroyed on alkali treatment. The styrenated oil films showed better resistance than the un-styrenated oil films, which further improved if they were baked instead of air dried. The alkali resistance of the styrenated oil films was comparable to that of the alkyd resin prepared from isomerised tobacco seed oil. No improvement in the alkali resistance of this alkyd was observed by baking the sample instead of air drying. The styrenated alkyd films showed considerable improvements in alkali resistance, particularly when the films were baked. The post-styrenated tobacco seed oil alkyd film dried by baking showed high alkali resistance. This is surprising since alkyd films are known to have poor alkali resistance due to the ester linkages in the resin molecule.

Conclusion

The present study indicates a potential use for styrenated alkyds, particularly post-styrenated alkyds, in surface coatings requiring improved resistance to water and

chemicals. The styrenated alkyd of tobacco seed oil in particular can produce high class non-yellowing fast drying films. Isomerisation in the presence of anthraquinone is potentially a useful method for improving the conjugation in the oil for styrenation and for other general purpose binders.

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Next month's issue

The Honorary Editor has accepted the following papers for publication. They are expected to appear in the December issue:

Stability and flocculation in latex paint. Part 1: the influence of dispersants on particle stability by *M. J. Jaycock and G. J. Kayem*

Organosilanes as adhesion promoters for organic coatings. Part 2: silanes in the paints by *P. Walker*

The future of the refinishing industry by *A. D. White*

Organosilanes as adhesion promoters for organic coatings. Part 1: silanes on the metal surface

By P. Walker

Atomic Weapons Research Establishment, Building SB43, Aldermaston, Reading RG7 4PR, England

Summary

A range of organo-functional silanes of the general formula $R-Si(OR)_3$ have been examined as adhesion promoters for urethane and epoxide paints on aluminium and mild steel substrates when used as pre-treatment primers. Torque shear and direct pull-off adhesion tests have shown that dramatic improve-

ments in the initial adhesion to degreased and grit blasted metal substrates can be obtained, and this improvement is maintained after exposure to cyclic humidity and accelerated weathering cycles.

Keywords

Types and classes of coatings and allied products

epoxy coating
urethane finish

Raw materials for coatings

miscellaneous paint additives

adhesion promoter

Properties, characteristics and conditions primarily associated with

dried or cured films

adhesion

Les organosilanes en tant que promoteurs d'adhérence en revêtements organiques 1^{ère} Partie

Résumé

On a étudié une gamme de silanes organo-fonctionnels de formule générale, $R-Si(OR)_3$, en tant que promoteurs d'adhérence en peintures uréthanes et époxydes utilisés comme primaires de prétraitement pour subjectiles en aluminium ou en acier doux. Les essais d'adhérence par

arrachage et de torsion ont montré que l'adhérence initiale aux subjectiles dégraissés et nettoyés par sablage peuvent être fortement améliorée et que cette amélioration persiste après l'exposition aux cycles d'humidité et de vieillissement accéléré.

Die Organosilane als Haftvermittler für organische Beschichtungen. Erster Teil

Zusammenfassung

Als Haftvermittler für Urethan- oder Epoxidlacke, die als Vorbehandlungsgrundierungsmittel auf Substraten aus Aluminium und niedriggekohlten Stahl benutzt wurden, wurden eine Reihe organofunktioneller Silane der allgemeinen Formel $R-Si(OR)_3$ untersucht. Haftestigkeitsversuche mittels Abreiss- und

Drehmomentmethoden zeigten, dass die Anfangshaftung an entfetteten und sandgestrahlten Substraten stark verbessert werden kann, und dass diese Verbesserung nach Auslegung zu zyklischer Feuchtigkeit und Schnellbewitterung bleibt.

Introduction

Refs. 1-20

The importance of adhesion in the science and technology of organic surface coatings is well recognised. It is reflected in many literature references to methods of measuring this property¹⁻³, achieving good adhesion by surface preparation, and its importance in obtaining good surface protection^{4,7}.

The effects of hostile environments on paint adhesion have also been studied⁸⁻¹⁰. Much less work has been

reported in the surface coatings literature on the use of "adhesion promoters" although this aspect of technology has received considerable attention in the literature of other technologies.

Adhesion promoters investigated include electrodeposited composite coatings¹¹, tannins¹², poly-n-isopropyl acrylamide¹³, epoxidised organosilicon compounds¹⁴, organophosphonic esters¹⁵, imidazoline compounds¹⁶, titanates¹⁷, and silanes¹⁸. The latter have been investigated over a period of 20 years, initially by workers in the reinforced plastics field and more recently in adhesive technology. However, very little work has been

reported on the use of silanes with organic surface coatings. Work by the Cleveland Society has indicated an improvement in the initial bond strength of a vinyl-based coating using *n*-beta(aminoethyl)aminopropyl trimethoxysilane as an adhesion promoter¹⁹. A recent paper by Walker²⁰ has described the results of a reasonably intensive programme of evaluation of silanes used in conjunction with two-pack epoxide and polyurethane coatings.

Improvements in the initial bond strength of a coating to a metallic substrate and its "wet" adhesion after exposure to a hostile environment would be of great interest to the surface coatings and metal finishing manufacturing industries alike, particularly if improvements could be gained on surfaces given a minimum pretreatment or on metals normally considered to be difficult to paint. The present paper describes other aspects of the work previously described²⁰ on the use of organosilanes as adhesion promoters for epoxide and polyurethane coatings applied to metallic substrates. Further papers will cover the incorporation of silanes into paints and adhesion measurements on cadmium, lead and zinc.

Experimental

Refs. 1, 2, 20-22

Paints

The work reported was carried out on single coats of a two-pack polyamide cured epoxide and on aliphatic isocyanate adduct cured polyester paint both pigmented with rutile titanium dioxide and conforming to DTD 5555 A and DTD 5580.

Silanes

Five organosilanes were examined:

γ -methacryloxy propyltrimethylsilane	A174
β -(3, 4-epoxycyclohexyl)ethyltrimethoxysilane	A186
γ -glycidoxy propyltrimethoxysilane	A187
γ -mercaptropropyl trimethoxysilane	A189
<i>n</i> -beta aminoethyl aminopropyltrimethoxysilane	A1120

All supplied by the Union Carbide Corporation.

Substrates

The work reported was carried out on mild steel panels to BS 1449, Steel Plate, Part 1B, CR3/FF and aluminium 99.9 per cent pure to BS 1470, Grade SIC-H8.

Panel preparation

All panels, each 150 × 100 mm (6 × 4 in), were cut from the same batch of sheet stock and individually identified. All panels were degreased to the dictates of BS 3900, section A3, and those selected for grit blasting were brushed to remove particulate matter and re-degreased on completion of this operation. These pretreatment operations were performed immediately prior to coating. Both paints were applied by spray as a single coat at the dry film weights recommended by the appropriate specification and allowed to cure at room temperature and humidity for 14 days prior to testing.

Adhesion tests

Direct pull-off

Four squares, 31 × 31 mm ($1\frac{1}{4} \times 1\frac{1}{4}$ in), were cut from each test panel, avoiding the panel edges, and sandwiched between cylindrical test specimens using a two-pack cold curing polyamide cured epoxide adhesive (Versamid 115/Epikote 828 50/50 %/w). After 24 hours in an alignment jig under a pressure of 45N (10 psi), the resulting doublets were broken on a Hounsfield Tensometer¹ at a cross-head speed of 2 mm/min ($1/16$ in/min).

Torque shear test

Five cylindrical specimens of known diameter were bonded to the surface of the test panel after lightly abrading the paint surface. The adhesive previously described was used after curing for 24 hours. The specimens were twisted off using a recording torque spanner².

Environmental tests

Three test environments were used.

Humidity under condensation conditions

This exposure was carried out to the dictates of BS 3900, Method F2, in which the panel is subjected to humidity under condensation conditions cycling between 42 and 48°C. The period of exposure was 500 or 1000 hours.

Accelerated weathering

This exposure was carried out to the dictates of BS 3900, Method F3, in which the panel is subjected to the radiation from a 1600 watt enclosed carbon arc with water spray. A fuller description of the total cycle may be obtained by reference to the original specification²¹. The period of exposure was 1000 hours.

Water immersion

Total immersion in distilled water for 500 hours.

Recording of results

In recording the results obtained from the adhesion tests, the average failing load and approximate area of detachment has been recorded. Where the detached area differed widely between individual test specimens, the range has been recorded.

