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**JOURNAL OF THE
OIL &
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An appraisal of artificial weathering methods for assessment of the durability of paint films

C. E. Hoey and H. A. Hipwood

Flow and ageing of polyvinyl chloride pastes

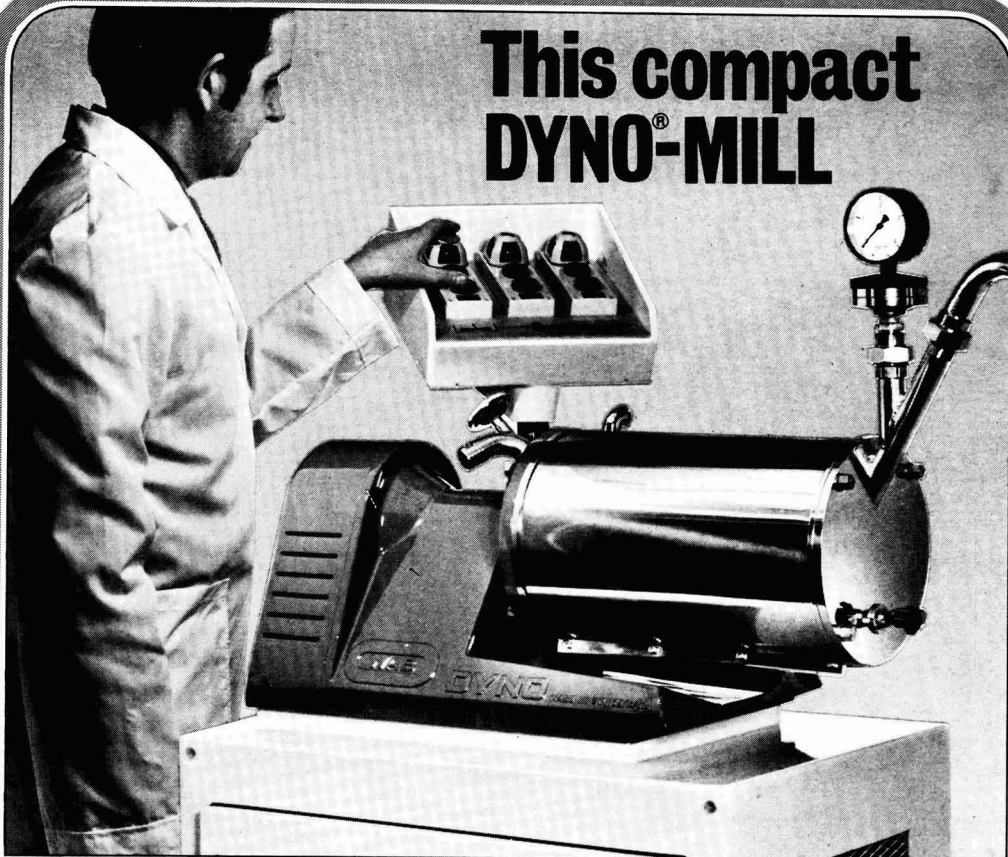
Raj. K. Khanna

Water absorption by polymeric films

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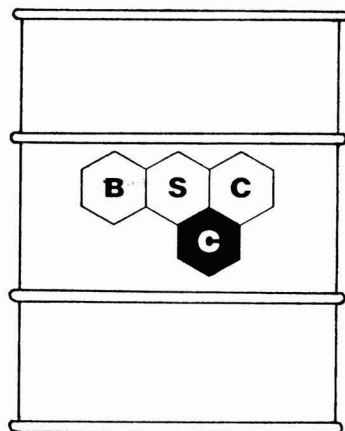
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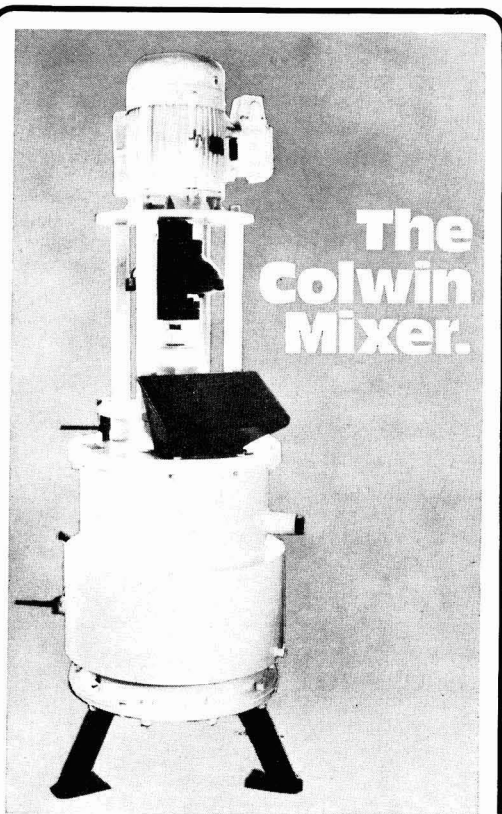
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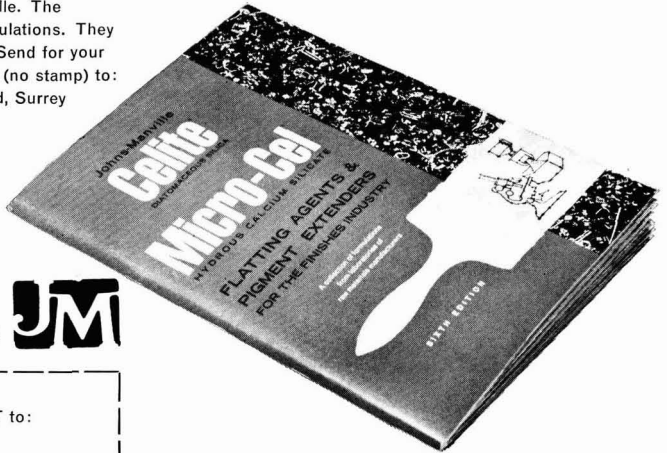
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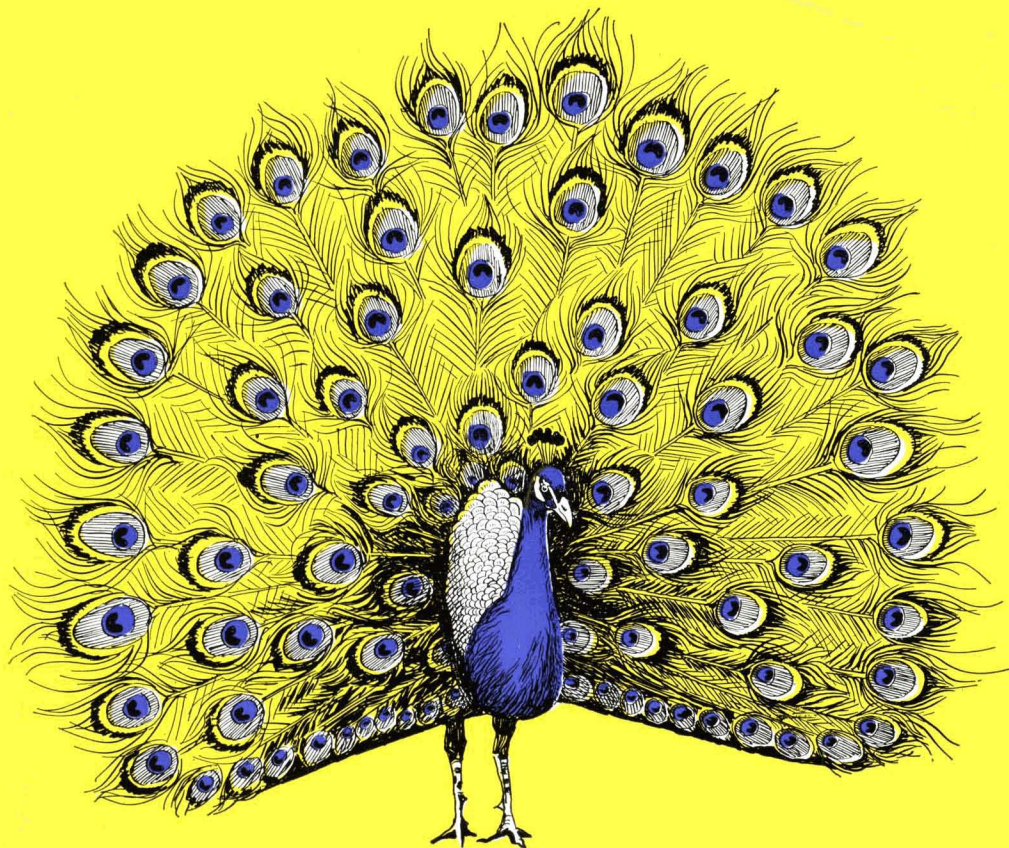
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Transactions and Communications

Owing to the illness of the translator, the papers published in this issue do not include translations of the summaries in Russian.

В виду болезни переводчика нет возможности публиковать выдержки статей на русском языке.

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Transactions and Communications

An appraisal of artificial weathering methods for assessment of the durability of paint films

A report by C. E. Hoey* and H. A. Hipwood† on the findings of the Sub-Committee on the Prediction of Performance of Paints and Varnishes

Summary

A review of investigations on artificial weathering tests by the "Prediction of Performance" Sub-Committee responsible formerly to the "Joint Services Research and Development Board" and latterly to the "Advisory Council of Materials", is reported. The investigations describe comparative tests on a wide range of paint

films exposed to artificial weathering in BS 3900 Part F3, ASTM E42 Type E, the Dew cycle, Xenotest, Climatest and Emmaqua apparatus. Exterior durability results on the paint films exposed at Glascoed (UK), two Australian sites, Phoenix (Arizona) and Singapore are also reported.

Keywords

Equipment primarily associated with analysis, measurement or testing
weatherometer

Properties, characteristics and conditions primarily associated with:

raw materials for coatings and allied products
colour stability

dried or cured films
chalk resistance

Processes and methods primarily associated with: analysis, measurement or testing
accelerated weathering

manufacture or synthesis
gloss reduction

service or utility
weathering

Une évaluation des méthodes de vieillissement artificiel pour l'appréciation de la durabilité des feuillets de peintures

Résumé

On donne un compte rendu des investigations sur les essais de vieillissement artificiel mis au point par le "Prediction of Performance" Sous-Comité qui était responsable d'abord au "Joint Services Research and Development Board" et ensuite à l'"Advisory Council of Materials". Les investigations décrivent les essais comparatifs effectués avec une gamme étendue de feuillets de

peintures exposés au vieillissement artificiel dans les appareils suivant: BS 3900 Partie F3, ASTM E42 Type E, Dew Cycle, Xenotest, Climatest et Emmaqua. On donne un compte rendu également des résultats des essais de vieillissement à partir des feuillets de peintures exposés à Glascoed (GB), à deux emplacements en Australie, à Phoenix (Arizona) et à Singapour.

Eine Bewertung Künstlicher Bewitterungsmethoden zur Beurteilung der Wetterbeständigkeit von Anstrichfilmen

Zusammenfassung

Eine Übersicht wird gegeben von Untersuchungen über Bewitterungskurzprüfungsmethoden, die von dem "Voraussage über das Verhalten" Sub-Committee, früher gegenüber dem "Joint Services Research and Development Board" und seit kurzem gegenüber dem "Advisory Council of Materials" verantwortlich, durchgeführt worden waren.

In den Untersuchungen werden vergleichende Prüfungen mit einer

grossen Reihe von Anstrichfilmen beschrieben, die künstlicher Bewitterung in BS 3900 F3, ASTM E42 Type E, dem Tancyclus, Xenotest, Climatest sowie dem Emmaquaapparat ausgesetzt worden waren. Die Ergebnisse der Aussenbewitterung der in Glascoed (UK), zwei australischen Stationen, Phoenix (Arizona) und Singapur exponierten Anstrichfilme werden gleichfalls berichtet.

Introduction

Ref. 1-8

Several different artificial weathering cycles are available for assessing the durability of paint films. An earlier report¹ by the "Joint Services Research and Development Committee on Paints and Varnishes" described tests carried out in an apparatus operating with a 1600 watt DC enclosed carbon arc and water spray on a range of air-drying oleo-resinous and alkyd paint films. The success of this work led to the introduction of the method of test described in British Standard 3900 Part F3.² Weaver and Hipwood³ have described

tests in an ISO collaborative investigation in which the BS method was preferred for the assessment of durability of paint films exposed to natural weathering in the United Kingdom and at Singapore.

Experience with the BS cycle has shown that, although useful when comparing alkyd based paints exposed under temperate conditions, there are limitations when very durable materials are considered. The "Prediction of Performance" Sub-Committee has, over the past eight years, investigated the effectiveness of alternative artificial weathering equipment. The equipment investigated included ASTM E42 (1964)

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Type E,⁴ Dew cycle,⁵ Xenotest WL,⁶ Climatest⁷ and Emmaqua⁸ apparatus operating as described in Table 2, and comparisons of these have been made with BS 3900/F3.

The purpose of this paper is to summarise the results obtained in these cycles together with exposure out-of-doors, and to express the present view of the Sub-Committee in relation to the methods which have been examined so far.

Experimental

Paint systems examined

Thirty-four different paint systems of various colours were examined in four separate projects of the Committee. The types of media, substrates used and number of finishing paints tested for each class of medium, are given in Table 1. In each case, the surface preparation and cleaning of the substrate was carried out in accordance with the procedures given in British Standard 3900 Part A3. Where necessary, the primers and undercoats appropriate to the Service specifications or proprietary paints were included in the system.

Table 1
Media of finishing paints examined and substrates coated

Media	Substrates	No of paints in each class
1. Air-drying alkyds	Burnished steel, grit blasted steel, galvanised steel, chromated aluminium	14
2. Stoving alkyd	Burnished steel	1
3. 2 pack epoxy/polyamide	Chromated aluminium	1
4. 2 pack polyurethane (aromatic isocyanate)	Chromated aluminium	1
5. 2 pack polyurethane (aliphatic isocyanate)	Chromated aluminium	1
6. Epoxy ester	Burnished steel, grit blasted steel	1
7. Vinyl-toluene alkyd	Burnished steel, grit blasted steel	1
8. Chlorinated rubber	Burnished steel, grit blasted steel	1
9. Oleoresinous	Burnished steel	2
10. Oil-based (micaceous iron oxide)	Burnished steel, galvanised steel	1
11. Stoving amino/epoxide	Phosphated steel, grit blasted steel	2
12. Stoving solvent based acrylic	Phosphated steel	1
13. Stoving dispersion based acrylic	Phosphated steel	1
14. Emulsion paint (pva and acrylic)	Parana pine, western red cedar	6

Artificial weathering methods examined

Six different types of artificial weathering cycle were investigated by the Committee. These are described in Table 2.

Natural exposure tests

Natural exposure tests were conducted at five sites. Their characteristics and meteorological data (where available) are given in Table 3.