Silane adhesion promoters

The silane adhesion promoters (coupling agents) used in this investigation were all monomeric species of the general formula R-Si(OR)³, where R represents a group capable of reaction with a polymer matrix and (OR)³ is a hydrolysable ester group. The chemical compositions of the silanes investigated are shown in Appendix 1 to this paper.

The mechanisms by which the silanes function as adhesion promoters to glass and metal substrates have attracted wide attention and many theories have been proposed. A discussion of these is beyond the scope of this present paper but an excellent review article has been



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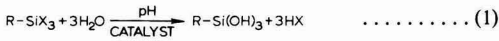
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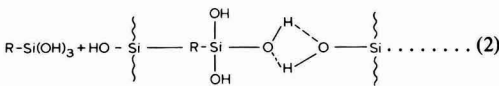
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written by Rosen²². However, the reactions of interest may be summarised as:

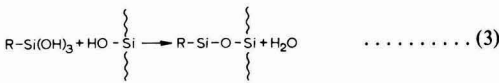
1. Hydrolysis of ester group



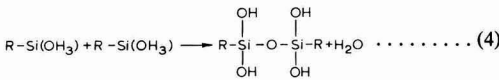
2. Hydrogen bonding of surface



3. Condensation with the surface



4. Polymerisation



5. Reaction with polymer matrix. These are potentially many and varied and will be discussed as appropriate.

Method of applying silane

To determine the effect of the method of application of the silane used as a pretreatment primer on paint adhesion, silanes A 189 and A 1120 were applied to degreased aluminium panels as 2 per cent %_w solutions in dry methyl ethyl ketone, dry petroleum ether and an 80 : 20 %_w mixture of ethyl alcohol and water. Some of the panels treated with silane applied from dry methyl ethyl ketone were given a water rinse after application of the silane. All panels were allowed to dry in the laboratory for two hours before application of the paint. Panels were tested after 14 days and after water immersion for 500 hours. The results obtained from the panels treated with n-beta aminoethyl aminopropyl-trimethoxysilane are shown in Table 1. Reference to Table 1 will show that the use of the silane

materially improved the bond strength of the polyurethane paint to degreased aluminium when tested by both the torque shear and direct pull-off techniques. All four methods gave higher failing loads initially but after water immersion the bond strengths (with one exception) showed a marked fall. The panels treated with silane applied from dry solutions and not subsequently water rinsed showed considerably less residual adhesion than those in which water was present either in the original solution or in the form of a rinse. Similar results were obtained from the use of γ-mercaptopropyl trimethoxysilane in that both the initial bond strengths and bond strengths after water immersion were higher than the non-silane control. The data suggested that the dry MEK/water rinse method gave the highest initial bond strengths and this method was adopted for the subsequent tests, although it was apparent that the acetone/water mixture method was only slightly inferior. In fact the latter method offered the advantage of being a one step process.

Effect of silane type on bond strength

Polyurethane paint on aluminium

Table 2 shows the bond strengths achieved on aluminium substrates given either a simple solvent degrease without abrasion or a grit blast pretreatment prior to application of a range of silanes. It can be seen that, as measured by the torque shear technique, all the bond strengths on the silane treated panels were greater than on the non-silane control. In fact the bond strengths on some of the degreased panels were nearly as great as on the grit blasted non-silane panel. In several cases there was no detachment of the paint from the substrate and the values recorded represent the minimum adhesion of the paint. The direct pull-off results show a similar pattern and although lower in value clearly indicate that in general the silanes improve the measured bond strength, although there were two cases in which a silane treated panel showed a bond strength lower than that of the control. Of the five silanes tested γ-methacryloxy propyltrimethoxysilane and n-beta aminoethyl aminopropyltrimethoxysilane gave the highest bond strengths.

Polyurethane paint on mild steel

Table 3 shows the data from polyurethane paint applied to silane treated surfaces. Again it can be seen that the bond strengths on the treated panels, as measured by the torque shear technique, were considerably greater than on the non-silane control panel both on the degreased and grit

Table 1
Effect of method of application of silane on the bond strength of a polyurethane paint (degreased aluminium) (silane A1120)

Method of application	Torque shear adhesion						Direct pull-off adhesion					
	Initial failing load			After 500 h water immersion			Initial failing load			After 500 h water immersion		
	MPa	psi	Area of detachment (%)	MPa	psi	Area of detachment (%)	MPa	psi	Area of detachment (%)	MPa	psi	Area of detachment (%)
No silane applied	32.4	4680	100	10.1	1450	100	12.8	1820	100	4.1	590	100
Dry methyl ethyl ketone	36.5	5300	0-100	15.3	2200	100	23.0	3340	0-90	8.4	1220	30-100
Dry petroleum ether	40.4	5860	0-30	25.6	3670	100	25.9	3760	60-100	11.2	1630	50-90
Dry methyl ethyl ketone/water wash	47.4	6880	0	41.8	6000	0-50	22.0	3190	45	25.8	3700	80
Acetone/water solution	39.3	5700	0-60	44.9	6450	20	21.3	3060	60-100	19.4	2780	90

Table 2
Effect of silane type on bond strength – polyurethane paint on aluminium

Silane	Surface preparation	Torque shear			Direct pull-off		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment
None	Degreased	15.8	2300	100	11.4	1660	100
None	Abraded	28.4	4130	10-20	29.4	4270	30-80
A 174	Degreased	41.5	6030	0	33.1	4800	0-60
A 186	Degreased	21.0	3050	100	14.7	2140	100
A 187	Degreased	24.4	3540	60-100	10.1	1470	100
A 189	Degreased	34.4	4990	0-80	19.8	2880	100
A 1120	Degreased	40.3	5850	0	23.2	3370	5-50
None	Grit blasted	45.8	6650	0-20	27.6	4000	10
A 174	Grit blasted	48.9	7100	0	26.9	3900	10
A 186	Grit blasted	46.6	6770	0-10	31.9	4630	0
A 187	Grit blasted	48.2	7000	0	29.1	4220	0
A 189	Grit blasted	46.7	6780	0-5	31.2	4530	0
A 1120	Grit blasted	49.6	7200	0	33.2	4820	0

Table 3
Effect of silane type on bond strength – polyurethane paint on mild steel

Silane	Surface preparation	Torque shear			Direct pull-off		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment
None	Degreased	22.6	3280	20-100	15.4	2230	100
None	Abraded	37.0	5370	5-30	28.2	4010	30
A 174	Degreased	38.7	5620	0-30	24.4	3550	0-80
A 186	Degreased	37.7	5470	100	12.3	1790	100
A 187	Degreased	47.1	6830	0	24.2	3520	40-100
A 189	Degreased	36.4	5280	0-10	23.6	3430	20-40
A 1120	Degreased	48.0	6970	0	29.6	4300	0-5
None	Grit blasted	40.0	5800	5-10	35.8	5200	10-40
A 174	Grit blasted	45.8	6650	0-20	34.6	5020	0-90
A 186	Grit blasted	46.6	6770	0-10	29.1	4230	20-90
A 187	Grit blasted	48.2	7000	0	31.6	4590	50-70
A 189	Grit blasted	46.7	6780	0-5	34.0	4930	0-90
A 1120	Grit blasted	49.6	7200	0	39.9	5800	0

blasted panels. The direct pull-off values followed a similar pattern with the marked exception of those for the β -(3,4-epoxycyclohexyl)ethyltrimethoxysilane, which were lower on both the degreased and grit blasted panels.

Epoxide paint on aluminium

Table 4 shows the data obtained from the epoxide paint applied to aluminium panels. The torque shear values from the silane treated panels are invariably greater than for the non-silane controls and in all cases the values are greater than those for the grit blasted non-silane control. The direct pull-off data is not in such close agreement with the torque shear data for the polyurethane paint, but the values are close and are not in serious conflict. A considerable improvement in bond strength is indicated.

Epoxide paint on mild steel

Table 5 shows the data for epoxide paint applied to mild steel panels. On the degreased only panels the torque shear values show that three of the five silanes resulted in an improvement in bond strength with an apparent anomaly in the results for the two control panels, where the abraded panel shows a lower bond strength than the degreased panel. All the other values are greater. Again a considerable improvement in bond strength is indicated.

Effect of environment on bond strength

Paints on aluminium

In this series of tests the coated panels were subjected to

Table 4
Effect of silane type on bond strength – epoxide paint on aluminium

Silane	Surface preparation	Torque shear			Direct pull-off		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment
None	Degreased	27.9	4050	30-90	20.3	2950	50-90
None	Abraded	29.2	4250	5-80	17.4	2530	50-80
A 174	Degreased	42.2	6130	0	30.8	4470	0
A 186	Degreased	44.2	6420	0	18.1	2630	30-70
A 187	Degreased	43.9	6370	0-10	18.0	2610	10-90
A 189	Degreased	42.2	6130	0	22.7	3300	20-50
A 1120	Degreased	41.5	6030	0	29.7	4310	0-5
None	Grit blasted	40.0	5800	10-30	27.6	4000	0
A 174	Grit blasted	43.5	6320	10	29.2	4240	0
A 186	Grit blasted	47.1	6840	0	31.2	4530	0
A 187	Grit blasted	42.4	6160	0	32.2	4680	0
A 189	Grit blasted	43.9	6380	0	32.7	4750	0
A 1120	Grit blasted	45.8	6650	0	33.6	4880	0

Table 5
Effect of silane type on bond strength – epoxide paint on mild steel

Silane	Surface preparation	Torque shear			Direct pull-off		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment
None	Degreased	36.2	5260	100	19.5	2830	20-40
None	Abraded	32.8	4760	80	23.2	3370	10-30
A 174	Degreased	39.7	5770	10-30	27.6	4000	30
A 186	Degreased	36.2	5250	0-20	26.6	3860	0-20
A 187	Degreased	35.7	5180	0-20	23.2	3370	0-50
A 189	Degreased	42.8	6220	0	22.7	3290	10-60
A 1120	Degreased	45.0	6530	0	30.6	4440	0
None	Grit blasted	40.0	5800	10-30	25.9	3760	30
A 174	Grit blasted	42.4	6160	0-30	31.1	4520	0
A 186	Grit blasted	46.8	6800	0	30.4	4410	0
A 187	Grit blasted	47.3	6870	0-5	31.9	4630	0
A 189	Grit blasted	49.6	7200	0	31.1	4520	0
A 1120	Grit blasted	49.6	7200	0	33.5	4870	0

cyclic humidity and accelerated weathering as described for 500 and 1000 hours respectively. On completion of the test cycle the panels were removed from the test chamber and allowed to recover for a period of 72 hours at room temperature and humidity before testing. Tables 6 and 7 show the data obtained by the torque shear and direct pull-off techniques respectively.

Polyurethane paint

Reference to Table 6 will show that the torque shear data indicate that the effect of γ -methacryloxy propyltrimethoxysilane and *n*-beta aminoethyl aminopropyltrimethoxysilane was outstanding. The results from both panels were consistently high and only in the case of *n*-beta aminoethyl aminopropyltrimethoxysilane in

the cyclic humidity test was any paint detached. The bond strengths were at least double that of the degreased non-silane control and slightly higher after the test than the grit blasted control.

All five silanes produced greater bond strengths than the degreased non-silane control. The direct pull-off data (Table 7) accurately reflected the torque shear results for γ -methacryloxy propyltrimethoxysilane and *n*-beta aminoethyl aminopropyltrimethoxysilane, but the values were consistently lower and in general there was a much greater tendency for more paint to be detached. In the case of the β -(3,4-epoxycyclohexyl)ethyltrimethoxysilane accelerated weathering results and the γ -glycidoxy propyltrimethoxysilane control values, lower bond strengths were obtained than on the non-silane controls, but these were isolated instances and the general picture is

Table 6
Effects of environment on bond strength of paints to aluminium – torque shear

Silane	Surface preparation	Controls			Cyclic humidity (500 h)			Accelerated weathering (1000 h)		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment	MPa	psi	Area of detachment
Polyurethane paint										
None	Degreased	15.8	2300	100	14.7	2130	100	21.7	3150	80
None	Grit blasted	45.8	6650	0-20	28.2	4100	30-50	38.4	5570	2-5
A 174	Degreased	41.5	6030	0	45.8	6650	0	40.0	5800	0*
A 186	Degreased	21.0	3050	100	34.6	5030	30-100	27.3	3960	20-90
A 187	Degreased	24.4	3540	60-100	29.5	4280	70-100	29.3	4260	0-100
A 189	Degreased	34.4	4990	0-80	38.4	5580	10-40	29.5	4280	0-60
A 1120	Degreased	40.3	5850	0	40.6	5900	0-30	40.3	5850	0*
Epoxide paint										
None	Degreased	27.9	4050	30-90	32.7	4750	60	32.0	4650	5-30
None	Grit blasted	40.0	5800	10-30	29.2	4250	30-60	41.3	5990	0*
A 174	Degreased	42.2	6130	0	41.5	6030	0*	42.5	6170	0-2*
A 186	Degreased	44.2	6420	0	41.0	5950	0*	41.4	6000	0*
A 187	Degreased	43.9	6370	0-10	41.7	6050	0*	42.7	6200	0*
A 189	Degreased	42.2	6130	0	41.3	6000	0*	44.1	6400	0*
A 1120	Degreased	41.5	6030	0	39.5	5730	0-10*	44.1	6400	0*

*Cohesional failure of the paint

Table 7
Effect of environment on bond strength of paints to aluminium – torque shear

Silane	Surface preparation	Controls			Cyclic humidity			Accelerated weathering		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment	MPa	psi	Area of detachment
Polyurethane paint										
None	Degreased	11.4	1660	100	11.6	1680	100	13.1	1900	100
None	Grit blasted	27.6	4000	0	28.2	4090	60-100	26.0	3780	60
A 174	Degreased	33.1	4800	0-60	28.0	4070	0-5	28.4	4130	0-60
A 186	Degreased	14.7	2140	100	15.0	2180	100	9.3	1350	100
A 187	Degreased	10.1	1470	100	15.8	2300	100	17.8	2580	100
A 189	Degreased	19.8	2880	100	20.2	2930	50-90	22.4	3250	40-70
A 1120	Degreased	23.2	3370	5-50	30.4	4410	0-5	28.4	4130	0
Epoxide paint										
None	Degreased	20.3	2950	50-90	22.0	3200	60-90	25.0	3630	10-30
None	Grit blasted	27.6	4000	0	27.6	4000	60-90	27.3	3960	50
A 174	Degreased	30.8	4470	0	30.8	4470	0	27.6	4010	10-30
A 186	Degreased	18.1	2630	30-70	29.7	4310	0	28.7	4160	0
A 187	Degreased	18.0	2610	10-90	29.7	4310	0	27.3	3960	0-60
A 189	Degreased	22.7	3300	20-50	33.6	4880	0	24.4	3540	30-60
A 1120	Degreased	29.7	4310	0-5	30.8	4470	0	28.9	4190	10-30

one of considerable improvement in bond strength with good agreement between the two bond test measurements.

Epoxide paint

The torque shear values for all the silane treated panels were consistently high and almost all represented minimum bond strengths as very little paint was detached in any test.

The pattern of results for the direct pull-off technique was less consistent as the control values for the β -(3,4-

epoxycyclohexyl)ethyltrimethoxysilane and γ -glycidoxy propyltrimethoxysilane were lower than those for the degreased non-silane control. However, the results indicated the superiority of γ -methacryloxy propyltrimethoxysilane and n-beta aminoethyl aminopropyltrimethoxysilane.