Table 2

Artificial weathering cycles

Specification or designation	Radiation source etc.	Operating cycles, dimensions etc.
1. BS 3900 Part F3	1600W DC enclosed carbon arc; borosilicate filter mounted off centre, arc to panel distance 35 to 80cm	Drum diam 120cm 1 rev/20min. Water spray 90° before nearest approach to carbon arc 4hr radiation + water spray 2hr radiation + fan cooling 10hr radiation + water spray 2hr radiation + fan cooling 5hr radiation + water spray 1hr stopped for cleaning etc.—Air temp not specified
2. ASTM E42 (1964) Type E (Atlas XW)	3000W AC open carbon arc with "Corex" filter, central, distance to panel approx 45cm	Drum diam 96cm 1 rev/min. Water sprays approx 27cm of circumference of drum 18hr (18min radiation + water) (102min radiation alone) 6hr no light or spray Black panel temp during dry period 63° ± 5 C
3. "Dew Cycle"	As above, no filter	Drum diam 96cm 1 rev/min 1hr radiation 1hr dark, back of panels cooled to induce condensation on front
4. "Xenotest" WL	1500W Xenon arc with borosilicate and IR filters, central, distance to panels 8cm	Drum diam, approx. 50cm 1 rev/15 secs. Panels rotated away from arc every 2nd rev 25min radiation 5min radiation + spray Air temp controlled 45 C
5. "Climatest"	32 × 20W fluorescent tubes 16 "Sun lamps", colour 12 + 16 "Blacklight, Blue" or 16 "actinic" colour 05. Tube to panel distance 10cm	Panel frame diam 150cm 1 rev/25secs 18min radiation + water 102min radiation Air temp controlled 45 C
6. "Emmaqua"	Sunlight intensified 8-10 times by polished aluminium mirrors	Continuous radiation during sunshine. Water spray (deionized) 7 periods daily of 10min duration. Air cooling Note: The panels received 229,300 langley's (266.3 watt-hr cm ⁻²) in the first 7 weeks. Mean annual total natural sunlight at this site 197,200 langley's (229 watt-hr cm ⁻²)

Table 3
Natural exposure sites

Type of site	Meteorological data (1960 for Phoenix, 1963 other sites)				
	Annual sun hours/radiation	Annual rainfall mm	Mean temperature/Range °C	Humidity	
				% time above 75% RH	% time below 60% RH
1. UK/Glascoed, Monmouthshire Temperate climate Rural	1,323 sun hrs. Approx. †100,000 langleys per annum*	1 016	Mean = 9°C Varied from -15°C to 27°C	75%	11%
2. Australia/ Innisfail Tropical wet Rural	2,472 sun hrs.	3 730	Mean = 23.5°C Varied from 7°C to 37°C	83%	9%
3. Australia/ Clonclurry Tropical dry Rural	3,466 sun hrs.	322	Mean = 26°C Varied from 4°C to 44°C	9%	81%
4. Singapore Navy site Equatorial wet Industrial Marine	1,844 sun hrs.	2 820	Mean = 27°C Varied from 21°C to 34.5°C	73%	4%
5. USA Phoenix Hot dry	4,135 sun hrs. Approx. 197,200 langleys per annum*	150	Mean = 22°C	Mean = 38.9% RH Most of the time below 60% RH	

*1 Langley = 1.162 mwh cm⁻²

†The langleys were not determined in 1963. In 1965 there were 1,399 sun hours and 100,000 langleys

Assessment of film deterioration

Ref. 9

A numerical form of assessment, of the pattern adopted by The Materials Quality Assurance Directorate,⁹ was used to record the type and degree of deterioration of paint films over the period of exposure to natural and artificial weathering.

The standards of assessment were:

- 0 = No failure
- 1 = Very slight failure
- 2 = Slight failure
- 3 = Definite failure
- 4 = Severe failure

Results and discussion

The work was conducted as four separate projects which at the time, were unrelated. As a result, the evidence is complete for all paints in the BS 3900/F3 cycle and natural weathering at Glascoed but not for the other artificial and natural exposure tests. One paint, however, is common to all four projects, the white two pack epoxy/polyamide (system 5) and serves as a link.

The results of all the exposures are combined in Table 4 and a number of generalisations can be made:

British Standard 3900 Part F3

A detailed comparison of the results from the BS 3900/F3 cycle and the out-door exposure at Glascoed (UK) confirms

that the cycle is useful for predicting chalking and colour changes. It is relatively slow however, and 12 weeks exposure is necessary to produce changes comparable with two years natural weathering in the UK.

The paints which exhibited early definite chalking (at least stage 3) after less than two years natural exposure at Glascoed are:

- grey epoxy ester (System 4)
- white epoxy/polyamide (System 5)
- white polyurethane (aromatic isocyanate) (System 6)
- green oleoresinous (System 21)
- emulsion paints (Systems 9-14)

The BS 3900/F3 cycle has induced definite to severe (stages 3 to 4) chalking on all the above systems, although the ratings given at the earlier times (six week BS 3900, one year natural exposure at Glascoed) do not necessarily correspond for the six emulsion systems.

Alkyd systems (25-34) were specially chosen to provide various degrees of colour change on outdoor exposure in UK. The BS 3900/F3 cycle after 12 weeks correlates reliably the extent of colour change compared with two years at Glascoed. Colour changes of certain other paint systems tended to be less severe than those obtained on natural exposure for two years at Glascoed and one year at sites overseas.

Checking, cracking and flaking failures of films were evident on exposure out-of-doors at Glascoed on the six

Table 4
Results of exposure tests

Type of finishing paint	Types of failures observed	Artificial cycles									
		Rating of failures in artificial cycles and natural exposure									
		BS 3900/F3		ASTM E, 8 weeks	Dew cycle		Xenotest, 12 weeks	Climatest, sun actinic, 675hr	Climatest, sun black- light, 675hr	Emmagua	
		6 weeks	12 weeks		89 hours	204 hours				7 weeks	14 weeks
1. Mid-brunswick green air-drying alkyd, glossy, DEF 1156	Loss of gloss ..	—	4	—	—	—	—	—	—	—	
	Colour change ..	—	4D	—	—	—	—	—	—	—	
	Chalking ..	—	0	—	—	—	—	—	—	—	
2. Royal blue air-drying alkyd, glossy, DEF 1044	Loss of gloss ..	1	2	4	—	—	—	—	2-3*	2-4*	
	Colour change ..	2D	2D	4F	—	—	—	—	2F	2-4*	
	Chalking ..	0	0	2	—	—	—	—	0	0	
3. Orange VT alkyd, glossy, proprietary	Loss of gloss ..	1	4	4	—	—	—	—	4	4	
	Colour change ..	1F	3F	4F	—	—	—	—	4F	4F	
	Chalking ..	0	1	3	—	—	—	—	3	3	
4. Light grey epoxy ester, glossy, proprietary	Loss of gloss ..	—	4	—	—	—	—	—	4	4	
	Colour change ..	—	3F	—	—	—	—	—	4F	4F	
	Chalking ..	—	4	—	—	—	—	—	3+	4	
5. White two-pack epoxy/polyamide, glossy, DTD 5555	Loss of gloss ..	4	4	4	0	4	4	4	4	4	
	Chalking ..	1	4	4	0	1	2	3	1	4	
6. White two-pack polyurethane (aromatic isocyanate), glossy, proprietary	Loss of gloss ..	0	4	4	—	—	4	4	—	4	
	Chalking ..	1	4	4	—	—	1	1	—	4	
7. White two-pack polyurethane (aliphatic isocyanate), glossy, DTD 5580	Loss of gloss ..	0	0+	—	3	4	—	4	4	0-1*	
	Chalking ..	0	0	—	0	3	—	0	0	1	
8. Pale blue, chlorinated rubber, glossy, DEF 1402	Loss of gloss ..	3	3	4	—	—	4	—	—	4	
	Colour change ..	3F	3F	3F	—	—	2F	—	—	1F	
	Chalking ..	0	3	2	—	—	0	—	—	0	
	Blistering (corrosion type) ..	0	1	1	—	—	1	—	—	0	
	..	0	1	1	—	—	1	—	—	0	
9. White matt pva co-polymer, emulsion, proprietary	Chalking ..	—	2	—	—	—	—	—	—	—	
	Checking and cracking ..	—	0	—	—	—	—	—	—	—	
	Blistering ..	—	0	—	—	—	—	—	—	—	
10. White matt homo-polymer, emulsion proprietary	Chalking ..	—	4	—	—	—	—	—	—	—	
	Checking and cracking ..	—	3	—	—	—	—	—	—	—	
	Flaking ..	—	0	—	—	—	—	—	—	—	
11. White matt pva homo-polymer, emulsion, experimental	Chalking ..	2	4	4	—	—	—	3	—	0	
	Checking and cracking ..	0	3	0	—	—	—	0	—	0	
12. White matt pva acrylate co-polymer, emulsion, experimental	Chalking ..	1	4	3	—	—	—	1	—	0	
	Checking and cracking ..	4	4	1	—	—	—	0	—	0	
13. White matt pva caprate co-polymer, emulsion, experimental	Chalking ..	0	4	3	—	—	—	1	—	0	
	Checking and cracking ..	2	2-3	0	—	—	—	0	—	0	
	Flaking ..	0	0	0	—	—	—	—	—	0	
14. White matt acrylic, emulsion, experimental	Chalking ..	1	3	0	—	—	—	1	—	0	
	Checking and cracking ..	1	2	1	—	—	—	0	—	4	
	Flaking ..	0	0	0	—	—	—	0	—	0	
	Blistering ..	0	0	0	—	—	—	0	—	0	
15. Grey micaceous iron oxide, CS 3005	Colour change ..	—	2F	—	—	—	—	—	—	1F	
	Chalking ..	—	2	—	—	—	—	—	—	2	
16. Black stoving epoxide, glossy, DEF 1059	Loss of gloss ..	0	1	—	—	—	—	—	—	1-2*	
	Colour change ..	0	1F	—	—	—	—	—	—	2-3	
	Chalking ..	0	0	—	—	—	—	—	—	1-2F	

F = Colour change by fading on unwashed areas of film.

D = Colour change by darkening on unwashed areas of film.

Natural weathering tests

0-4 scale, 0 = no failure; 4 = severe failure

1 year's exposure				2 years' exposure				3 yrs'	4 years' exposure			
Glascoed	Innisfail	Singapore	Cloncurry	Glascoed	Innisfail	Singapore	Cloncurry	Phoenix	Glascoed	Innisfail	Singapore	Cloncurry
1 3D 0	4 3D 2	4 4D 2	4 4F 2	2 3D 0	4 4D 2	4 4D 2	4 4F 2	— — —	4 3D 2	4 4D 3	4 4D 3	4 4F 3
0 2F 0	4 4F 3	4 4F 3	4 3F 3	0 2F 2	4 4F 4	4 4F 4	4 4F 3	4 4F 3	2 2F 2	4 4F 4	4 4F 4	4 4F 4
2 3F 0 0	4 4F 3 0	4 4F 3 0	4 4F 4 0	4 4F 2 0	4 4F 4 0	4 4F 3 0	4 4F 4 0	4 4F 4 0	4 4F 2 0	4 4F 3 0	4 4F 3 0	4 4F 4 0
4 4F 4	4 3F 4	4 3F 4	4 3F 4	4 3F 4	4 4F 4	4 4F 4	4 4F 4	4 4F 4	4 3F 4	4 4F 4	4 3F 4	4 4F 4
4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4
2 1	4 3	4 4	2 0	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4	4 4
0 0	— —	— —	— —	0 0	— —	— —	— —	4 1	0 0	— —	— —	— —
0 2F 0	4 3F 2	4 3F 1	4 4F 4	4 3F 2	4 4F 4	4 4F 3	4 4F 4	4 4F 4	4 3F 3	4 4F 4	4 4F 3	4 4F 4
0 0	2 2	2 0	0 0	3 3	3 3	3 3	4 4	1 1	4 4	4 4	4 4	4 4
4 0 0	4 0 1	2 0 0	2 0 0	4 0 1	4 0 0	4 0 0	4 0 0	— — —	4 1 0	4 0 1	4 4 0	4 2 0
4 0 0	4 0 0	4 0 0	3 0 0	4 0 0	4 0 0	4 0 0	4 0 0	— — —	4 2 0	4 4 4	4 4 0	4 4 0
3 0 2 0 3 0 0 0	— — — — — — — —	— — — — — — — —	3 1 — — — — — —	— — — — — — — —	— — — — — — — —	— — — — — — — —	— — — — — — — —	4 0 4 3 4 3 4 2	4 2 — — — — — —	— — — — — — — —	— — — — — — — —	— — — — — — — —
1D 1	2D 1	1D 1	2F 1	2D 2	2D 2	1D 2	2F 2	1F 1	1D 2	2D 2	1D 2	2F 2
0 1F 1	2 2F 1	2 2F 1	2 2F 2	0 2F 2	2 2F 3	2 2F 2	2 2F 2	4 2F 2	0 1F 2	3 2F 2	4 2F 3	3 2F 2

* = Uneven results in Emmaqua exposures.

Table 4
Results of exposure tests—continued

Type of finishing paint	Types of failures observed	Artificial cycles									
		Rating of failures in artificial cycles and natural exposure									
		BS 3900/F3		ASTM/E, 8 weeks	Dew cycle		Xenotest, 12 weeks	Climatest, sun/actinic, 675hr	Climatest, sun/black- light, 675hr	Emmaqua	
		6 weeks	12 weeks		89 hours	204 hours				7 weeks	14 weeks
17. Light grey stoving epoxide, glossy, DEF 1059	Loss of gloss ..	0	2	4	—	—	4	4	—	—	
	Colour change ..	0	3F	4F	—	—	3F	3F	—	—	
	Chalking ..	0	2	4	—	—	2	1	—	—	
18. Deep bronze green stoving alkyd, glossy, DEF 1045	Loss of gloss ..	—	4	—	—	—	—	—	—	—	
	Colour change ..	—	3D	—	—	—	—	—	—	—	
	Chalking ..	—	0	—	—	—	—	—	—	—	
19. Blue-grey stoving dispersion acrylic, glossy, proprietary	Loss of gloss ..	1	1	—	—	—	—	0	2	—	
	Colour change ..	1F	2F	—	—	—	—	1F	0	—	
	Chalking ..	0	0	—	—	—	—	0	0	—	
20. Blue-grey stoving solvent acrylic, glossy, proprietary	Loss of gloss ..	1	1	—	4	4	—	1	1	—	
	Colour change ..	1F	3F	—	3F	3-1 F	—	2F	3F	—	
	Chalking ..	0	0	—	0+	1	—	0	0	—	
21. Green oleoresinous air-drying paint, glossy, proprietary	Loss of gloss ..	4	4	4	3	4	4	4	4	—	
	Colour change ..	3F	4F	4F	2F	3F	4F	4F	4F	—	
	Chalking ..	1	3	3	0	1	1	3	2	—	
	Checking ..	0	4	1	0	0	4	3	4	—	
22. Signal Red oleoresinous air-drying paint, glossy, proprietary	Loss of gloss ..	1	2	4	3	4	4	4	4	—	
	Colour change ..	3D	3+D	4D	2D	4D	4D	3D	3D	—	
	Chalking ..	0	0	2	0	0	1	0	1	—	
	Checking ..	0	0	0	0	0	4	4	4	—	
23. Red oxide air-drying alkyd, glossy, BR spec.	Loss of gloss ..	2	2	4	—	—	0	3	—	—	
	Colour change ..	2D	2D	3D	—	—	0	2D	—	—	
	Chalking ..	0	0	1	—	—	0	1	—	—	
	Blistering ..	4	4	0	—	—	0	4	—	—	
24. Light grey air-drying alkyd, Weatherwork paint, glossy	Loss of gloss ..	1	4	—	—	—	—	3	—	—	
	Colour change ..	1F	1F	—	—	—	—	1F	—	—	
	Chalking ..	0	3	—	—	—	—	1	—	—	
25. Brunswick green air-dry alkyd, glossy (organic pigments)	Loss of gloss ..	1	2	—	1	4	—	4	4	—	
	Colour change ..	1D	2D	—	1F	2F	—	2F	2F	—	
	Chalking ..	0	0	—	0	1	—	—	1	—	
26. Brunswick green air-dry alkyd, glossy (inorganic pigments)	Loss of gloss ..	1	1	—	1	4	—	4	4	—	
	Colour change ..	3D	4D	—	2F	3F	—	3F	4F	—	
	Chalking ..	0	0	—	0	0	—	1	0	—	
27. Red air-dry alkyd, glossy (organic pigments)	Loss of gloss ..	1	1+	—	1	4	—	2	4	—	
	Colour change ..	2D	3D	—	2+ F	4F	—	3F	4F	—	
	Chalking ..	0	0	—	0	0+	—	0	1	—	
28. Red air-dry alkyd, glossy (inorganic pigments)	Loss of gloss ..	1	1	—	1+	4	—	4	4	—	
	Colour change ..	2D	4D	—	2+ D	4D	—	4F	4F	—	
	Chalking ..	0	0	—	0	0+	—	0	0+	—	
29. Orange air-dry alkyd, glossy (organic pigments)	Loss of gloss ..	0	1	—	3	4	—	2	3	—	
	Colour change ..	2D	3D	—	3F	3F	—	3D	3D	—	
	Chalking ..	0	0	—	0	0	—	0	0	—	
30. Orange air-dry alkyd, glossy (inorganic pigments)	Loss of gloss ..	0	1	—	1	4	—	4	4	—	
	Colour change ..	2D	4D	—	3F	4F	—	4F	4F	—	
	Chalking ..	0	0	—	0	0	—	0	0	—	
31. Pink air-dry alkyd, glossy	Loss of gloss ..	1	3	—	3	4	—	4	4	—	
	Colour change ..	3F	4F	—	2F	3F	—	3F	4D	—	
	Chalking ..	0	0	—	0	0-1	—	1	0	—	
32. Pale blue air-dry alkyd, glossy	Loss of gloss ..	1	3	—	3	4	—	4	4	—	
	Colour change ..	1D	3D	—	2F	3F	—	3F	3D	—	
	Chalking ..	0	0	—	0	1	—	1	0	—	

F = Colour change by fading on unwashed areas of film.

D = Colour change by darkening on unwashed areas of film

Natural weathering tests

0-4 scale, 0 = no failure; 4 = severe failure

1 year's exposure		2 years' exposure				3 yrs'	4 years' exposure					
Glascoed	Innisfail	Singapore	Cloncurry	Glascoed	Innisfail	Singapore	Cloncurry	Phoenix	Glascoed	Innisfail	Singapore	Cloncurry
2	—	—	—	3	—	—	—	—	4	—	—	—
2F	—	—	—	2F	—	—	—	—	3F	—	—	—
2	—	—	—	3	—	—	—	—	3	—	—	—
—	—	—	—	—	—	—	—	4	4	—	—	—
—	—	—	—	—	—	—	—	4F	4F	—	—	—
—	—	—	—	—	—	—	—	2	1	—	—	—
0	—	—	—	0	—	—	—	1	1+	—	—	—
2F	—	—	—	3F	—	—	—	3F	3F	—	—	—
0	—	—	—	0	—	—	—	0	0	—	—	—
0	—	—	—	0	—	—	—	4	4	—	—	—
2F	—	—	—	3F	—	—	—	4F	4F	—	—	—
0	—	—	—	0	—	—	—	2	1	—	—	—
4	—	—	—	4	—	—	—	—	4	—	—	—
4F	—	—	—	4F	—	—	—	—	4F	—	—	—
3	—	—	—	3	—	—	—	—	3	—	—	—
0	—	—	—	3	—	—	—	—	4	—	—	—
3	—	—	—	4	—	—	—	—	4D	—	—	—
4D	—	—	—	4D	—	—	—	—	2	—	—	—
1	—	—	—	1	—	—	—	—	4	—	—	—
2	—	—	—	4	—	—	—	—	—	—	—	—
0	—	—	—	1	—	—	—	—	—	—	—	—
2D	—	—	—	2D	—	—	—	—	—	—	—	—
1	—	—	—	1	—	—	—	—	—	—	—	—
4	—	—	—	4	—	—	—	—	—	—	—	—
1F	—	—	—	3	—	—	—	—	—	—	—	—
0	—	—	—	1F	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
2D	—	—	—	2D	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
0	—	—	—	3D	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
2F	—	—	—	3F	—	—	—	—	—	—	—	—
0	—	—	—	1	—	—	—	—	—	—	—	—
0	—	—	—	4D	—	—	—	—	—	—	—	—
3D	—	—	—	0	—	—	—	—	—	—	—	—
0	—	—	—	2	—	—	—	—	—	—	—	—
0	—	—	—	3D	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
1	—	—	—	1	—	—	—	—	—	—	—	—
4D	—	—	—	4D	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
0	—	—	—	1	—	—	—	—	—	—	—	—
3F	—	—	—	4F	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—
0	—	—	—	1	—	—	—	—	—	—	—	—
3F	—	—	—	3F	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—

* = Uneven results in Emmaqua exposures.

Table 4
Results of exposure tests—continued

Type of finishing paint	Types of failures observed	Artificial cycles									
		Rating of failures in artificial cycles and natural exposure									
		BS 3900/F3		ASTM/E, 8 weeks	Dew cycle		Xenotest, 12 weeks	Climatest, sun/actinic, 675hr	Climatest, sun/black- light, 675hr	Emmaqua	
		6 weeks	12 weeks		89 hours	204 hours				7 weeks	14 weeks
33. Pale yellow air-dry alkyd, glossy	Loss of gloss ..	1	3	—	4	4	—	4	4	—	—
	Colour change ..	3F	4F	—	3F	4F	—	4F	3D	—	—
	Chalking ..	0	0	—	0+	1	—	1	0	—	—
34. Pale green air-dry alkyd glossy	Loss of gloss ..	1	3	—	3	4	—	4	4	—	—
	Colour change ..	3F	4F	—	3+F	4F	—	3+F	3F	—	—
	Chalking ..	0	0	—	1	1	—	1	0	—	—

F = Colour change by fading on unwashed areas of film.

D = Colour change by darkening on unwashed areas of film

emulsion paint systems (9 to 14), green oleoresinous (21) and signal red oleoresinous (22). A comparison of these results with those obtained in BS 3900/F3 showed that the ratings for emulsion paint films do not correspond and that the artificial cycle fails to identify the severe checking failure of system 22. The cycle was effective, however, for inducing checking failure of system 21.

The severe blistering of the red oxide alkyd (23) reported within the first year of natural weathering was identified within six weeks in BS 3900/F3. However, the artificial cycle failed to induce the blistering reported in natural exposure tests at the overseas sites on the chlorinated rubber (8) but was quite fair for Glascoed. In the latter case, it should be noted that the blisters were not water blisters but were caused by corrosion. There was also a minor failure to predict slight blistering of one of the emulsions (9) at Innisfail, but since this was itself not repeated at any of the other sites it probably can be discounted. In one other case, an acrylic emulsion (14) showed considerable blistering at the end of four years exposure (Glascoed). This failure was absent in the BS 3900/F3 tests which were terminated at 12 weeks, so that it is impossible to say whether blistering would have developed if the test had been continued.

No failures were observed with the white polyurethane (aliphatic isocyanate) (7) in BS 3900/F3 and this result is in good agreement with Glascoed. The black stoving epoxide (16) on the other hand showed only slight change in BS 3900/F3 but showed chalking failure on exposure at all the outdoor sites. The white epoxy/polyamide (5) may have been a little slower in developing chalking in BS 3900/F3, but correlated well in a test after 12 weeks exposure.

In general, the repeatability of tests in the BS 3900/F3 cycle (with each successive investigation) and reproducibility (with tests conducted in different laboratories) have been found very satisfactory given rigid adherence to the operating instructions.

ASTM E42 (1964) Type E

This cycle is judged by comparison with BS 3900/F3 and with natural exposure on the results obtained in tests of systems, 2, 3, 5, 6, 8, 11, 12, 13, 14, 17, 21, 22 and 23. The period of exposure in the cycle was limited to eight weeks and although this is a shorter period than that adopted for tests by the

BS method, the film deterioration was in most cases more severe in the ASTM cycle, apart from the tests on emulsion paints.

The cycle predicted correctly the chalking and colour change of the paint films tested and the more severe results corresponded quite closely with those obtained in the overseas exposures. The three pva emulsion paints (11-13) performed differently at the hot dry Phoenix site compared with Glascoed and the ASTM cycle tended to correlate better than BS 3900/F3 with Phoenix, but the BS cycle was preferred for comparisons with Glascoed. The ASTM cycle, however, failed to predict the severe chalking and cracking of the acrylic emulsion (14) after an eight week exposure test.

The ASTM cycle may be regarded, therefore, as more reliable than BS 3900/F3 for comparisons of paint films exposed in hot sunny climates. It is felt that because of the relatively long dry phases in the cycle, the ASTM method failed to identify the severe blister failure of red oxide alkyd (23). Unfortunately, this paint was not included in any of the overseas exposure tests and so further comment is not possible on the correlation with blistering failures, although the indications are that longer wet phases are desirable for comparisons with temperate climates.

Repeatability and reproducibility of tests by ASTM E42 Type E were not investigated, but the specification is very precise and it must be assumed that, as with BS 3900/F3, good repeatability and reproducibility is possible by observing all requirements of the specification.

Dew cycle (Atlas XW-R)

Paint systems 5, 7, 20, 21, 22, 25 to 34 (inclusive) were tested in this cycle for 89 and 204 hours exposure.

The cycle operates with the same arc lamp as ASTM E42 Type E, but unlike the ASTM method, no filters are used. There is, therefore, a considerable amount of unrealistic short wave radiation (below 290nm) present. In addition, the ratio of wet to dry phases is higher in the Dew cycle.

The Dew cycle did not correlate well with natural weathering at either temperate or hot sites and failed to predict the marked chalking which occurred with the white epoxy/polyamide (5) at every site. Moreover, it predicted marked

Natural weathering tests

0.4 scale, 0 = no failure; 4 = severe failure

1 year's exposure				2 years' exposure				3 yrs'	4 years' exposure			
Glascoed	Innisfail	Singapore	Cloncurry	Glascoed	Innisfail	Singapore	Cloncurry	Phoenix	Glascoed	Innisfail	Singapore	Cloncurry
0	—	—	—	1	—	—	—	—	—	—	—	—
3F	—	—	—	3F	—	—	—	—	—	—	—	—
0	—	—	—	1	—	—	—	—	—	—	—	—
0	—	—	—	1	—	—	—	—	—	—	—	—
3F	—	—	—	4F	—	—	—	—	—	—	—	—
0	—	—	—	0	—	—	—	—	—	—	—	—

* = Uneven results in Emmaqua exposures.

chalking with the polyurethane (aliphatic isocyanate) (7) which did not occur on natural weathering and completely reversed the known characteristic behaviour of the polyurethane system. The cycle also failed to produce the severe film deterioration observed out-of-doors with the two oleoresinous paints (21 and 22) within the exposure period.

Repeatability is probably similar to the ASTM method on which the apparatus is based.

Xenotest WL apparatus

Ref. 10

Systems 2, 3, 5, 6, 8, 17, 21, 22 and 23 were exposed for 12 weeks in the Xenotest WL apparatus. Over the same exposure period, the cycle was somewhat more effective than the BS method in reproducing colour change, chalking and checking of the two oleoresinous paints (21 and 22), but rather less so for the other paints tested, particularly in respect of chalking.

Repeatability tests in the cycle were not investigated, but it is reported¹⁰ that with ageing of the lamp there is a reduction of energy and change in the spectral distribution. If this is so, then repeatability may be affected by the age of the lamps if the same exposure time is used. Similar results should be obtained in the Xenotest 450 apparatus, which is now favoured in preference to the Xenotest WL.

Climatest apparatus

Two series of tests were conducted in the Climatest apparatus. As the fluorescent tubes are known to have a limited life, in order to ensure consistency of emission, two pairs of tubes were replaced with new ones during each week of exposure. All tests were conducted at an air temperature of 45°C and a relative humidity of not less than 95 per cent.

In the first series, sixteen Sunlamp and sixteen Actinic tubes were used to irradiate the paint films during a 675 hour (28 day) exposure period. Systems 2, 3, 5, 6, 7, 11, 12, 13, 14, 17 and 19-34 (inclusive) were tested.

In the second series, which was carried out in a different (but identical type) apparatus, sixteen Sunlamp and sixteen Blacklight Blue tubes were used on systems 5, 7, 19, 20, 21, 22 and 25-34 (inclusive).

The difference between results obtained with different lamp combinations was slight, but the tendency was for the Blacklight Blue combination to be more severe for colour change and loss of gloss, but chalking more severe for the Actinic combination.

When results from the Climatest exposures are compared with BS 3900/F3 (12 weeks) and Glascoed, it is observed that the loss of gloss is generally more severe in the Climatest than either of these other cycles and, in this respect, it was less reliable than BS 3900/F3. The cycle also failed to induce chalking in the polyurethane (aromatic isocyanate) (6) but caused loss of gloss in the polyurethane (aliphatic isocyanate) (7) which reversed the order of performance. It did, however, induce checking in the signal red oleoresinous paint (22), which BS 3900/F3 failed to do, but chalking of the emulsion paints was not severe enough.

Panels 45cm in length were used in the Climatest apparatus and the investigations confirmed that surface deterioration was evenly induced over the total area of exposed film. Reproducibility and repeatability were not investigated.

Emmaqua

The "Emmaqua" programme included 7 and 14 week exposure tests in the apparatus. Systems 2, 3, 4, 5, 6, 7, 8, 11, 12, 13, 14, 15, 16, 18, 19 and 20 were tested in this programme.

Apart from tests of the four emulsion paint systems 11 to 14, results from the 14 week Emmaqua exposures were very slightly more severe than those obtained in the 12 week BS 3900/F3 tests. The pva emulsion paints showed less severe chalking in Emmaqua than occurred on natural weathering at Glascoed or Phoenix and the cycle was less reliable than BS 3900/F3 in this respect. However, the acrylic emulsion showed very considerable checking and cracking in Emmaqua, which did not occur to anything like the same extent at the natural sites even on long term exposure.

Emmaqua correlated reasonably with hot sunny exposures but the test produced an uneven deterioration over the exposed surface. Repeatability was not investigated, but some variation can be expected according to season and climatic changes.

Conclusions

The work described was carried out as four unrelated exercises which have been combined to give a broad comparison of various artificial weathering methods and several natural exposure sites. As a result, there are some gaps in the comparisons, but a number of observations can be made.

A summarised view of the cycles tested in these investigations is as follows:

The method of test described in BS 3900 Part F3 is considered useful for predicting chalking and colour change failures on paints exposed to temperate climatic conditions. A 12-week exposure period is considered desirable to identify failures likely to occur within the first two years of out-door exposure.