Paints on mild steel

Polyurethane paint

Table 8 shows the torque shear values for the

Table 8
Effect of environment on bond strength of paints to mild steel – torque shear

Silane	Surface preparation	Controls			Cyclic humidity (500 h)			Accelerated weathering (1000 h)		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment	MPa	psi	Area of detachment
Polyurethane paint										
None	Degreased	22.6	3280	20-100	24.8	3600	100†	31.5	4580	5-20†
None	Grit blasted	40.0	5800	10	34.8	5050	5-100	39.5	5730	5-10†
A 174	Grit blasted	45.8	6650	20	38.6	5600	5-10	41.3	6000	0†
A 186	Grit blasted	46.6	6770	0-10	41.0	5950	0	41.3	6000	0
A 187	Grit blasted	48.2	7000	0	42.9	6230	0	44.1	6400	0
A 189	Grit blasted	46.7	6780	0-5	43.1	6250	0	44.1	6400	0
A 1120	Grit blasted	49.6	7200	0	41.3	6000	0-100†	42.7	6200	0
Epoxide paint										
None	Degreased	36.2	5260	100	30.8	4460	40	35.0	5080	10-30
None	Grit blasted	40.0	5800	10-30	40.8	5930	0-20	42.4	6150	0-10
A 174	Grit blasted	42.4	6160	0-30	45.9	6660	20-60	38.6	5610	10-60
A 186	Grit blasted	46.8	6800	0	45.5	6600	0-5	43.3	6280	0-10
A 187	Grit blasted	47.3	6870	0-5	44.6	6480	0-5	48.2	7000	0-5
A 189	Grit blasted	49.6	7200	0	46.6	6770	10-20	42.7	6200	0-30
A 1120	Grit blasted	49.6	7200	0	43.9	6370	0-20	46.4	6730	0*

†Some corrosion on the panel *cohesional failure of the paint

Table 9
Effect of environment on bond strength of paints to mild steel – direct pull-off

Silane	Surface preparation	Controls			Cyclic humidity (500 h)			Accelerated weathering (1000 h)		
		MPa	psi	Area of detachment	MPa	psi	Area of detachment	MPa	psi	Area of detachment
Polyurethane paint										
None	Degreased	15.4	2230	100	3.4	500	100	14.9	2030	100†
None	Grit blasted	35.8	5200	10-40	15.5	2210	100	23.5	3410	5
A 174	Grit blasted	34.6	5020	0-90	17.8	2580	10-50	31.0	4500	0
A 186	Grit blasted	29.1	4230	20-90	25.5	3700	0-10	31.3	4540	0
A 187	Grit blasted	31.6	4590	50-70	26.9	3900	0-10	29.8	4320	0
A 189	Grit blasted	34.0	4930	0-90	21.9	3180	0-2	31.3	4540	0
A 1120	Grit blasted	39.9	5800	0	21.6	3130	80	31.4	4560	0
Epoxide paint										
None	Degreased	19.5	2830	20-40	17.4	2520	100	16.1	2340	80
None	Grit blasted	25.9	3760	30	21.8	3160	100	26.0	3770	10
A 174	Grit blasted	31.1	4520	0	26.4	3830	60	22.9	3320	0*
A 186	Grit blasted	30.4	4410	0	28.3	4110	0-2	31.4	4560	0*
A 187	Grit blasted	31.9	4630	0	26.4	3840	0-20	29.1	4220	0*
A 189	Grit blasted	31.1	4520	0	24.7	3590	10-80	27.8	4040	0*
A 1120	Grit blasted	33.5	4870	0	30.1	3830	50-80	22.5	3270	0-70

†Some corrosion on the panel *cohesional failure of the paint

polyurethane paint on mild steel. It should be noted that the silanes have been applied to grit blasted panels. The bond strengths show a consistent pattern with high values for all the silane coated panels both before and after the environmental tests. Many of the values reported represent minimum; bond strengths as most samples showed zero detachment. In every case the values recorded were greater than those for the non-silane controls. The direct pull-off values, Table 9, show a very similar pattern.

Epoxide paint

Table 8 shows the direct pull-off values for the epoxide paint on mild steel. With only one exception the values for the silane treated panels are greater than the non-silane controls under all conditions of test. The bond strengths show clearly the increase in bond strength resulting from the use of silanes. The direct pull-off values, Table 9, show a very similar pattern.

Sites of failure

Throughout this present paper the sites of failure on individual specimens have been recorded as "percentage area of detachment", this represents an apparent adhesion failure of the paint from the substrate. The remainder of the area may have failed cohesively within the paint film or some failure associated with the adhesive used, i.e. adhesion failure from the paint film, cohesive failure within the adhesive layer, or detachment from the reverse side of the test panel or the bonded on specimen. In these latter cases a "zero" detachment has been recorded. The percentage area detached is, then, the area on the test panel over which the paint has apparently been removed. There is therefore an implication that the area of detachment is an actual adhesion failure from the substrate, and for all *practical* purposes this may be regarded as being the case. From a strictly scientific standpoint there is no intended statement of a *true* adhesion failure from the substrate. It has been demonstrated that for the paints under test with and without silane pretreatments, the site of failure is in fact cohesive within a pigment free layer of similar, but not identical, composition to the bulk polymer²⁰.

Discussion

Refs. 23-25

It has been demonstrated in this paper that the use of silanes as pretreatment primers materially improves the initial adhesion of urethane and epoxide resin-based paints to aluminium and mild steel substrates. This improvement is sufficiently large to result in the adhesion to degreased substrates to be as great or even greater than that to grit blasted surfaces, even on aluminium. Further, this improvement is maintained even after high humidity and accelerated weathering tests. Although there were differences in the effect of the five silanes tested, all produced an improvement in adhesion.

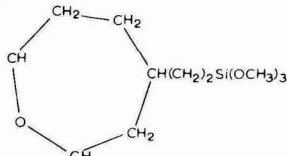
Several theories have been proposed to explain the mechanism by which silanes improve adhesion under dry and wet conditions, the major interest being that of adhesion to glass²³⁻²⁵, thereby reflecting the early technological interest in glass reinforced plastics. The current theory, "the Reversible Hydrolytic Bond Mechanism", is largely a combination theory and postulates a reaction via the organic radical of the silane with the resin matrix and an elimination of water from the glass or metal surface due to the strong hydrogen bonding capacity of the SiOH. The hydrated silanol bonds to the surface OH groups with the elimination of water. The interface between polymer and substrate is stated to be a plastic restrained layer of intermediate modulus allowing a transfer of stress from the high modulus surface to the low modulus polymer. A reversible breaking and remaking of stressed bonds between the silane and the substrate is proposed which allows stress relaxation without loss of adhesion in the presence of water.

If we examine the possibility of chemical reaction between the silanes and the various components of the organic binders present in the two paints, the γ -methacryloxy propyltrimethoxysilane (A 174):

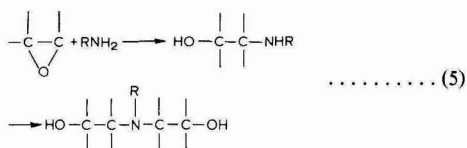


seems unlikely to react chemically. However, the presence of the polar C = O group introduces the possibility of a hydrogen bonding mechanism with the epoxide, and anionic addition at the double bond in the isocyanate curing agent in the polyurethane.

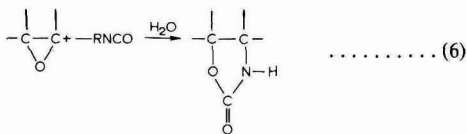
β -(3,4-epoxycyclohexyl)ethyltrimethoxysilane (A 186):



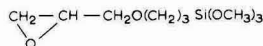
may be expected to react with the amine groups on the polyamide curing agent according to Equation (5):



With the polyurethane paint, a reaction between the isocyanate group and the epoxide group on the silane to produce an oxazolidone appears to be a distinct possibility, Equation (6):

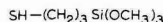


γ -glycidoxy propyltrimethoxysilane (A 187):



may be expected to react with the polyamide curing agent in the epoxide paint as in Equation (5), and with the isocyanate adduct curing agent in the polyurethane as in Equation (6).

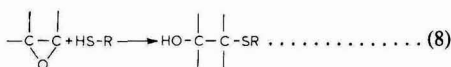
In the case of γ -mercaptopropyl trimethoxysilane (A 189):



a reaction with an isocyanate in the polyurethane system via the terminal -SH group functioning as an -OH reactive site according to Equation (7) appears possible:



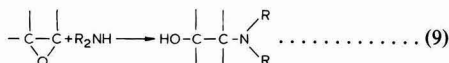
The -SH group may also react with an epoxide group in the epoxide system according to Equation (8):



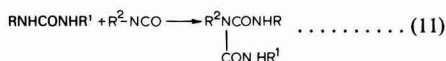
The *n*-beta aminoethyl aminopropyltrimethoxysilane (A 1120):



may be expected to react with the epoxide resin via the epoxide group using the terminal primary amine group in the silane according to Equation (5), and possibly via the secondary amine group on the silane as in Equation (9):



In the case of polyurethane paint, A 1120 might be expected to react with the isocyanate group in the curing agent to form, initially, a substituted urea as in Equation (10), with the possibility of a further reaction between this substituted urea and a further isocyanate group to form a biuret as in Equation (11).



Thus it would seem that with the exception of γ -methacryloxy propyltrimethoxysilane, some chemical reaction between the silanes and some component of both paints exists. However, the likelihood of any individual reaction will depend on the structure of the resin and curing agent, the presence of water, the type of solvent and the presence or otherwise of catalytic agents, and although a reaction may be theoretically possible, it is not necessarily probable.