The method described in ASTM E42, Type E produces more severe film deterioration than that produced in BS 3900/F3 within a shorter time of exposure, and this method is to be preferred for comparisons with natural exposure in tropical and hot sunny climates. There may be deficiencies arising from defects induced by damp conditions, for example blistering, but further comparisons would be necessary to confirm this point.

Durability predictions based on the "Dew cycle" are considered unreliable. Shortwave radiation (below 290nm) is thought to be responsible for inducing the wrong order of performance of the paints which were tested in this investigation.

The cycle used in the "Xenotest WL" apparatus has been shown to be slow in inducing film deterioration, but when breakdown has occurred there have been no abnormalities.

The "Climatest" apparatus produced more severe loss of gloss in paint films than those obtained with BS 3900/F3 within a shorter period of exposure, but other types of film failure were less advanced. Correlation with natural exposure (UK) at two years tended to be less close than with the BS method.

"Emmaqua" is considered more useful for acceleration of film deterioration observed in hot sunny climates, such as

Phoenix, Arizona. However, the test induced an uneven deterioration over the area of test film.

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Flow and ageing of polyvinyl chloride pastes

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Summary

The theoretical treatment of flocculation kinetics and dispersion stabilisation is reviewed and applied to practical polyvinyl chloride (pvc) paste systems. The results suggest that at the particle concentration concerned in pvc pastes, the role of the electrical effects is secondary to that of the steric barrier. The modification of latex with fatty alcohols or esters of higher fatty acids can improve both the initial viscosity and ageing. The paper also

describes the effects of (a) the adsorbed layer, (b) the effective solid volume fraction, (c) the particle size, and (d) the particle concentration on the ageing and flow behaviour of pvc pastes. A concept of particle/particle interaction and the development of a new dynamic network model can be used to explain the flow behaviour of pvc pastes.

Keywords

Types and classes of coatings and allied products

plastisol coating

Properties, characteristics and conditions primarily associated with materials in general

particle size
rheological property
yield value

Les caractéristiques d'écoulement et de vieillissement des pâtes de chlorure de polyvinyle

Résumé

On passe en revue le traitement théorique de la cinétique de la flocculation et aussi de la stabilisation des dispersions et l'on l'applique aux systèmes de pâtes de chlorure de polyvinyle qui se trouvent dans le domaine pratique. Les résultats suggèrent qu'à la concentration particulière dont il s'agit aux pâtes de cpv., le rôle des effets électriques est secondaire à celui de la barrière stérique. La modification du latex par les alcools gras ou les esters des acides gras supérieures peut effectuer une amélioration de la viscosité initiale ainsi que du vieillissement.

Dans cet exposé on décrit d'ailleurs les effets suivants: (a) de la couche adsorbée, (b) de la teneur effective en matière sèche par volume, (c) de la grandeur particulière, (d) de la concentration particulière, sur le vieillissement et les propriétés d'écoulement des pâtes de cpv. On peut expliquer ces propriétés en utilisant à la fois un concept d'interaction particulière et le développement d'un nouveau modèle de réseau dynamique.

Verlauf und Alterung von Polyvinylchloridpasten

Zusammenfassung

Eine Betrachtung der theoretischen Behandlung von Ausflockungskinetik und Dispersionsstabilisierung, angewandt auf in der Praxis benutzte Polyvinylchlorid (PVC) Pastensysteme. Die Resultate legen nahe, dass bei der für PVC Pasten in Frage kommenden Partikelkonzentration die von elektrischen Wirkungen gespielte Rolle kleiner ist, als die durch sterische Hinderung gespielte. Die Modifizierung von Latex mit Fettalkoholen oder Estern höherer Fettsäuren kann sowohl die anfängliche Viskosität als auch die Alterung günstig beeinflussen.

In der Abhandlung werden ebenfalls die folgenden Auswirkungen beschrieben

- (a) der adsorbierten Schicht
- (b) der tatsächlichen Feststoffvolumenfraktion
- (c) der Teilchengröße
- (d) der Teilchenkonzentration auf Alterungs- und Verlaufsverhalten von PVC Pasten.

Ein Konzept der Wechselwirkung zwischen Teilchen und Teilchen und die Entwicklung eines neuen dynamischen Modells der Vernetzung kann verwandt werden, um das Verlaufsverhalten von PVC Pasten zu erklären.

Introduction

Ref. 1, 2

The rheology of pvc pastes has been the subject of rather intensive investigation for many years, because of their practical importance. Pvc pastes, or plastisols, are dispersions of powders of small particle size in a low viscosity plasticiser, such as di-octyl phthalate (dop), tri-cresyl phosphate (tcp) and di- α -naphthyl phthalate (dap). The synthetic leather industry with its fabric coating processes still consumes a larger proportion of pastes than the section of processors who employ casting and dipping methods. A knowledge of the behaviour of pvc powder in plasticiser is an extremely important factor in production for those using the latter processes.

The main problem associated with pvc pastes is their instability with respect to time, ie the change in viscosity with time during storage. The initial viscosity of a pvc paste, and

the speed at which it ages, indicates its suitability for processing and also determines shelf life. The practical method^{1, 2} of judging the stability of the pvc paste is to determine the change in viscosity after storage for several days at room temperature.

The flow properties of vinyl plastisols affect operations such as spreading, spraying, coating, moulding, and dipping.

Unfortunately, it is a very common experience that the ageing problem tends to arise for no apparent reason, and whilst a number of remedies may be applied to the system, frequently with some measure of success, the cause of the phenomenon has never been clearly understood. The present research programme was undertaken to investigate a number of factors which might serve to amplify the existing knowledge and to give a better understanding of ageing behaviour of pvc plastisols.

Experimental

Ref. 3, 4

Formulation details

A relatively simple composition was chosen for this study, comprising a mixture in the proportions:

Polymer	100g
Plasticiser	66.6ml

Since the density of the plasticiser is 0.901, the pastes correspond to 60 per cent solids on a weight/weight basis; they were prepared in a Hobart mixer.

Viscosity and thixotropic measurements

Measurements at low shear rates were made using a Ferranti-Shirley cone and plate viscometer. A 0.553° cone of 7cm diameter was used. The temperature was maintained at 25°C. The flow curves were measured by increasing the rate of shear in equal steps to a maximum of 100sec⁻¹ and then decreasing the rate of shear in the same equal steps.

Adopting this procedure, the pvc plastisol systems generally describe a thixotropic hysteresis loop of the type shown in Fig. 1. The various systems are compared in terms of the following values obtained from the flow curve:

- Apparent viscosity: the apparent paste viscosity is, in fact, recorded, since suspensions of this type exhibit varying viscosity behaviour depending on the method and range of shear used.
- Aged viscosity: this is the net change in viscosity with time during storage.
- Area of hysteresis loop: this is the measure of paste structure. The loops were recorded by increasing the rate of shear in equal steps to a maximum of 100sec⁻¹ and then decreasing the rate of shear in the same equal steps.

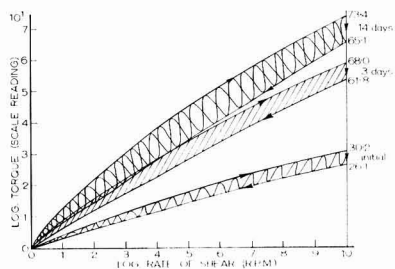


Fig. 1. Build up of structure

Electrical and zeta potential measurements

The electrical conductivity, dielectric constant and volume resistivity of pvc pastes were measured, using a 3-terminal liquid electrode system and a General Radio capacitance bridge type 1620A. Measurements were made at room temperature at 1 kHz frequency. Description and details can be found in a General Radio experiment³.

The zeta potential⁴ was measured by imposing a potential gradient across the system being studied. Small charged particles suspended in the system move under the influence of

the applied potential gradient and from the deposition rate the zeta potential can be calculated.

Free plasticiser determination

A specially designed dilatometer was used for the determination of free plasticiser. It consisted of a 50cc bottle fitted with a KB-14 clear fit socket and cone containing a capillary 6cm long (graduated in mm) and a bore of 2mm. The dilatometer was filled with recently prepared air-free paste and kept in a water bath maintained at 23.5°C. The level of paste in the capillary was recorded every day over a period of two weeks. Differences in the readings gave the apparent loss of plasticiser.

Results and discussion

Rheological studies

Undoubtedly, the major source of the difficulties surrounding the ageing and rheology of dispersed systems such as pvc plastisols is the fact that no adequate, fundamental concept of the interaction taking place between polymer particles and plasticiser has been forthcoming. Furthermore, there has been a failure to appreciate the full significance and effects of the coexistence of viscoelasticity, plastoelasticity and time dependent properties on the ageing and flow behaviour of pastes.

The fundamental ideas of the new theory will first be presented and it will then be demonstrated that these ideas lead directly to structural features which are in line with experimental observations. The stabilisation of small particles in plasticiser by an adsorbed layer mechanism will then be discussed. Finally, the significance of the effective solids volume fraction on flow behaviour of paste will be emphasised.

Dynamic network model

A three-dimensional dynamic network model is developed from the fact that some interaction between the pvc powder particles and the plasticiser molecules is necessary. This interaction must not be too weak, otherwise the individual components will separate out and the sedimentation of pvc particles will create another problem. The finely divided polymer particles are caused to flocculate and agglomerate by agitation with plasticiser, which preferentially wets the surface of the particles. The importance of flocculation as a factor determining the flow properties of pvc pastes has been underestimated. The physical properties of such a system can be related to the extent and character of the interface between solid and liquid. The author emphasises that the interfacial energy will influence such interrelated characteristics as flow and ageing. In many dispersed systems, at concentrations of practical importance, the rheological properties depend on the attractive and repulsive forces. The interaction of these forces between dispersed particles determines the nature of the paste structure as shown in Fig. 2. Both attractive and repulsive forces result in an increased viscosity. In order to achieve a stable dispersed system, such as that shown in Fig. 2(a), the forces of repulsion between two particles must exceed the forces of attraction which cause particles to adhere on contact. In a flocculated system (Fig. 2(b)) to which is attributed an increased viscosity, the attractive forces predominate. Particle aggregation due to interaction of the plasticiser molecules and the pvc particles, should result in a viscosity which is shear dependent as well as time dependent. Such a model will also account for normal stress

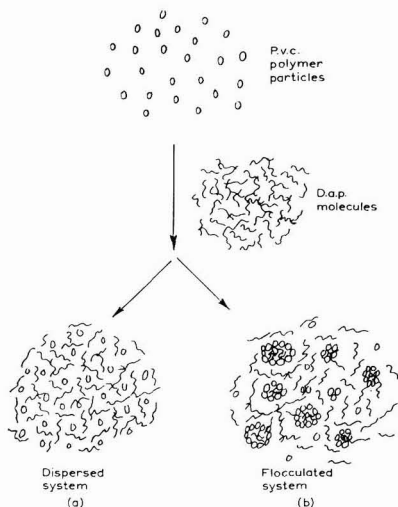


Fig. 2. Interaction of the forces between dispersed particles determines the nature of the paste structure

differences, viscoelasticity, yield value and build up of a time dependent structure. It is supported by experimental observations. The interaction energy curves and flocculation kinetics will be discussed later.

The flow curve loop for a paste is shown in Fig. 1. The classical hysteresis loop generated by the sample is sensitive to measurement conditions and, particularly, to the rate of change of shear rate or the time of shearing; however, for a comparative study it is adequate to standardise the procedure to illustrate the rheological effects produced by structural changes in a system. The various systems are compared in terms of the following values obtained from the flow curve:

- (a) apparent viscosity.
- (b) area of hysteresis loop.
- (c) yield value.

The plotting of rheological data as log shear stress vs log shear rate provides a useful means of estimating the yield value

stress by extrapolation to very low shear rates (10^{-2} sec^{-1}). The extent of paste structure was measured by cutting out the areas described by loops on Fig. 1 and weighing the papers. The paste structure in general was found to increase with time.

The results obtained on the viscosity and ageing behaviour of the pastes are given in Table 1 and shown graphically in Fig. 3, as rheograms of paste viscosity at a shear rate of 4 sec^{-1} against ageing time. The samples 10, 6 and 1 can be seen from Table 1 to be inferior to samples 3 and 2. It is also clear from Table 1 that both the initial viscosity and the overall ageing rate, ie rate of change of viscosity as a function of initial viscosity, are far in excess of those observed for the samples 3, 5, 9 and 2. This difference is particularly marked in sample 7, where the viscosity after four weeks is many times that of samples 2 and 3. The important feature of the table is that the measurements at high shear rates indicate ageing behaviour of pvc pastes and that the shear rates relating directly to ageing are likely to be very small, of the order of a few reciprocal seconds.

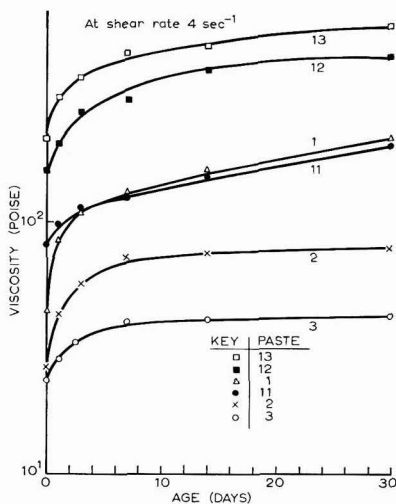


Fig. 3. Ageing behaviour of pastes

Table 1
Viscosity and ageing behaviour of pvc pastes at 25°C

Sample no	Latex particle size (μ)	At shear rates									
		4sec ⁻¹				100sec ⁻¹					
		Initial	1 week	2 weeks	4 weeks	Aged	Initial	1 week	2 weeks	4 weeks	Aged
1	0.1 to 1	44.2	130	161	226	181.8	31.4	70.7	76	95.7	64.3
2	Competitive polymer	26.0	72.8	73.2	78	52.0	23.0	56.2	57.4	60.3	37.3
3	0.12 to .95	23.4	39.0	41.6	41.6	18.2	21.2	34.3	37.4	40.6	19.4
4	60% 0.3 40% 1	49.4	135	177	250	200.6	38.7	30.6	102	108	69.3
5	25% 0.3 75% 1	20.8	24.7	31.2	38.8	18.0	18.4	20.6	30.8	36.8	18.4
6	0.8-1 + 10% fines	44.2	70.2	78.0	88.4	44.2	51.2	88.6	92.6	94.2	43.0
7	0.15-0.25	291	1,380	1,730	—	$\geq 1,439$	—	—	—	—	—
8	60% 0.95 40% 0.25	36.4	107	132	199.6	163.2	28.7	74.0	86.8	102.6	73.9
9	Same as above + Sytol AF (ester with higher fatty acid chain) added in the latex	23.4	57.2	67.6	82.4	55.0	24.1	48.9	52.0	63.2	39.1
10	50% 0.5 50% 0.15	—	—	—	—	—	—	—	—	—	—

Yield value

As the polymer/plasticiser ratio of a dispersion increases, flocculation may occur due to particle/plasticiser and particle/particle interaction. The particle structure is connected and completely continuous but not oriented. Flow cannot take place until the applied stress exceeds the force of flocculation. On the scaffolding theory, the yield value can be obtained directly from the strength and number of links. If each link is able to withstand, on average, a force in the direction of shear of f_c , and can transmit this force through a distance x in the direction of shear, then the yield value is given in dyne/cm² by:

$$F_y = n_o \times f_c$$

where n_o is the number of links per cm³

It is shown in Table 2 that not only does the viscosity increase with time, but also both the yield value and structure increase. The paste which ages rapidly appears to have a higher initial yield value and more structure. The increase in structure and yield value of the inferior paste over a period of 14 days are more than four times and six times respectively those recorded for sample 3 and the other pastes which age well.

to particle/particle interaction as the packing became more open.