From the results presented it would seem that γ -methacryloxy propyltrimethylsilane and n-beta aminoethyl aminopropyltrimethoxysilane are both particularly effective as pretreatment primers on aluminium for polyurethane and epoxide paints; and γ -mercaptopropyl trimethoxysilane and n-beta aminoethyl aminopropyltrimethoxysilane for mild steel. The actual differences in their performances are small under any given set of experimental conditions, and the n-beta aminoethyl aminopropyltrimethoxysilane, A 1120, gives an excellent all-round performance on aluminium and mild steel.

In this experimental programme the method chosen for the application of the silane was the dry MEK/water rinse combination, but it would seem likely that the use of an alcohol/water mixture would be equally effective and offer the advantage of a one stage application.

Conclusions

It may be concluded that the use of organo-functional silanes as pretreatment primers on aluminium and mild steel substrates results in a dramatic increase in the initial adhesion of two-pack polyurethane and epoxide paints.

Further, this improvement in adhesion due to the use of a silane is retained even after exposure to cyclic humidity under condensation conditions and to accelerated weathering.

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Appendix 1

Silanes under test

Chemical name	Structure	
γ -methacryloxy propyltrimethylsilane	$\text{CH}_2 = \overset{\text{CH}_3}{\text{C}} - \overset{\text{O}}{\parallel} - \text{CO}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$	A174
β -(3,4-epoxycyclohexyl)ethyltrimethoxysilane		A186
γ -glycidoxy propyltrimethoxysilane	$\text{CH}_2 - \text{CH} - \text{CH}_2\text{O}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$	A187
γ -mercaptopropyl trimethoxysilane	$\text{HS}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$	A189
n-beta aminoethyl aminopropyltrimethoxysilane	$\text{H}_2\text{N}(\text{CH}_2)_2\text{NH}(\text{CH}_2)_3\text{Si}(\text{OCH}_3)_3$	A1120

letters

All correspondence should be addressed to: Mr D. S. Newton, Honorary Editor, OCCA, Priory House, 967 Harrow Road, Wembley, Middlesex, HA0 2SF, England.

In reply

I appreciate your giving me the opportunity to reply to Mr Van Der Walt's comments of 31 August on "Getting the most from your test-budget". Actually, I agree with what he has to say, but would like to clarify a few points.

I have no doubt that "sound reasons" motivated SABS's use of the 68-12 cycle. My disappointment lies in the fact that excellent results were being obtained and that in spite of nine years of experience with the 68-12 cycle selected for use as their standard weathering cycle, the SABS reverted to the 102-18 cycle in the interest of international standardisation. What good is an international standard if its use does not provide the required results?

I must question Mr Van Der Walt's statement regarding the ASTM cycle and black panel temperature. If ASTM "investigations" revealed that these conditions gave maximum acceleration and best correlation with actual weathering tests for paints manufactured at the time, I have not been able to track down the investigation or its results. I think it more likely that the test parameters are historical and a result of apparatus limitations. The old National Carbon device (around which early standards were written) operated at one revolution per three minutes. As this device was replaced by later Atlas models operating at one revolution per minute, the 102-18 cycle came into being in order to maintain the same light/water spray ratio provided by its ancestor. It is interesting to note that this ratio still exists in many of the ASTM listed cycles – e.g., 51-9 or 17-3.

The need for an "ultimate accelerated durability test" is recognised in the paper's conclusion. However, the intent of Programmed Environmental Testing is to provide more reliable test results to correlate better to specific geographic areas and/or specific in-service applications. By no means do I consider this theory a panacea for all the mental malaise one encounters in durability testing. Also, I must add that I do not believe the test conditions currently suggested/recommended by ASTM, or other organisations with which I am familiar, fit the bill either.

I agree that an accelerated weathering test for areas or countries of widely diverse climatic conditions would be more useful, but such an individual test method may never exist. Nor, in some cases, would compromise be realistic. It would be ludicrous to assume that the parameters for an accelerated test designed for off-shore platforms, ocean-going vessels, aircraft flying at 11,000 metres, automotive coatings and house paints should be the same. Yet, apparently, this is what is occurring in many laboratories throughout the world.

"Getting the most from your test-budget" and a follow-up paper "Programmed Environmental Testing – A PET Theory" (scheduled for publication in *JOCCA* are attempts to encourage the reader to depart from historical test methods and use their laboratory equipment to its full capabilities. The thought was to stimulate interest by re-defining problems (geographic location, climatic variations, in-service applications, etc.), stressing the flexibility/programmability of new laboratory apparatus, and suggesting alternative approaches.

Although South Florida Test Service has not completed

Accelerated weathering

We have noted in J. L. Scott's paper "Getting the most from your test budget" in the May 1982 edition of *JOCCA*, his reference to J. E. Fullard's 1965 paper regarding the laboratory accelerated weathering cycles investigated at the South African Bureau of Standards in Pretoria. Mr Scott "assumes that the climatic conditions referred to would be somewhere in the vicinity of Pretoria, yet upon examining the variety of cycles used, not one of them represents a simulation of that climate".

The similarity between the North American (USA) and South African climatic conditions influenced the SABS (South African Bureau of Standards) to evaluate the cycles investigated and used in the USA. Sound reasons for using the 68-12 cycle for a limited period are given by Fullard. The SABS eventually decided, in the interest of international standardisation to adopt the ASTM cycle and black panel temperature specified. These conditions, to the best of our knowledge, were chosen by ASTM because investigations revealed that these conditions gave maximum acceleration and the best correlation with actual weathering tests for paints manufactured at the time. The South African climate like that in the USA varies widely from continental to sub-tropical to semi-desert to desert conditions. The operating cycle should therefore be such that it would provide for the most severe or at least a compromise of these conditions. Assuming that the estimated cycles given in Table 5 of Scott's paper are representative of the actual conditions for the locations listed, the average for Durban and Phoenix (desert-like conditions) by coincidence actually gives 102-18 and that for Durban, Johannesburg and Phoenix gives 100-20.

Scott certainly has a novel and logical approach to the complicated subject of accelerated or artificial weathering. His idea of programmed environmental testing to simulate as closely as possible a specific geographical area can be regarded as the ideal procedure for research for individual projects. To provide for accelerated testing in countries with varying climatic conditions such as the USA, Republic of South Africa and Australia, a broadly accepted cycle would be more realistic both from a testing and economical point of view. We note from Scott's technical articles published in *Atlas Sunspots* that he has initiated an experimental programme at South Florida Test Service and we are looking forward to the outcome of these tests and ASTM's reactions to his recommendations.

*Yours faithfully,
L. van der Walt
Principal Scientist*

*Paints & Sealants Division
South African Bureau of Standards
Private Bag X191
Pretoria 0001
Republic of South Africa*

31 August 1982

its research comparing test results using various test cycles and light sources to outdoor exposures, one committee of ASTM has already reacted. Committee E-44 on Solar Energy Conversion has specified the Miami test criteria from Table 5 of the paper for E-765 Standard Practice for Evaluation of Cover Materials for Flat Plate Solar Collectors. Only the future will reveal the reaction(s) of other technical and material committees.

South Florida Test Service Inc.
9200 NW 58 Street
Miami
Florida 33178
USA

Yours faithfully,
John L. Scott
Vice President

22 September 1982

Further information on any of the publications reviewed may be obtained by circling the appropriate *Reader Enquiry Service* number on the form at the back of the *Journal*. Enquiries will be forwarded to the publisher.

The World Paint Industries. A Review 1980-1981

Helma Jotischky
Paint Research Association, Waldegrave Road,
Teddington, Middlesex
April 1982. pp. 388

In the introduction the President of the PRA comments "That facts have the elusive quality of seldom being at one's fingertips, figures even more so." The author of this reference volume has removed this problem with respect to the paint industry.

In this well referenced publication the major (and many minor) paint producing countries are grouped geographically and the producers identified. Types of product produced, export and import figures and total production figures are given where these are available. Some interesting information is also given in the "Who owns what" sections of the book, and indicates the level of investment throughout the world by the major chemical companies "downstream" as well as that of the western paint industries overseas.

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

West Riding

Purchasing in the difficult 80s

The opening lecture of the 1982-83 session of the West Riding Section was held at the Mansion Hotel, Leeds on 7 September 1982.

Mr O. Brosh, purchasing director of Silver Paint & Lacquer Holdings Ltd, gave a most enlightening lecture on "Purchasing in the difficult 80s".

Mr Brosh's opening statement was, "Do you believe you only get what you pay for." It was suggested that a professional buyer must consider every aspect of a proposition, including quality, service, reliability and technical performance, as well as price.

It was the opinion of Mr Brosh that a buyer contributes strongly to the profitability of his or her company.

He went on to emphasise this point with several examples. Also, he suggested that a professional buyer

letters

reviews

As your reviewer has pointed out previously, this type of book depends upon statistical information being published by governments, who have a habit of changing the rules of reporting from time to time, so making comparisons difficult. Moreover, there are countries which although they have a reasonably well developed paint industry, do not provide figures on production and type of product, e.g. Syria, Iraq and Iran.

These facts of industrial life do not in any way detract from the overall usefulness of this book, which your reviewer believes to be unique, and both the author and the PRA are to be congratulated on the meticulous attention to detail, the layout, and particularly the speed at which the book has been produced.

Reader Enquiry Service No. 21

D. S. Newton

occa meetings

should be a buyer only and not an amateur chemist or financier.

It was the opinion of Mr Brosh that a buyer must allow his suppliers to make a profit, this remark indeed surprised the majority of the audience. However, he went on to emphasise that it must be considered that a supplier is professional enough to make a profit and, therefore, a buyer must achieve the best price possible.

A buyer, it was considered, must be well trained and also skilled at the art of negotiating. The current policy of recruiting from unrelated departments, principally the technical department, wasn't judged to be satisfactory.

Mr Brosh proposed that in a negotiating situation it would be useful for the buyer to know as much about the supplier as possible. This information would be used to produce a better deal for both parties since it was

OCCA meetings

suggested that during negotiations, delivery schedules, revised specifications, methods of transport, packaging and reciprocal trading could be considered to mutual benefit.

In conclusion Mr Brosh quoted a version of a John Ruskins maxim which went as follows:

"There is hardly anything in the world that different suppliers somewhere cannot make as well or a little better and sell a little cheaper, and people who hardly consider price at all are their lawful prey."

After a most intriguing question period, a vote of thanks was proposed by Mr Hugh Young.

D. V. Maltman

Newcastle Section

Fundamentals of paint technology

On Thursday 9 September 1982, a Seminar entitled "Fundamentals of paint technology" organised by the Newcastle Section of OCCA was held at Durham University. The seminar was attended by 54 delegates, representing a wide spectrum of interest: raw material suppliers, paint manufacturers, equipment manufacturers and academics.

The broad spectrum of topics covered served as a general introduction to the paint industry for both junior technologists and qualified personnel new to the paint and allied industries.

"Coloured pigments"

By A. Abel, Colour & Surfactants Div, Hoechst (UK) Ltd

A general review of inorganic and organic pigments, followed by a detailed discussion of the relevance of the data provided by suppliers. The relevance of colour index and pigment properties, e.g. hiding power, dispersibility and stabilisation, lightfastness and durability, were detailed.

"White pigments"

By J. Hall, BIP Tioxide Ltd

The properties of common white pigments were detailed with special reference to opacity, colour and durability parameters. The speaker demonstrated the influence of refractive index, particle size and dry hiding, and completed his paper by discussing reflectance characteristics, dispersibility and gloss factors.

"Binders in paint formulation"

By G. Carr, Tor Coatings Ltd

Basic paint composition was reviewed. All commonly used binder types were detailed, convertible and non-

convertible. The characteristics of oil, oleoresins and alkyds were detailed, and there was discussion on chlorinated rubber and vinyl resin media. Data on chemical cure systems, e.g. epoxy resin and urethanes, were shown. The water-borne binder types were detailed as well as their performance properties.

Films

Two films were shown covering the historical background to paint coatings and modern paint manufacturing methods.

"Film testing"

By L. Morpeth, Byk Mallinckrodt

This paper covered equipment and test methods for assessing coatings in liquid form through the transitional stages to the dry film. Equipment was available for discussion and scrutiny.

A lively forum discussion followed the series of lectures to complete an interesting and beneficial day.

B. Robinson

Midlands

Memoirs of a Birmingham paint man

The first technical meeting of the 1982-83 session of the Midlands Section was held on 16 September at the Clarendon Suite, Stirling Road, Edgbaston, Birmingham.

Members and guests heard Mr R. L. Staples, managing director of Midlands Speciality Powders Ltd, speak of the "Memoirs of a Birmingham Paints Man".

Mr Staples said that during his 40 years in the industry he had seen many significant changes in the raw materials used and the hours worked. He started his career in the resin laboratory of Arthur Holden and Sons Ltd. The raw materials used at that time were rather crude and limited in variety.

In 1952 the speaker moved to Gittings and Hill where eventually he became production manager dealing mainly with industrial coatings. His next move was to Blundell-Permoglaze as works manager. Here he was confronted with very many products and pack sizes as was common to the decorative trade. During his time with the company he managed to rationalise and reduce many of these lines.

Finally, Mr Staples with Mr Sid Harris whom he had known since their time at Arthur Holdens set up a powder coating manufacturing company at Bilston. Initially things did not go very well and they lost money on their first year of operation. Later on they joined up with Briggs and Townsend who injected some capital into the company, eventually acquiring a majority shareholding.

Throughout his talk Mr Staples kept the audience highly amused with stories about people with whom he had worked - many of them well known to the members of the Section.

After question time Mr B. Fowler proposed a vote of thanks which was warmly endorsed by the audience.

B. E. Myatt

Further information on any items mentioned below may be obtained by circling the appropriate Reader Enquiry Service number on the form at the back of the Journal. Enquiries will be forwarded to the organisation concerned.

news

Paint trade struggles through recession

The effect of recession on the paint trade and allied industries in the UK is illustrated when analysing the latest financial survey from Inter Company Comparisons. The survey, entitled "Paint and Allied Trades" (13th edition), profiles the fortunes of 306 companies in terms of two years' comparative figures on turnover, total assets, current liabilities, profits before tax and payments to directors. The survey incorporated a directory section listing principal place of business, principal activities and holding companies (if any) for each company surveyed. Twenty-one of the companies surveyed are quoted on the Stock Exchange, with the remaining 285 being unquoted or "private" companies.

An analysis of the data given in the survey reveals the following statistics:

	Quoted	Unquoted
Increased turnover	71.4%	71.6%
Increased profits	61.9%	32.3%
Loss in latest account year shown	14.9%	21.7%
Increased payments to directors	81.0%	67.4%

These figures emphasise the scale of the problems felt by much of the industry in recent times. Quoted companies have, on the whole, performed slightly better than their smaller more vulnerable counterparts. The fact that under a third of the unquoted companies surveyed could boast an increase in profits illustrates the tough time much of the UK paint industry has had to endure in recent years. The level of losses, more than a fifth for the unquoted companies, is an alarming one, and casts a degree of doubt on the ability of the industry to pull through the recession in its present form.

Prospects for the future of the UK paint industry depend to a large extent on the ability of UK manufacturing industry to pull through the recession, as until this occurs, overall industrial demand for paint is unlikely to increase by any significant degree. The domestic side of the industry is healthier and as long as the large DIY retailers keep in business, volume sales will be at a reasonable level; although profits to manufacturers may not be, as margins have been cut back.

Reader Enquiry Service No. 31

Burmah acquires adhesives company

Burmah Speciality Chemicals Ltd has acquired all the shares of Industrial Adhesives Ltd, previously a privately owned British company with a turnover of £5.5M.

The company makes and markets a

well established range of specialised water-based, solvent-based and hot melt adhesives to customers in the packaging, construction and manufacturing industries. It is also a prominent producer of pressure sensitive adhesives for such applications as labels and tapes.

Industrial Adhesives Ltd will continue to trade under its own name from its headquarters and principal plant at Chesham, Bucks, where it employs 86 people.

Mr M. J. Moore, chief executive of the Adhesives and Sealants Division of Burmah Speciality Chemicals, will become chairman of Industrial Adhesives and Mr B. J. Wardle, its founder, chairman and principal shareholder, will remain managing director for a transitional period and thereafter continue as a member of the Board.

Reader Enquiry Service No. 32

Preparing selected resins

An improved process for preparing air drying alkyd resins has been developed at Battelle's Columbus Laboratories.