The paste 6 made from a large particle size polyvinyl chloride latex ages less rapidly than a similar system 7 of small particle size. The increase in viscosity for paste 7 over 14 days is more than 30 times that recorded for paste 6. The reason for this is that the surface area is greater for a small particle paste and the hydrodynamic viscosity would be expected to increase more rapidly. In addition, the structural contribution to viscosity depends upon the square of the number of particles per cubic centimetre. This structural contribution rises rapidly with decreasing particle size and an increase in the number of particles per cubic centimetre is, therefore, more effective. From the discussion, it follows that the incorporation of small particles with large ones should give the desired flow behaviour and slow ageing of pvc pastes. Replacement of some of the larger particles by smaller ones increases the distance between the large particles, as would also be the case if the plasticiser content were raised. The smaller particles move about between the larger particles, and as they are mobile they do not prevent the larger particles from slipping past each other. The importance of primary particle size distribution in the rheological proper-

Table 2
Increase in viscosity, structure and yield value with time

Paste no	Time (days)	Viscosity (poise)		Increase in structure (g)	Yield value (dynes/cm ²)	Amplitude (Mn)	Paste density (g/cm ³)
		4sec ⁻¹	100sec ⁻¹				
1	Initial	44.2	31.4	0.146	11.5	2.78	1.2050
	3 Days	107	61.3	0.266	41	3.35	
	7 Days	130	70.7	0.347	60	4.98	
	14 Days	161	76.0	0.490	70	6.50	
3	Initial	23.4	19.4	0.042	6.0	1.62	1.2020
	3 Days	30.6	26.0	0.047	6.3	1.67	
	7 Days	39.0	34.3	0.051	6.5	1.72	
	14 Days	41.6	37.4	0.058	6.9	1.94	

Effect of particle size

Ref. 5

The effect of particle size on viscosity is illustrated in Fig. 4, which shows that paste viscosity at low shear rates decreases as the particle size is increased. The paste 6, which consisted of larger particle size and was not blended with smaller particles, shows shear thinning followed by shear thickening. When a dilatant suspension is sheared the particles move out of a relatively close packed arrangement into a more open packing. The need for additional liquid caused by the change in packing may give a tendency for the surface to dry up, accounting for the dilatant behaviour. Shear thinning at low shear rates was explained by the breakdown of clumps or networks (Fig. 2), whilst the subsequent dilatancy was due

ties of pvc paste has been considered by Peggion and his co-workers⁵.

Flocculation and dispersion stability

Ref. 6, 7

A considerable amount of theoretical work exists on the subject of flocculation kinetics and the stabilisation of dispersions⁶. Whilst a large number of practical investigations has been made of the stability of aqueous dispersions, the amount of published work on non-aqueous systems is very limited. In the following section, the theoretical approach to flocculation and its possible effects on viscosity and ageing of pvc pastes will be reviewed, followed by application of the principles to the results obtained with pvc powder particles in plasticiser (dap).

An unstabilised dispersion will flocculate by collision of the primary particles, as a result of their thermal energy motion. This reduces the total effective number of particles in the system. The theory of rapid flocculation developed by von Smoluchowski⁷ postulates that each collision is effective in producing a flocculate, so that a decrease N_1 in the number of primary particles is given by

$$-(dN_1/dt) = 8\pi D R N_1^2 \dots\dots\dots(1)$$

where R is the collision radius of the particles and D is the diffusion coefficient

$$D = kT/(\delta\pi \eta a) \dots\dots\dots(2)$$

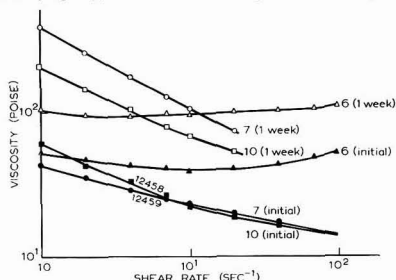


Fig. 4. Effect of particle size on viscosity



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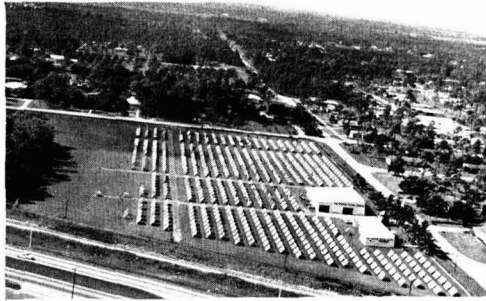
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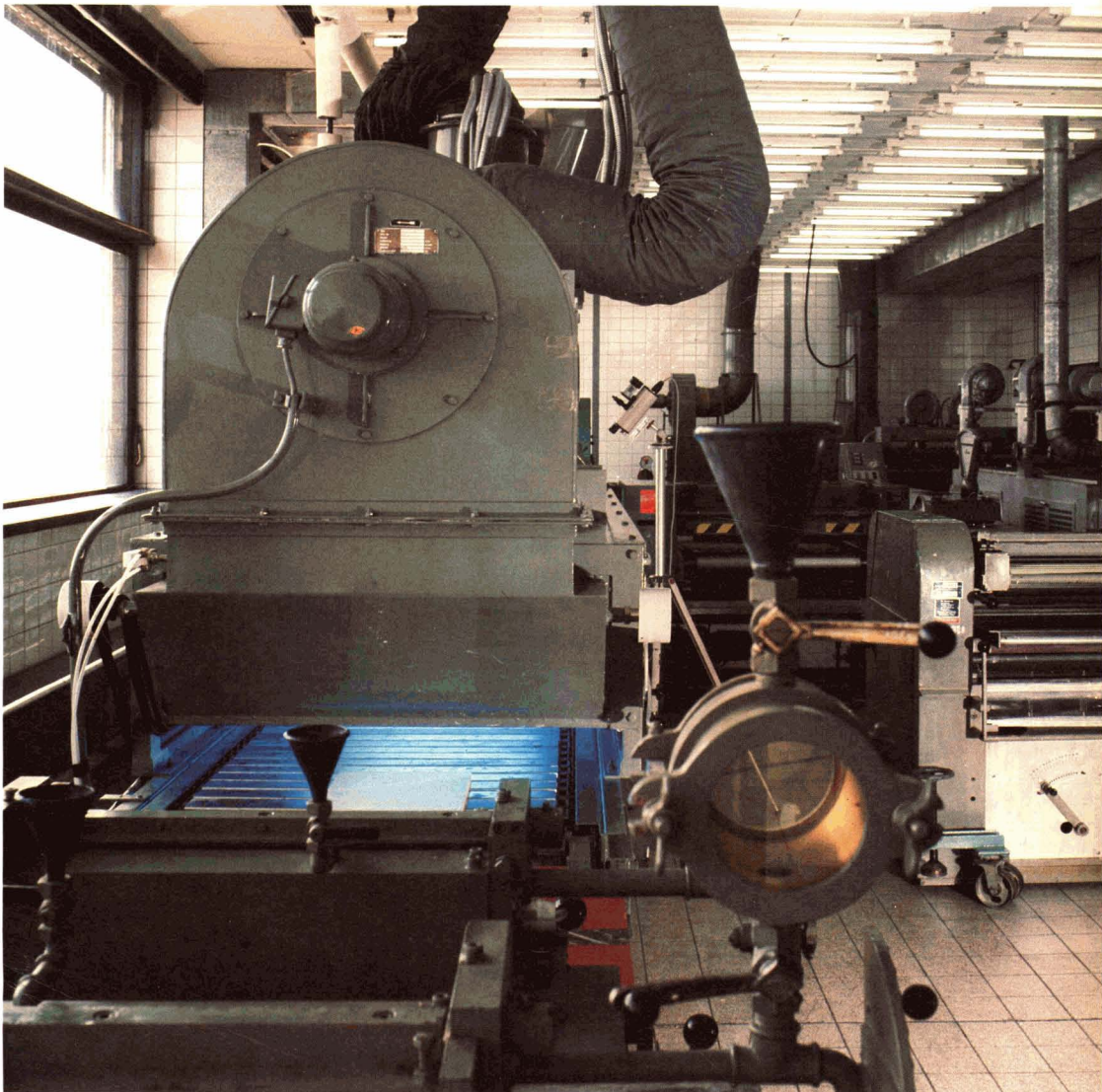


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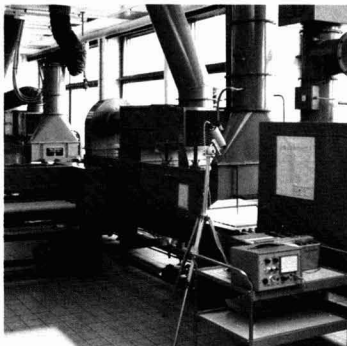
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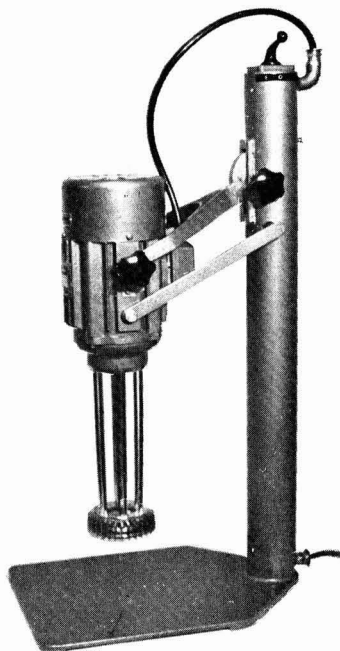
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for particles of radius a in a medium of viscosity η , where k is the Boltzman constant (at normal temperature $kT = 4.1 \times 10^{-14}$ ergs).

For each collision of two primary particles, one flocculate is produced, which is capable of interacting with further primary particle. Therefore, the decrease N in the total number of particles in the system is half that of Equation 1.

$$-(dN/dt) = 4\pi D R N^2 \dots\dots\dots (3)$$

From this, the time for the number of particles to be reduced to half the initial value ($t_{1/2} = (4\pi D R N_0)^{-1} \dots\dots\dots (4)$) is obtained by integration. N_0 is the initial concentration of particles.

The collision radius, R , can normally be taken to be twice the radius of the particle and so equal to $2a$. With this assumption, Equations 4 and 2 can be combined to give

$$t_{1/2} = 3/(4kT N_0) \dots\dots\dots (5)$$

Dispersions in low viscosity liquids such as water or solvents, with a particle concentration of about 10^7 cm^{-3} give $t_{1/2} \sim 10^4$ seconds (3 hours), whilst at a particle concentration of 10^{11} cm^{-3} , $t_{1/2} \sim 2$ seconds. A pvc dispersion of particle diameter 0.4 microns, containing 10^{11} particles per cm^3 has a polymer volume concentration of about 1 per cent, so that all dispersions of pvc particles in a suspension, at concentrations of technological interest, are inherently unstable, unless some form of energy barrier is provided to prevent each collision from being effective in producing a stable flocculate. The magnitude of the forces of attraction between particles in dispersions, and the mechanisms of stabilisation available will now be considered. The total potential energy of interaction between a pair of hydrophobic colloidal particles can be written as:

$$V = V_R + V_A \dots\dots\dots (6)$$

where V_R is the repulsive potential energy and V_A the attractive potential energy. V_R depends on the size and shape of the particles, the ionic strengths and the potential ψ at or near the interface, which determines the stability of the dispersion. For two spherical particles each of radius a , with their surfaces and centres H_0 and R apart respectively, V_R is given in an approximate form for small K_a values by:

$$V_R = \frac{a^2 \psi^2}{H_0 + 2a} e^{-KH_0} \dots\dots\dots (7)$$

and for large K_a values, V_R is as follows:

$$V_R = \frac{1}{2} e a \psi^2 \ln(1 + e^{-KH_0}) \dots\dots\dots (8)$$

where e is the dielectric constant at the medium in which the particles are suspended, and K^{-1} the thickness of the double layer.

For spherical particles of equal size, the potential energy of attraction can be considered by means of the approximate equation

$$V_A = -\frac{A a}{12} \cdot \frac{1}{\dots\dots\dots} (9)$$

where A is the London-van der Waals, or Hamaker constant of attraction.

Combination of Equations (7) and (9) or (8) and (9) enables potential energy curves to be computed. Two cases are shown schematically in Fig. 5, namely those for interaction between two spherical particles at $K_a \ll 1$ (Fig. 5a) and $K_a \gg 1$ (Fig. 5b). Fig 5 shows that an energy barrier exists, and as the particles approach one another there is a net force of repulsion, increasing to a maximum and then falling rapidly, as the particles come closer into the primary minimum of strong attractive forces. Whether or not such a maximum exists, and the magnitude if it does exist, depends upon the dimensions of the particles and their surface potentials. Whether or not a dispersion will flocculate, will depend on whether there are a sufficient number of particles with energies high enough to overcome the energy barrier. It is generally considered that an energy barrier of $15kT$ is sufficient to produce a dispersion that is highly stable. Fig. 5 also shows that attraction due to van der Waals' forces decreases, however, only very slowly with increasing separation between the particles. Most types of repulsive forces, and more specifically electrostatic repulsions, decrease much more rapidly with increasing particle separation and their effect has become negligible at distances where van der Waals attraction is still noticeable. With large particles, the net potential energy curve may take the form shown in Fig. 5b with the development of a secondary minimum 'S' at appreciable distances of separation. It is suggested that such minima, a few kT deep, would give rise to a more open type of flocculation, with considerable distance between the particles as shown in Fig. 2a. This form of flocculation would be quite distinct from that occurring at the primary minimum, since there is no rapid decrease in potential energy as particles approach each other more closely.

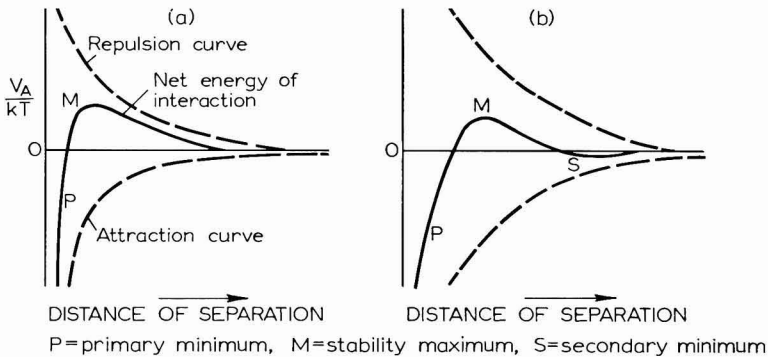


Fig. 5. Schematic diagram of potential energy of interaction against distance for two spherical particles, (a) $K_a \ll 1$ and (b) $K_a \gg 1$

Theoretically, if pvc powder particles in plasticiser can acquire a sufficiently high charge, it is possible to obtain a stable paste.

Zeta potential

The determination of the zeta potential of pvc particles in dap was carried out in an attempt to establish any possible correlation with stability.