The new process is said to improve the quality of the resins while reducing the energy and time necessary to prepare them.

Battelle recently received a US patent for the process and now hopes to commercialise it.

According to Battelle co-inventors George A. Cremeans and Richard A. Markle, air drying alkyd resins are widely used in paints and other products because when exposed to air the resins form a tough coating that does not easily chip or break. To prepare paints, the resins are mixed with organic solvents, pigments, and drying agents. With the new Battelle process, the organic anhydrides and glycidyl esters are firstly mixed with metal salt catalysts. The mixture is heated in reactors at temperatures not exceeding 100°C for between two and four hours. In the reactors the compounds polymerise and form a resin.

Cremeans and Markle explained that with conventional processing, different compounds are used as the resin feedstock. These compounds require heating at between 230 and 250°C for as long as 12 hours.

The experts said that in addition to energy and time savings, the new process offers several other advantages over conventional methods.

First, under the new process the resins are colourless. Under conventional

methods the finished resins take on a darker colour that is difficult to remove. The colour often results because substantial amounts of by-products are produced during the long heating periods.

Second, the alkyd resins produced by the new Battelle process have a narrower molecular weight range, imparting excellent levelling or flow-out characteristics to coatings formulated with these resins.

Reader Enquiry Service No. 33

ICI's hyper-dispersants for inks

A novel class of dispersing aids is now being sold on a worldwide basis by ICI Organics Division following encouraging customer reaction in Europe.

The Solsperse agents are intended principally for use in printing ink manufacture. They have been progressively introduced by ICI over the last two years in the UK and in Western Europe, but in recent months the Solsperse agents have also been introduced to major customers in the USA and Japan. Substantial sales are expected in Japan from about January when registration of the key products with the appropriate government authorities should be completed.

The advantages of using Solsperse agents to ink manufacturers are said by ICI to be dramatic. One major advantage is said to be much improved productivity from existing milling equipment, in many cases, it is claimed, this can be more than doubled. Flexibility of production, extra colour strength development and improved print quality are also of great benefit to the manufacturer.

As an alternative to the ink manufacturer adding Solsperse agents to his ink formulation, pigment manufacturers can treat pigments with the agents to provide easily-dispersible products for the ink industry.

An active research programme is aimed at developing the hyper-dispersant concept further and developing products for other applications. Considerable scope also exists for developing applications for the present product range outside the inks field, and various technical evaluations are under way both in Blackley, Manchester and in the laboratories of ICI Americas in Delaware.

Reader Enquiry Service No. 34

news

new products

New powder coating system

Elgada is the name of a new range of electro gas dynamic powder coating equipment produced by Electropaint Ltd.

The product, of extensive research and development, is described as revolutionary.

Two unique features of the system are:

1. The coating tunnel, which is constructed entirely of non-conductive materials and acts to inhibit the deposition of powder upon itself thus ensuring that the maximum amount is deposited on the transient workpiece.
2. The gun, with its internal system of charging powder which ensures that a charge is applied to almost every particle of powder sprayed, whatever the powder type. Conventional electrostatic guns call for particles to travel along fixed applied lines of force. The phenomenon referred to as Faraday caging prevents charged particles from penetrating recesses. With the Elgada gun there are no fixed lines of force, the powder is simply emitted as individual charged particles with an affinity for earth, so complex shapes may be coated.

Due to the above features, it is claimed a powder deposition efficiency at first pass of 80 per cent to 85 per cent is common, while some goods may be coated at greater than 90 per cent efficiency.

This high efficiency, coupled with the design of the control console and the powder feed unit, which constitute the remainder of the Elgada equipment, means that colour changes can be effected literally in minutes with a minimum of waste and no contamination.

Elgada small systems are also available. These incorporate all the features associated with the large systems, but will coat parts as small as 10 mm square.

Reader Enquiry Service No. 35

New ink pigment

BASF has introduced Heliogen Green D 8605. It is a new phthalocyanine green pigment for letterpress and offset printing inks.

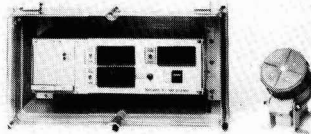
Reader Enquiry Service No. 36

Measuring wet time and coating thickness

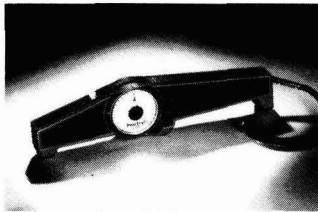
Sheen recently announced the availability of two new instruments.

With paint test panels being exposed naturally or under simulated conditions, it is important to know the time during which the panel is wet due to rain or dew, and the time that the paint film is damp due to retained moisture. The Startronic discriminating event time recorder incorporates three elapsed time indicators which indicate the total test time, the wet period and the total damp period.

Also from Sheen is the PosiTest gauge for the non-destructive measurement of non-magnetic coatings on steel. The PosiTest banana gauge – so called because of its shape – is recommended for measuring film thicknesses of paints, plastics, galvanising, non-ferrous plated deposits, phosphate coatings, etc. on steel.



Wet time recorder



Coatings thickness gauge

It functions on a cobalt rare earth permanent magnet, so no power source is required. Weighing less than 200 grams it is believed to be the lightest thickness measurement gauge of its type on the market.

The PosiTest gauge is available in two scales: 0-200 microns or 0-2000 microns.

Reader Enquiry Service No. 37

New electrostatic handgun

Because of their greater complexity, electrostatic spray guns have tended to be larger and heavier than their conventional air spray equivalents. However, Ransburg's new R-E-A IV is only 9.5 inches long and a mere 24 ounces in weight.

Small in size and light to handle, the Ransburg R-E-A IV is claimed to set new standards of ease of use and manoeuvrability.

Reader Enquiry Service No. 38

New ICI pigments

A total of nine new pigments covering a wide variety of applications have been added to ICI Organics Division's sales range. The new pigments fall into three groups. Firstly there are the high performance aqueous pigment pastes for exterior quality PVA, acrylic emulsion paints and aqueous leather finishes. These products are said to give excellent light and weathering fastness even in pale shades and to have excellent storage stability, flow properties and fastness to acids, alkalis and over-painting. They contain humectant to minimise drying-out in partially opened storage containers.

Secondly, aqueous pigment pastes for leather finishes, textile printing and emulsion paints. These pigments have been developed primarily for use in textile printing outlets but are also recommended for aqueous leather finishes and emulsion paints. They reportedly give excellent all round fastness properties, are fast to over-lacquering, have high strength and give very bright yellow and red shades.

Thirdly the pigments for industrial finishes and printing inks. Monolite Yellow GR has been specially developed to give high transparency and gloss in viscous and liquid printing inks, and very good opacity and strength in lead-free industrial finishes. The high strength of this pigment allows maximum use of titanium dioxide, nickel titanate and yellow iron oxide and hence optimum economy in industrial finishes. ICI describes Monolite Yellow GR as having excellent heat, solvent and overspray fastness, and very good light and weathering fastness in medium and strong yellows; an economical means of producing lead-free industrial yellows.

Rubine Toner 6BM is a very blue shade manganese toner and is recommended for industrial finishes as an economical way of bluing molybdated chromes, such as Scarlet Chrome C-HM. This pigment is said to give excellent heat, solvent and overspray fastness and good light and weathering fastness in mixtures with molybdated chromes.

Reader Enquiry Service No. 39

Spray painting

Graco is introducing a line of accessories designed for air spray and other spray systems.

This line includes the following products: air and fluid hoses, a half gallon remote pressure cup, a small portable paint supply system, and an air transformer.

The air transformer is compact and said to offer precise pressure control. It is available with single or double outlets and has a maximum regulated pressure of 125 psi (8.5 bar) and a maximum pressure intake of 250 psi (17 bar).

Reader Enquiry Service No. 40



Improvements to the Lovibond Three-Field Comparator include: slot-in discs of Lovibond glass colour standards and a new prismatic viewer

Oil and solvent grading by colour

Important improvements have been included in the latest version of an instrument for grading by colour a wide range of oils and solvents used in paints, shellacs and varnishes.

The new Three Field Comparator from Tintometer Ltd uses the internationally accepted Gardner scale of colour. This is carried as standards of non-fading Lovibond glass on discs of plastic which slot easily into the instrument cabinet.

The instrument has a new prismatic viewer, which makes comparison of sample and colour standards easier. A quartz halogen daylight corrected lamp illuminates standards and samples, and is controlled by an automatic timer to avoid wasting lamp hours.

Solution cells of up to 40 mm in path length are held in a plastic moulding which contains drips or spills and which, like all the instrument's components, is said to be easily removed, cleaned and replaced.

Reader Enquiry Service No. 41

Cost saving powder coatings

A new powder coating which it is claimed can reduce the cost of product finishing by up to 50 per cent compared to traditional powders has been developed by Carrs Paints Ltd.

Combiflex is a low density powder coating system which gives low film thickness without sacrificing overall performance. Carrs state that a powder would normally require 12 minutes curing time at a temperature of 180°C. Carrs have reduced curing time and the temperature to only five minutes at 130°C. Combiflex can be cured in conventional liquid paint stoves.

Reader Enquiry Service No. 42

Breakthrough in rheological additives

A new rapidly dispersing organoclay thickener offering cost savings to paint manufacturers is being launched worldwide by NL Chemicals. According to the company the new organoclay, Bentone SD-1, represents a significant breakthrough in rheological additive technology.

NL go on to state that the outstanding dispersion characteristics of the new product, the first of a family of improved organoclays, will greatly simplify the manufacturing process of solvent-based paints, speeding up throughput, achieving more flexible and trouble-free production whilst maintaining product quality.

Organoclay rheological additives are not only thickeners but also provide control of viscosity and pigment settling and at the same time ensure correct application properties.

Existing organoclay additives give excellent control of coatings rheology, but their use requires precise control of paint manufacturing conditions. In order to obtain full dispersion and activation a time-consuming procedure must be followed, often involving a premix. Even small errors at this stage can result in paint with viscosity, storage and application problems.

Bentone SD-1 is said to be the first rheological additive that enables coatings makers to take full advantage of today's most advanced manufacturing techniques. New types of dispersion equipment with faster throughput rates and, often, lower shear, work best with easy-to-disperse raw materials such as this new product.

A variety of paints made using the new rheological additive are claimed to show excellent properties in both application and performance.

Reader Enquiry Service No. 43

New epoxy resins

A new range of epoxy resins is now available from Dow Chemical Europe.

The DER E-Series resins are designed to give closer manufacturing control and greater end-product quality and consistency in a wide range of applications in the coatings and varnish industries.

An example of the improvement in the E-series specifications is the narrowing of the epoxy equivalent weight of DER 662 from 575-700 to 600-640. The softening point for the same resin has been tightened to 86-94°C from 84-94°C.

Narrower melt viscosity ranges typically are 5000-9000 (at 150°C) for DER 664 UE compared with 4000-10,000 previously, and 2500-4500 for DER 663 UF compared with 2000-4500. Resin colour has been improved in most

news

cases to less than 1 on the Gardner scale.
Reader Enquiry Service No. 44

literature

Airless spraying

A complete technical guide to air-operated airless spraying systems is given in a 16 page publication now available from Binks-Bullows.

The guide covers system components and material feed methods. A substantial section deals with selection and sizing of airless equipment so that users can specify the correct systems for all applications. Detailed tables relate spray tip orifice sizes to common fluids including stains, solvents, lacquers, enamels, sealers and bitumastics.

All aspects of operation including spraying techniques and preventive maintenance are described in detail. A special section deals with fault-finding in terms of spray patterns, spray gun operation and airless pump operation.

Full information is given for operators of airless spraying equipment with step-by-step start-up, shut-down and safety procedures.

Reader Enquiry Service No. 45

people

Mr Sydney Blurton, chairman of ICI Organics Division since 1979, is to retire on 31 March next year after 32 years' service with the company. He will be succeeded from that date by **Mr Tony Rodgers**, who recently returned to Organics Division as a deputy chairman after nearly four years as a senior vice-president of C-I-L Inc., the ICI subsidiary company in Canada.

Peter Stemp has joined the Paintmakers Association (PA) to become its first training specialist. As deputy director (training) his initial task will be to visit every member to establish individual company training needs and thence to determine the needs of the industry. He has gone to the PA directly from the Industrial Society, where he was in a senior position responsible for their entire operations in the southern half of the UK.

OCCA news

For information on membership of OCCA, enquiries should be sent to the Association's offices, see front cover for address.

News of members

Mr Eric Hare

Mr Eric Hare, a former member of OCCA who was technical director of the Via Gellia Colour Co. Ltd, Matlock until his retirement in 1976, died recently aged 71.

Retained by Via Gellia Colour Co. Ltd as a technical consultant until his death, he had spent all his working life in the colour industry.

In 1927 he joined W. Hawley & Son Ltd of Duffield, moving to Via Gellia Colour Co. Ltd as works manager in 1946.

Midlands Section

Ladies' night

The annual ladies' night of the Midlands Section was held on Friday 24 September 1982 in the Westbourne Suite of the Botanical Gardens, Edgbaston, Birmingham.

In his speech, the Chairman of the Midlands Section, Mr D. D. Kimber, welcomed the guests which included the President of OCCA, Mr D. J. Morris, and Mrs Morris, the President of the Birmingham Paint, Varnish and Lacquer Club, Mr J. Green, and Mrs Green, the Director & Secretary of the Association, Mr R. H. Hamblin, and various section chairmen and their ladies. The Chairman paid tribute to the Social Secretary of the Section, Mr K. Chater, for organising the evening and thanked the members' companies for their continuing support of the Section's activities.

The President responded on behalf of the ladies and guests. In his speech he spoke of the various functions he had attended around the world since he had been in office, and kept the audience amused with jokes and lighthearted quotations.

After an enjoyable meal the members and guests adjourned to the ballroom where dancing to the Smoky Bears Big Band continued to 1.15 a.m.

B.E.M.



Shown at the Midlands Section's annual ladies night are — front row seated left to right: Mrs P. Weiss, Mrs B. Gilliam, Mrs D. Morris, Mrs B. Kimber, Mrs J. Green, Mrs R. Fowkes. Ladies standing left to right: Mrs A. Eades, Mrs J. Burns, Mrs J. Kitchen, Mrs V. Devenish. Gentlemen left to right: Mr A. Eades, Mr J. Burns, Mr J. Kitchen, Chairman, Trent Valley Branch, Mr B. Gilliam, Chairman, London Section, Mr D. D. Kimber, Chairman, Midlands Section, Mr G. Fowkes, Chairman, Bristol Section, Mr R. Devenish, Mr D. Morris, President of OCCA, Mr J. Green, President of the Birmingham Paint Varnish and Lacquer Club, Mr P. Weiss, President of the Midlands Section of the Paint Makers Association, and Mr R. H. Hamblin, Director & Secretary of OCCA.



Shown above at the Manchester Section's past-chairmen's lunch are — standing left to right: Sam Duckworth, Tony Jolly, Tom Johnson, Harry Smith, Roy Touchin, Jack Mitchell, Victor Jolly and Bill McDonnell. Seated left to right: Frank Redman (Chairman), Francis Smith, Harry Gosling and Gordon Robson

Manchester Section

Past-chairmen's lunch

On Monday 20 September 1982 a dinner party was held at The Last Drop Village, Bolton, attended by ten past-chairmen of the Manchester Section of OCCA and the present Chairman and Vice-Chairman.

Gordon Robson proposed the toast to

"Past-chairmen of Manchester Section" with a fascinating review of their history.

The senior past-chairman attending, Dr V. G. Jolly, proposed the toast to the "Manchester Section" with an extremely witty speech which speculated on the present and future of the Section, as well as the origins of the venue and the inspiration behind its name.

The evening sped by, punctuated by other unrehearsed but equally relevant toasts and reminiscences.

F.B.R.

CLASSIFIED ADVERTISEMENTS

APPOINTMENTS

**UNION
CARBIDE****CHEMIST**

Our Chemical & Plastics Laboratory in Versoix (Geneva) has an opening for a Chemist

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COLOUR CHEMIST

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LABORATORY TECHNICIAN

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CLASSIFIED ADVERTISEMENTS

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WANTED

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Offers to Box No. 522

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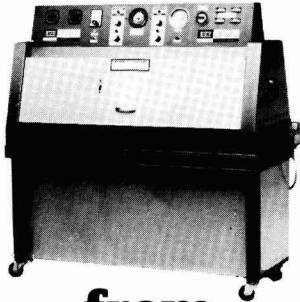
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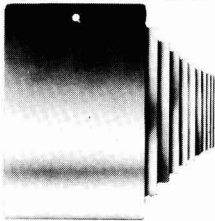


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