The concept of Zeta potential or surface potential is schematically illustrated in Fig. 6 for a solid in contact with

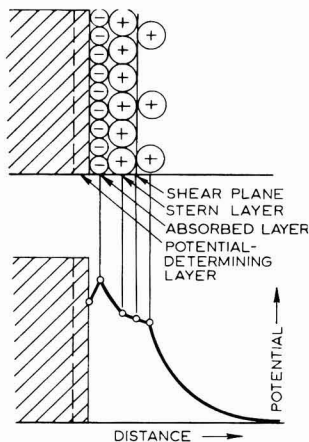


Fig. 6. Concept of surface potential

an electrolyte solution. According to the double-layer hypothesis, the solid will acquire a certain charge density localised in the plane of its surface; thus, a difference in electrical potential between the surface and the bulk solution will be produced. Because the system as a whole is electrically neutral, the surface charge must be exactly balanced by an opposite excess charge in the liquid phase. Because of columbic attraction, the counter ions will tend to concentrate in the vicinity of the solid surface, whilst ions of similar charge are repelled. The model of the double layer, therefore, involves an immobile surface-charge layer and a diffuse layer of counter-ions distributed in the adjacent solution according to some equilibrium function; this results in a more or less gradual decrease in potential with increasing distance from the surface.

Measurements of the zeta potentials of some of the pvc powder particles in di-alphanol phthalate (dap) were made, using an electrophoretic method. The method uses a simple principle that small charged particles suspended in a system, will move under the influence of an applied potential gradient; from the measurement of the deposition rate, the zeta potential is calculated from the theory described in appendix 1. The results obtained with some of the pastes are given in Table 3, which also contains data on electrical conductivity, dielectric constant and volume resistivity. In such non-aqueous systems, the surface potential may be equated to the zeta potential.

It is of interest that the values of electrical conductivity of pastes are much higher than that of either dap or pvc polymers. This immediately suggests that some conducting material, such as surfactants, is extracted from the surface

Table 3
Electrical and electrophoresis measurements

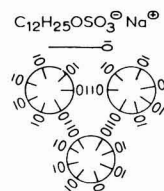
Paste no	Electrical conductivity	Dielectric constant	Volume resistivity (Ohms/cm)	Zeta Potential (mV)
1	0.0195	4.5893	8.963×10^7	2.98
2	0.0646	4.5387	2.667×10^7	2.05
3	0.0286	4.44160	5.900×10^7	4.30
8	0.0522	4.6070	3.250×10^7	3.50
9	0.0696	4.9500	2.400×10^7	2.98
Plasticiser (dap)	0.00045	5.1360	3.840×10^7	—

of the polymer particles into the continuous phase (dap). No such large variations in the values of dielectric constant and volume resistivity of paste and dap were found. Generally, the magnitude of the zeta potential (energy barrier) shown by pvc particles in dap was insufficient to provide a satisfactory charge stabilisation mechanism for the dispersions.

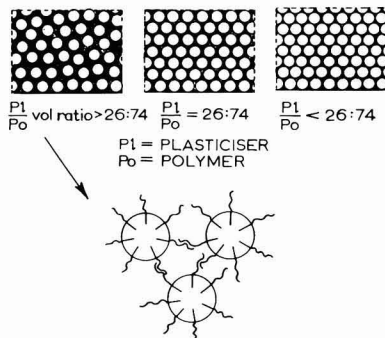
Steric hindrance

Ref. 8, 9, 8

Where the energy barrier is low or non-existent, the particles may still be prevented from approaching one another closely into the primary minimum distance by the presence of adsorbed layers on the surface of the polymer particles. This is illustrated in Fig. 7b. Such adsorbed layers would normally have a composition similar to the continuous phase, eg plasticiser or some polymer molecules. The physical interaction of these adsorbed molecules, as the particles approached one another closely, would act as a barrier. By this means, even when the net interaction curves are of the type shown in Fig. 5a, an adsorbed layer could provide steric hindrance to prevent the interaction potential falling into the primary minimum. Even when the net interaction energy is negative,



(a) Electrical stabilisation mechanism



(b) Adsorbed layer (steric hindrance) mechanism

Fig. 7. Stabilisation mechanism

eg a few kT, there would be a sufficient number of molecules or particles in the paste with energies of say 5-10kT to redisperse the flocculates as they are formed. The exact extent to which a negative interaction potential can be tolerated and yet allow stability is open to question, but Vold⁸ has suggested that values below 10kT may be tolerable.

This simple approach of stabilisation by a bulk layer of adsorbed molecules has been extended by Mackor and Van der Waals⁹ to stabilisation by adsorption of rod-shaped molecules, eg aliphatic chains, the interaction of which provides an energy barrier. The free energy of interaction of the adsorbed molecules F_R is given by

$$F_R = XN_S kT$$

where X is a constant (0.2) and N_S is the number of adsorbed molecules per square centimetre. With a mechanism such as the one illustrated in Fig. 7b, an appreciable repulsion potential can be obtained.

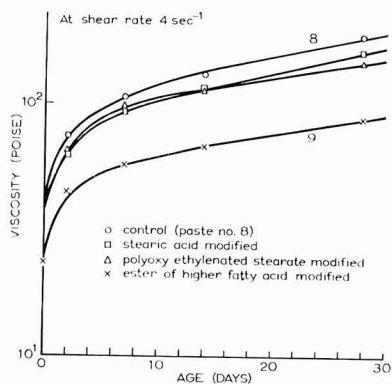
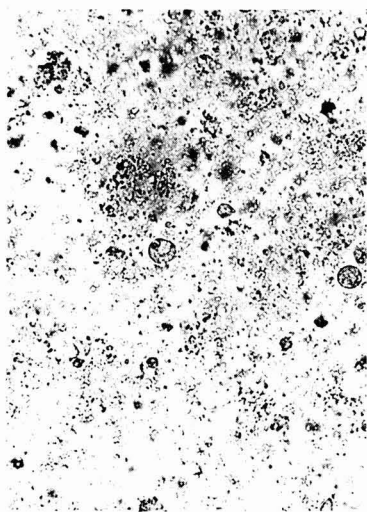
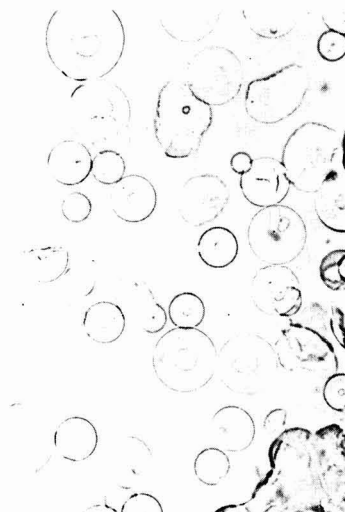


Fig. 8. Ageing behaviour of pastes



(a)



(b)

Fig. 9. Optical micrographs of pvc pastes; magnification $\times 324$

Cetyl alcohol and Sytol AF, an ester of a higher fatty acid, are readily adsorbed on to pvc latex particles. The effect of a number of additives on the ageing of pvc pastes is illustrated in Fig. 8, which shows that pastes made in di-phenolphthalate (dap) of Sytol AF modified latex age much less severely than the control (sample 8). Fig. 8 also shows that the initial viscosity, at low shear rate, of control paste 8 is nearly twice the viscosity of modified paste. Fig. 9 shows the optical micrographs of two pastes, one the control (Fig. 9b) and the other cetyl alcohol modified (Fig. 9a). Micrographs show that the secondary particles of modified pvc polymer, which is good both in initial viscosity and ageing, break down to smaller primary particles. The secondary particles (Fig. 9b) which do not break down may partly account for the high initial viscosity and rapid ageing of control paste. No measurement of adsorption isotherms was made, but the adsorbed layer can affect the stability of pastes in three main ways:

1. by increasing the charge on the particles (if a long chain ionic compound is used)
2. by altering the value of the Hamaker constant
3. by sterically hindering the approach of the particles.

Stabilisation by method 1 could occur by the adsorption of long chain ionic compounds. Naturally, increasing the dielectric constant will enhance ionisation of the compounds and usually increase the charge on the particles. Increase of charge will always increase stability, but in addition the stability may be enhanced by mechanisms 2 and 3 as well. The effect of adsorbed layers on the energy of attraction between two particles has been considered by Vold⁸. The effect of the adsorbed layer (20Å thick) is to decrease the potential energy of attraction to approximately one tenth of that for an uncoated particle. Thus, with all particles, an increase in stability should result. Reduction of the Hamaker constant owing to the presence of the adsorbed layer will clearly reinforce the effect of steric hindrance. It is not clear which of the two effects, steric hindrance or the reduction of the Hamaker constant, is the major one.

Effective solids volume concentration

The forces between dispersed pvc particles determine the stability of pvc pastes. Both attractive and repulsive forces occur; their net result must be repulsion if the particles become close packed; otherwise the interaction between the dispersed particles in a concentrated paste would result in partial aggregation. The rheological behaviour of a pvc paste depends to a large extent on the degree of aggregation. In particular, the notable increase in viscosity of a paste in which the particles have been partially agglomerated is attributed to the relative immobilization of a fraction of the volume of the plasticiser trapped in between the particles of the aggregate. A similar situation arises if the particles are porous with cavities at their surfaces. The apparent loss of plasticiser in agglomerates or "flocks" increases the apparent solid volume fraction, as would also be the case if the pvc particle concentration or pvc solids were raised. The effect of agglomeration on the effective total volume of the dispersed pvc particles, including trapped plasticiser, therefore, furnishes part of an explanation for the increase in viscosity of flocculated paste.

The effect of concentration on the flow behaviour of a pvc paste is illustrated in Fig. 10, which shows sharp changes both in initial viscosity and in ageing. This difference is particularly marked at 61 per cent w/w concentration, where the increase in paste viscosity over 14 days is more than three times that recorded for a paste having 60 per cent w/w solids. This is partly because of the increased available surface area, and the viscosity would be expected to increase more rapidly. In addition, the structural contribution to viscosity depends upon the square of the number of particles per cubic centimetre. This structural contribution rises rapidly with either decreasing particle size at a given paste solids concentration or increasing paste concentration at a given particle size.

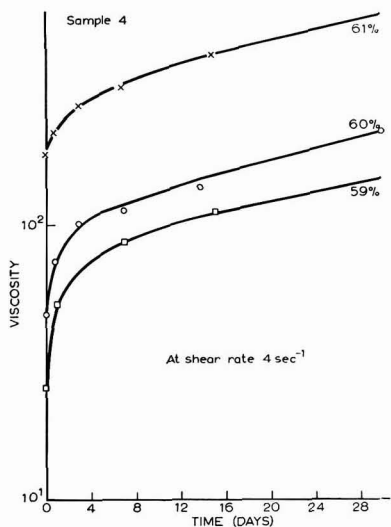


Fig. 10. Effect of polymer concentration on viscosity

A specially designed dilatometer was used to study the diffusion of plasticiser molecules in pvc particles and the subsequent change in effective solid volume fraction. The dilatometer was filled with recently prepared air-free paste, and kept in a water bath maintained at 23.5°C. The level of

the paste column in a capillary was recorded over a period of two weeks. The level could rise or fall, depending on whether or not the particle swelling outweighed the shrinkage caused by plasticiser diffusing into the particles. In general, pastes which shrink age more rapidly. The ageing of pvc paste is attributed to a combination of effects:

- loss of plasticiser into the particles through diffusion,
- swelling of pvc particles and the increase in apparent solid volume,
- relative immobilization of plasticiser in between the particles of agglomerates or flocks, and
- adsorption of plasticiser on the particles surface.

Factors (a), (b) and (c) will increase both the initial viscosity and the rate of ageing because they would increase the effective solids volume fraction and subsequently the particle/particle interaction. Factor (d), the adsorption of polymer with long chain fatty acid, on the particle surfaces will reduce the particle interaction, agglomeration and possibly may affect factors (a) and (b); thus improving the ageing behaviour of pvc pastes.

Conclusions

No evidence of an electrical stabilization mechanism was found for pvc particles in a dap at 60 per cent concentration.

It is concluded that stabilization occurs by an adsorbed layer mechanism.

By modifying the latex with esters of long chain fatty acid, it is possible to improve both the initial viscosity and ageing.

A concept of particle/particle interaction and the development of a new dynamic network model explain the flow behaviour of pvc pastes.

Microscopic examination of dispersions showed that the pastes that age rapidly contain small particles and these particles may agglomerate.

The ageing process apparently consists of two parts:

- the diffusion of plasticiser, which increases the effective solids volume fraction and subsequently increases the hydrodynamic viscosity.
- the expected large increase in the particle interaction as the solids volume fraction increases.

Acknowledgment

The author wishes to thank many of his colleagues for their assistance, and in particular the late Dr V. T. Crowl for valuable discussions.

[Received 3 October 1973]

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Appendix I

Zeta potential

The particle velocity, V , is calculated from the deposition rate as follows:

$$V = D_p/AC \text{ cm/sec} \dots\dots\dots(1)$$

where D_p is the deposition rate of polymer (g/sec)
 A is the area of electrode (32cm²)
 C is polymer concentration (g/ml) in paste

D_p , the true deposition rate of polymer, is calculated from the observed deposition rate, D_o , as follows:

$$D_p = D_o \cdot F \cdot e_f/(e_f - e_p) \dots\dots\dots(2)$$

where F is the weight fraction of polymer in the electro-phoretic deposit

e_f is the density of deposit (pvc powder)

e_p is the density of paste

According to Von Smoluchowski¹⁰, the theoretical relationship between the mobility of the particle and Zeta potential is as follows:

$$V/X = (\epsilon \psi)/(6\pi \eta) \dots\dots\dots(3)$$

where V is the electrophoretic velocity

X is the field strength

ϵ is the dielectric constant

η is the viscosity

ψ represents the Zeta potential

and therefore

$$\text{Zeta potential} = (6\pi \eta D_o e_f)/(\epsilon AC (e_f - e_p)) \dots\dots\dots(4)$$

Water absorption by polymeric films

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Summary

Water absorption by polymeric films depends on many factors, the more important ones being the monomeric constitution, the surfactant system used for emulsion polymerisation, and the use of various additives. In this work, an attempt has been made to establish also the practical importance of particle size of the

emulsion, the thickness of the films and the influence of the time allowed for the film to "mature" on water absorption by styrene-butyl acrylate films. It has been found that the quality of the polymeric films critically depends on the latter three parameters.

Keywords

Raw materials: binders (resins, etc)
emulsion resin

Processes and methods primarily associated with manufacture or synthesis
absorption

Properties, characteristics, and conditions primarily associated with:

raw materials for coatings and allied products
particle size

dried or cured films
film thickness

L'absorption d'eau par les feuil polymères

Résumé

La prise d'eau par les feuil polymères se dépend de plusieurs facteurs, dont les plus importants sont la constitution du monomère, la nature de l'agent tensio-actif utilisé au cours du procédé de polymérisation en émulsion, et les divers adjuvants éventuels. Dans cette étude on a tenté d'établir à l'égard des feuil de styrène-acrylate de butyle, l'importance au point de vue pratique

de la grandeur particulière de l'émulsion, l'épaisseur du feuil, et également l'influence sur l'absorption d'eau qu'exerce la longueur de l'intervalle qui se passe entre la formation du feuil et son exposition à l'eau. On a trouvé que la nature des feuil polymères se dépend dans une manière critique de ces trois derniers paramètres.

Wasserabsorption durch Filme von Polymeren

Zusammenfassung

Die Wasseraufnahme von Filmen polymerer Substanzen ist von vielen Faktoren abhängig, von denen am allerwichtigsten die Zusammensetzung der monomeren Bestandteile, das für die Emulsionspolymerisation benutzte System oberflächenaktiver Mittel und die Verwendung verschiedener Hilfsmittel sind. In dieser Arbeit wurde versucht, auch die praktische Bedeutung der

Teilchengröße der Emulsion, der Filmdicke sowie der Zeitdauer der Filmbildung auf die Wasserabsorption von Styrol-Butylacrylatfilmen festzustellen. Dabei wurde gefunden, dass die Qualität polymerer Filme in kritischer Abhängigkeit von den letzterwähnten drei Parametern steht.

Introduction

Ref. 1-7

Ever since synthetic emulsions were first introduced into the manufacture of coating materials, many attempts have been made to establish the factors which influence the properties and quality of the polymeric films. Much effort has been spent in studying the correlations between the chemical and physical properties of the polymeric films and their monomeric composition, which also determines the value of their glass transition temperatures. The influences of the unsaturated carboxylic acids, the surfactant systems used for polymerisation, the use of protective colloids, and so forth, have also been studied extensively.¹⁻⁴

The methods of preparation of the films and the film forming conditions also have important influences on the quality of the polymeric films.⁵⁻⁷

The various influences on the polymeric films can be complementary; therefore, it is sometimes very difficult to estimate the importance of any one particular parameter on the properties of such films, and the conclusions can be misleading.

The absorption of water by a polymeric film, and its mechanical and optical properties, are very important in determining the quality of a polymeric emulsion.

The influences of the film thickness, film forming time and particle size of the emulsion on the water absorption by a polymeric film are known qualitatively, and the work reported here was an attempt to establish the quantitative importance of these parameters. Attempts were also made to determine within which limits the above-mentioned parameters could be varied without altering the absorption of water by the polymeric film.

Experimental

Preparation of the emulsion

All the experimental work was carried out on emulsions of styrene/butyl acrylate/acrylic acid in the weight proportions 49 : 49 : 2. The emulsions were prepared by the delayed addition technique, using potassium persulfate as the initiator. A combination of anionic and nonionic surfactants was used. In order to obtain emulsions of different particle

size, the same quantity of nonionic surfactant was used in all cases, and only the quantity of the anionic surfactant was changed. All the emulsions were neutralised with NH_4OH to pH 9. The stoichiometric amount of NH_4OH required to neutralise the acrylic acid was never exceeded.

Film formation

Identical quantities of emulsions were poured on framed glass plates $21\text{cm} \times 30\text{cm}$. After 24 hours, the films were removed from the glass plates and dried at 25°C in a free-hanging position. Alternatively, the films were prepared on an Erichsen instrument, but because of the differing viscosities of the emulsions, the thickness of the films obtained in this manner varied too greatly.

Samples $2\text{cm} \times 7\text{cm}$ were cut from the films, their thickness measured at three different places and the only samples used were those for which the three measurements of thickness did not differ appreciably. The mean values of thickness are stated in the relevant Figs.

Before immersion in water to measure the water absorption, the films were allowed to mature for periods of from 3 to 31 days. All the determinations of water absorption were carried out on unpigmented films.

Particle size

The particle sizes for the emulsions were measured using an electron microscope with a final magnification of $\times 10\,000$.

Results and discussion

The water absorption of polymeric films was determined by differential weighing of the samples after from 5 to 120 hours' immersion in demineralised water at room temperature. It can be seen from the Figs that even after 120 hours' immersion, the absorption of water is still not always complete, but the experiments were not extended to longer times because longer times of immersion would exceed those likely to occur under practical conditions.

In no case was the weight loss caused by the elution of water-soluble materials from the film greater than 1 per cent.

Fig. 1 shows the percentage of water absorbed by films $340\mu\text{m}$ thick versus immersion time. The particle size of the

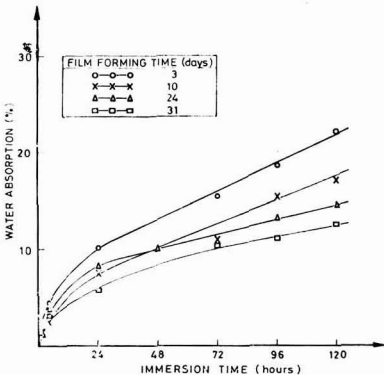


Fig. 1. Absorption of water versus immersion time. Particle size of the emulsion $0.25\mu\text{m}$

emulsion was $0.25\mu\text{m}$ and the curves in the Fig. correspond to films being maintained for 3, 10, 24 and 31 days before immersion. The range of immersion times is from 5 hours to 120 hours. A definite decrease in water absorption with increased time of maturing can be observed.

Fig. 2 shows the percentage of water absorbed by films $320\mu\text{m}$ and $350\mu\text{m}$ thick versus immersion time. The particle size of the emulsion was $0.55\mu\text{m}$ and the films were matured for 3 and 31 days. The range of immersion times is from 5 hours to 120 hours. It can be observed that a difference in thickness of only 8.5 per cent has a marked influence on water absorption of otherwise identical films. The influence of thickness is greater for films maturing for shorter times.

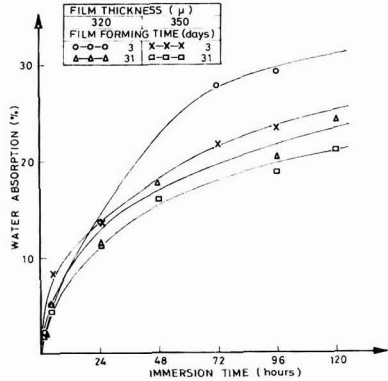


Fig. 2. Absorption of water versus immersion time. Particle size of the emulsion $0.55\mu\text{m}$

Fig. 3 shows the percentage of water absorbed by films with particle sizes $0.9\mu\text{m}$ and $0.25\mu\text{m}$ versus the immersion time. The mean thickness of all films was $327\mu\text{m}$ and the deviation from the average was less than 5 per cent. The maturing times of the films were 3 and 31 days. The range of immersion times was the same as above. The absorption of water by films with particle size $0.25\mu\text{m}$ is smaller than that by films with emulsions of greater particle size.

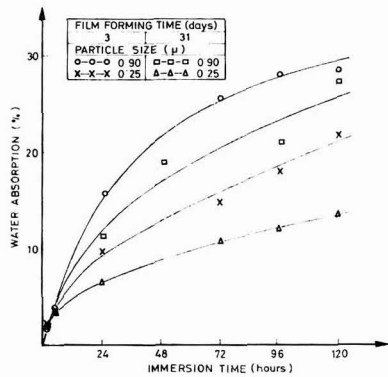


Fig. 3. Absorption of water versus immersion time. Mean film thickness $327\mu\text{m}$

Fig. 4 shows the percentage of water absorbed versus particle size. The range of particle size is from $0.2\mu\text{m}$ to $0.9\mu\text{m}$. The immersion times were 4 hours and 120 hours,

and the film maturing times 3 and 31 days. The mean thickness of films matured for 3 days was 318 μm and of films matured for 31 days 340 μm . It can be observed that after four hours' immersion, the film maturing time had practically no influence on the water absorption, but that the absorption is greatly influenced by the particle size of the emulsion.

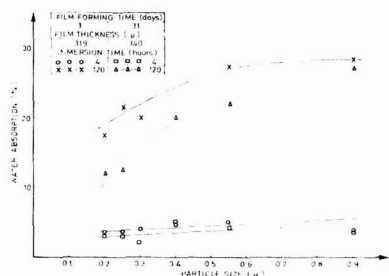


Fig. 4. Absorption of water versus particle size

Conclusions

The experiments have demonstrated that the water absorption of polymeric films (styrene/butyl acrylate/acrylic acid, weight proportions 49:49:2) is very much influenced by the following parameters: film thickness, film forming time and particle size of the emulsion.

The influences of these three parameters are so great that any data regarding the quality of polymeric films are completely meaningless if the values of the parameters are not specified. Significant changes of the quality of polymeric films are caused by only very small changes in the values of these parameters.

[Received 11 August 1973]

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Errata

Painting concrete

The following alterations to the paper "Painting concrete" by B. Lindberg (*JOCCA*, 1974, **57**, 100) should be noted:

Page 102, column 2, line 15: for "ratio cement to aggregate 1:35" read "ratio cement to aggregate 1:3.5"

Next month's issue

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

A parallel plate tackmeter for measuring the splitting resistance of printing inks by *V. Kelha, M. Manninen and P. Oittinen*

Gas chromatographic analysis of the carboxylic acid components of alkyd resins by *W. Mazurek and G. C. Smith*

Comparison of exposure sites at Innisfail, Australia, and at Canal Zone, Panama, for the testing of paint by *L. A. Hill, C. A. Grey and F. J. Upsher*

Page 103, column 2, line 1: for "surface strength from CO_2 to 4.1 N mm^{-2} for N_2 ," read "surface strength from 1.0 N mm^{-2} for CO_2 to 4.1 N mm^{-2} for N_2 ."

Page 106, column 2, line 21: for "0.1-0.2mm" read "0.1-2.0mm"

Page 107, column 2, item 4: delete second sentence

Page 109, column 2, last line: for "even slightly" read "or only slightly"

Page 110, column 2: the first equation should read " $G = k_d (\rho_1 - \rho_2)/d$ " and the second equation should read " $H = 2 \sigma \cos \theta / (r \rho g)$ ". In the paragraph between these two formulae, the value of k_d should be expressed in " $\text{g(h.m. mm Hg)}^{-1}$ " and the value of k_d/d in " $\text{g(h.m}^2 \cdot \text{mm Hg)}^{-1}$ ". In the first line of the following paragraph, for "date" read "data"

Page 111, paragraph 1: delete third sentence

Page 112, paragraph 4, line 6: for "the water has evaporated" read "the samples are removed and the water is evaporated"

Page 112, column 2, line 8: for "pelling" read "peeling"

Efflorescence on gypsum glass board

The following amendments should be made to the paper "Efflorescence on gypsum glass board" by G. A. King, M. J. Ridge and G. S. Walker (*JOCCA*, 1974, **57**, 127):

Page 127, last line of footnote: "which may be singly or doubly reinforced" should read "which may be singly or doubly reinforced!"

Page 127, second line of Introduction: for "sulfate!" read "sulfate?"

Page 129, column 1, paragraph 2, line 5: for "0.3 per cent" read "0.3 per cent?"

Page 130, column 2, paragraph 2, line 1: for "the highest contents" read "the highest content"

Addenda

The references 5 and 8 ("in press" when this paper was published) can now be read as follows:

5. King, G. A., Ridge, M. J., and Walker, G. S., *J. Appl. Chem. Biotechnol.* 1973, **23**, 635.

8. Maclagen, D. S., and Ridge, M. J., *Chem. Ind.*, 1973, **12**, 587.

Information Received

ICI scheme for joint research projects

Joint research projects operated by members of scientific faculties of universities and by ICI scientists are envisaged under a new scheme announced by ICI Limited after discussion with representatives from the academic world.

The scheme will replace the existing ICI Fellowship Scheme which will be phased out during 1976 and 1977. The new approach was designed to meet the often expressed need to establish closer and firmer relationships between industry and universities. It is hoped that it will promote a greater dialogue between academic scientists and their opposite numbers in the industrial environment.

The scheme is designed to be flexible and although it caters for the support of a single researcher in a university working closely with an opposite number in ICI, it will favour small joint teams. These will be known as ICI Research Project Groups. It will not cater for short-term projects but rather for those which take from two to five years to complete and for a total effort large enough to make a significant impact in the chosen field. All the research findings will be published and this will, of course, be of benefit to the scientists in the universities and to industry generally.

Contract awarded for new Spanish methylamines project

Ertisa SA, Madrid, a Spanish company owned jointly by Union Explosivos Rio Tinto SA (55 per cent) and ICI (45 per cent) has announced that McKee CTIP Ingenieros SA, Madrid, has been awarded the contract for Ertisa's new methylamines and derivatives project at Huelva. It is for engineering and design and procurement and construction management. The process technology for both process units will be provided by ICI Limited.

The project consists of a 10,000 tons per year methylamines unit, a 7,000 tons methylamines derivatives plant and the associated utilities. The project is scheduled for completion by late 1975.

Contract manufacture by Permutit

The Permutit Company Ltd. has entered the field of contract manufacture of chemicals. Process facilities currently existing in a flame proof area of the plant are capable of handling: polymer manufacture in bead and emulsion form; fluidised bed and oven drying; sulfonation; intermediate manufacture and catalyst mixing; chloromethylation; amination; distillation and solvent recovery.

Other plant can be installed as required and the company invites inquiries for all types of chemical and chemical process manufacture. All inquiries should be addressed to:

"Contract Manufacture," The Permutit Company Limited, Permutit House, 632/652 London Road, Isleworth, Middlesex, England.

Butler's recycling operation overcomes shortage

Butler Chemicals Ltd., manufacturers of the well known "Keroset" range of close-cut high boiling aliphatic petroleum distillates and a founder member of the Chemical Recovery Association has announced that

it was pleased to be able to give service recently to a number of engineering companies in the West Country who had found it impossible to obtain trichlorethylene from their normal supplier.

A large proportion of the production of these companies is exported.

The Petroleum Refining Division of Butler Chemicals Ltd. was able to come to the rescue by supplying re-distilled trichlorethylene, and instead of certain closure, production was maintained.

Re-distillation of solvents is a very small part of the work carried out by the Petroleum Refining Division of Butler Chemicals, the main task being the recovery of used lubricating oil. The refinery when built ten years ago had a rated output of 10,000 tons a year, and steps are currently being taken to take advantage of advances in techniques perfected in their research and development laboratories.

Swedish Institute tests dry-lubrication surfaces

The Royal Institute of Technology in Stockholm recently concluded a series of tests aimed at providing comparative data on the properties of various non-stick surfaces used as dry lubricants. Materials tested included pure polytetrafluoroethylene (ptfe), ptfe filled with glass fibre, and different grades of Du Pont "Teflon" and "Teflon S" non-stick finishes, which are based on fluorocarbons. The surfaces were applied to properly prepared substrates either by spraying and sintering or by gluing. Steel and polyacetal were used as counter-surfaces to measure friction and wear.

The Swedish tests have resulted in a number of tables not hitherto available. The results show differences in coefficient of friction ranging from 0.11 to 0.34. Wear resistance varied by a factor of 19,000, the best values being obtained with a surface of "Teflon S" non-stick and self-lubricating finish.

Distributor for Gates Hose

Rubber House Limited, the industrial distribution sector of Allied Polymer Group, has been appointed a main distributor in the UK for the hose products of Gates Europe SA.

The company will be marketing the complete range of Gates hose through its depots in London, Birmingham, Manchester and Glasgow. Initially Rubber House will be carrying stocks of wire braided steam hose, divers hose and acid chemical transfer hose, but it is intended to broaden the range of ex-stock products as demand is developed.

New products

Columbian offers alternatives to yellow chrome

The Runnymede Division of Columbian International (GB) Limited has announced that it is now able to supply alternative pigment dispersions for yellow chrome. The company explained that whilst these alternatives were more expensive, they did enable supply problems to be solved. The planned development programme started two years ago to extend the use of chip dispersions had now shown results. The

technical specifications were different from and in some ways more stringent than those of the ink and lacquer industries and the company had installed new testing equipment to meet the specifications, to monitor production, and to carry out further research into pigment dispersions.

Flowline converter thinner aids low-bake users

The Transport Paints, Division of International Pinchin Johnson has announced the availability of a "Flowline low-bake converter thinner". This new material, a specially formulated mixture of solvents containing a blend of resin media, is designed to convert "Flowline half hour enamel", an air-drying cellulose-synthetic product, into a material capable of being low-baked. It gives the low-bake user access to the ready mixed range of Flowline colours and the Flowline colour mixing scheme, which covers more than 3,000 UK and imported car colours.

Gasguard

Analytical Equipment Co. Ltd. of Marden, Kent, has introduced a new toxic/explosive gas alarm system called "Gasguard." It will detect explosive and toxic gases well before their concentrations in the atmosphere reach dangerous levels. The complete unit should have a life in excess of ten years and once installed should need no further attention for at least 100,000 hours.

Literature

Infrared spectra of surface active agents

Sadtler Research Laboratories Inc. of Philadelphia has published 1,000 infrared reference spectra of surface active agents comprising three new volumes, which will supplement the 14 volumes of infrared grating spectra previously published in this collection.

An alphabetical index by trade name and a classification index by chemical type is furnished with the collection.

Register of consulting scientists and contract research organisations

Fulmer Research Institute has announced the availability of the second edition of its register of all consulting scientists and contract research organisations, which contains over 150 new entries and many new subject headings. Further information is available from Fulmer Research Institute Limited, Stoke Poges, Slough, Buckinghamshire.

Vorwald expanding reel shafts

J. & William Burt Limited, distributor in the United Kingdom for Vorwald KG of Germany, has announced the availability of five new data sheets describing the basic range of Vorwald expanding reel shafts now available in the United Kingdom.

The shafts are particularly suitable for use where paper, converting, textile or other web fed material is wound on to steel or cardboard cores. Shaft diameters currently available range from 50 to 300mm in a wide variety of types.

Hull

Etch and blast primers

The final technical meeting of the present session was held at the Dorchester Hotel, Hull, on Monday 4 March with Mr F. D. Robinson in the chair. A lecture with the above title was given by Mr H. F. Clay, of SCC Colours Limited, to an audience of 13 members.

Mr Clay briefly reviewed the systems available as wash and blast primers, and described recent work which has been carried out in his laboratories on single-pack zinc phosphate wash primers.

In the discussion period which followed the lecture, Mr P. J. Gay commented on the deleterious effect of alkali on the phenolic resin constituent of etch primers when these were exposed to a marine environment.

The vote of thanks to the lecturer was proposed by Mr E. Armstrong.

J.A.H.

Ladies Evening: Walls talk

A meeting attended by some 18 members and their guests was held at the Dorchester Hotel, Hull, on Monday 4 February. The Section chairman, Mr F. D. Robinson, introduced Mr G. Marks of ICI Limited Wallcoverings Group, who gave an illustrated lecture entitled "Walls talk".

Mr Marks described the work of his design department referring to the stages of origination, adaptation, cylinder engraving, striking off, selection, pricing and compilation of the pattern book. This work resulted in an annual range of some 100 patterns, each in three colourways. Typical designs were then shown with comments when appropriate on colour, pattern, fashion and taste.

Following the discussion period, Mr E. Armstrong thanked the lecturer for an entertaining and instructive evening.

J.A.H.

Irish

President's visit to Dublin

Wednesday 16 January was a special occasion for the Section when it welcomed the President of the Association, Mr Silver, to a meeting at which he gave a talk on the subject of "Profitability in the paint industry". A notice of the meeting had been circulated to other bodies likely to be interested, and a welcome was extended to a number of visitors who responded to this invitation.

Mr Silver made it clear at the outset that the views he was about to express were his own, and did not in any way represent those of the Association. His lecture, illustrated with many tables and diagrams, evidence of a carefully studied survey, indicated the poor showing of the UK (and Irish) paint industry relative to that of the majority of other European countries, notably West Germany. In growth, and output per employee, the UK came near to the bottom of the list, and although home production was now in third place in Europe, its total was almost static and the country had lost its place at the top—a position it held a few years ago. Mr Silver was sceptical about the impact of advertising on paint sales, and in dealing with this subject he showed in a detailed series of tables how paint advertising in the UK only seemed to succeed (and in some cases only) in transferring from one supplier to another some portion of the

total paint pool. The UK position of low relative growth compared most unfavourably with that of West Germany where paint sales had increased by 50 per cent in recent years. Even in France, whose paint production now exceeded that of the UK, and where the visitor was not normally impressed by the amount of paint used, the growth rate had been high. The UK problem was now one of finding some way in which the home market could be increased and although exporting was necessary and insufficient (not to say inefficient) at present, Mr Silver was not sanguine about raising sales in the EEC, except perhaps in specialised lines, such as DIY paints. Nevertheless, the lecturer was a strong supporter of the UK joining the Common Market.

The position was no better in the survey of output per employee. Here again the UK was near the bottom of the table of European countries, ranking with Greece, and other less industrialised areas, and with about half the individual output figure of that of West Germany. On the other hand, where plant in the UK was designed for efficient output, results far in excess of the German average were obtainable.

Turning to export, Mr Silver had two major points to stress; one, that the cost of exporting was, contrary to a view often expressed by manufacturers, less than that of conducting home sales, and two, that insufficient attention was paid by many UK firms to the needs and preferences of the market they wished to enter.

In conclusion, Mr Silver was hopeful that the present upheavals in material supply would lead to a complete reassessment of how to expand the paint market and what could be done, at a time when every maker was having, in a sense, to start again from scratch, to improve methods and plant. The country's present difficulties could provide the inspiration for the future.

The discussions which followed the meeting were both lively and prolonged, and many questions were put to Mr Silver, none of which diverted him from his main thesis that the facts and figures presented had indicated a disturbing state of affairs, but that it was well within the power of the UK paint industry to turn this position to its advantage.

Following the lecture, the Chairman, Dr F. W. Stoye, presented the President with a shillelagh as a memento of his visit to Ireland, with the comment that the instrument might prove useful in maintaining order at the many meetings over which he had to preside. A vote of thanks to Mr Silver, proposed by Mr R. C. Somerville, for his interesting and stimulating address was passed with enthusiasm.

P.M.

Manchester

Colour difference

At the meeting held on Friday 11 January at the Literary and Philosophical Society, George Street, Manchester, 35 members and guests attended to hear Mr K. McLaren, of Imperial Chemical Industries Ltd, give a lecture entitled "Colour difference: the measure of the future".

Mr McLaren began with a historical survey of colour measurement, pointing out that since 1931 it had been possible to measure any colour by determining three numbers known as "tristimulus values". These could be calculated from reflectance curves or determined directly using "tristimulus colorimeters".

In the 1960s computer match prediction became a practical proposition but the problem of quantifying the closeness of a match remained. Two developments since 1969 were providing a solution to this problem. The first concerned the

identification of the most reliable colour difference equation from the 20 different ones available. This procedure had to be extended to achieve the best level of agreement with the judgement of professional tinters in the paint industry. These extensions required firstly the establishment of different pass/fail values of the colour difference (ΔE) not only according to end-use but also according to the colour involved and secondly, the setting of different values according to the nature of the colour difference; for example, a difference of hue is usually less acceptable than an equally perceptible difference in lightness.

The second development concerned a revolution in the design of colorimeters and the combination of colorimeters with mini-computers so that ΔE values could be displayed 35 seconds after the standard has been placed on the instrument, and repeat measurements would agree to 0.1 Adams-Nickerson units, a difference below the threshold of perception. Such instruments cost under £5,000.

Mr McLaren then demonstrated such an instrument, and this was followed by a lively discussion period.

A. McW.

Student Group

Pigments for aqueous finishes

A meeting for students was held at the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1, on Wednesday 30 January 1974, when 34 members attended. A lecture was given by Mr J. T. Hurst, of Ciba-Geigy (UK) Ltd. Pigments Division, entitled "Pigments for aqueous finishes".

This paper considered and reviewed the various criteria which must be met when pigmenting water based systems. Comparisons were made of the various present-day applications and their differing requirements in terms of pigment type and physical form.

It was shown that minor constituents, such as surface active agents, played a crucial role and this aspect was discussed at some length.

After the interesting discussion period, a vote of thanks was proposed by Mr F. Rourke.

A. McW.

Additives

A meeting was held for the registered students at the Manchester Literary and Philosophical Society, 36 George Street, Manchester, on Wednesday 27 February, when a talk on additives was given by Mr W. K. H. Lakin, of Hardman and Holden Limited.

The speaker covered a very wide field of additives, including those for anti-settle, anti-sag, anti-flood and -float, as well as slip aids, dispersion aids, static controls, levelling aids, viscosity stabilisers, driers, and others. Here was a list which might appear daunting to any lecturer, but Mr Lakin ably chose the right balance between essential detail and broad coverage. He supported his talk with a variety of exhibits which did much to maintain and deepen the interest aroused in his audience.

The appreciation of those present was capably expressed in a vote of thanks given by Mr B. Falder.

A. McW.

Midlands

Who needs paint anyway?

The Annual Dinner Lecture was held at the Birmingham Chamber of Commerce and Industry on Friday 18 January 1974. The speaker was Mr L. H. Silver, President of the Association, who had chosen for his topic "Who needs paint anyway?".

It was made clear that the lecture represented the speaker's own personal opinions, and as such should be divorced from his official capacity.

The lecture presented a broad analysis of the UK paint industry's lack of initiative in failing to achieve a satisfactory growth rate. The approach to markets tended to be that of a non-growth industry, and the industry has predicted over a number of years the steady replacement of paint products because of the increasing use of plastics. Statistics presented by the speaker indicated that paint manufacturers in many other countries had achieved excellent growth rates, thus suggesting that the pessimism of many in the UK was largely unnecessary.

The present industrial situation was unparalleled and, unfortunately, had come after an 18-month period of reasonable growth and improving profits for the paint industry. The chemical suppliers were open to criticism for the long-standing policy of underpricing their products, with the resulting cut-back in expansion and consequent shortages. The oil shortage was considered by some authorities to be largely artificial, and again the oil companies should be criticised for their failure to develop fully the many available resources in non-Arab localities. In the context of the lecture, however, the paint industry was discussed in terms of a return to normality, in perhaps two years' time.

The speaker considered that the world could carry on quite adequately without paint, which, whilst not essential, was psychologically desirable. Profitability was achieved where conflict occurred between salesmen and technicians, provided that the necessary compromise was reached. In the UK paint industry, this conflict tended to be negative.

In the field of exports, the enormous growth potential was being utilised much more fully by this country's European competitors, whilst advertising tended to divide the existing market between advertisers rather than achieving expansion.

Management had become much more professional during the last ten years, and professionalism at the technical and sales levels was also evident and had to continue to grow. Productivity had improved, but was still comparatively low. Further improvements should enable the industry to deal with such problems as poor labour relations and low wages.

The speaker was quite sure that paint had a great future. There was sufficient expertise to overcome all the current problems, but the industry in general had to convince itself of this, and thus make full use of resources.

After an extended period of questions, which reflected the controversial nature of the lecture, the vote of thanks was given by Mr D. E. Hopper.

F.W.C.

Newcastle

Student Group

Heavy industrial and marine contract painting

The second meeting of the session was held on Friday 15 February 1974 at Camrex Limited, when 25 members and visitors were present to hear Mr J. P. M. Latham talk on "Heavy industrial and marine contract painting problems". This was a very well-illustrated talk in which Mr Latham gave the benefit of his experiences in various parts of the world including Holland and the Middle East. The problems associated with dehumidification, temperature control and surface preparation of ships and their cargo tanks under a wide range of conditions were particularly stressed. Damage to tank coatings after application by the fitting of heating coils and welding in adjacent tanks gave further evidence of the problems contractors faced, in some cases due to the quality of the labour available. After a break for refreshments, a question period was held, which indicated the interest of those present.

J.B.

Association Conference 1975

The performance of surface coatings—does the reality match the theory?

The next Association Conference will be held at Scarborough from 17 to 21 June 1975. OCCA's Technical Committee met in December 1973, when it was decided that the theme "The performance of surface coatings—does the reality match the theory?" should be adopted, although not necessarily expressed in just these words.

An invitation is now extended to any person, whether or not a member of the Association, who feels that a report of his work may be suitable to be one of the technical papers, to submit a synopsis (of approximately 250-500 words) to the Honorary Research and Development Officer of the Association (Mr A. R. H. Tawn, 34 Crest View Drive, Petts Wood, Kent BR5 1BY) not later than 3 June 1974.

Papers selected for presentation at the Conference will be required in final draft by 1 October 1974 as it is necessary to

prepare full pre-prints to be sent to all those who have registered at least one month before the Conference.

It will be recalled that it is the custom at the Association's Conference for the authors merely to outline their papers, highlighting points of interest, and for a general discussion of the paper to follow. The author will not be expected to deliver the paper in toto, since the object of sending out the pre-prints in advance is that delegates may read these thoroughly before the Conference.

The venue for the Conference will be the Grand Hotel, Scarborough, with overflow accommodated at the St Nicholas Hotel. Full details concerning the registration fees and a form of application will be sent to all Members of the Association before the end of this year; non-members wishing to receive these details, when available, should apply in writing to the Association's offices.

Association Dinner Dance 1974

The Association's Biennial Dinner and Dance will be held on Friday 31 May 1974 at the Savoy Hotel, London WC2R 0EU.

The Reception will take place at 7.00 pm for Dinner at 7.30 pm and, on this occasion, there will be two short speeches only—a Welcome by the President and a Reply on behalf of the guests—so that dancing, to the Jerome Orchestra, can commence as soon as possible and continue until 1.00 am.

The price of single tickets is £7.00 plus 70p VAT each and applications should be made by Members as soon as possible. Tables will be arranged to accommodate 12 or 10 persons each, although it may be possible to arrange a few tables for eight persons. Non-members wishing to receive forms should contact the Association's offices.

Report of Council Meeting

A meeting of the Council was held at 2.00 pm on 26 February 1974 at the Great Northern Hotel, London, when 34 members were present. The President (Mr L. H. Silver) took the Chair.

The following members were appointed to serve on the BSI Committees shown against their names:

Mr J. E. Mitchell *BLCP/18*
Mr N. H. Seymour *ELE/61/53/6*
Mr A. R. H. Tawn *CIC/4*

The Annual Report of the Council for 1973 for presentation to the Members at the Annual General Meeting was approved, as were the provisional accounts for the year.

The Agenda for the Annual General Meeting was discussed and finalised.

Reports were received on the arrangements for the Joint Symposium with the Paintmakers Association, which will be held on the same day as the Annual General Meeting—26 June* at University College, London—and on the Association's Dinner Dance to be held on 31 May at the Savoy Hotel, London WC2, the Conference at Scarborough in June 1975 and the provisional booking for the 1977 Conference at Eastbourne in 1977. Consideration was also given to the Council Reunion Dinner in October 1974.

It was reported that both the Publications and Finance Committees had recommended to Council that the reprint *Resins, Drying Oils, Varnishes and Paints* from the Society of Chemical Industry's Annual Reports on Applied Chemistry should no longer be issued to members and this was agreed.

Prior to the meeting, the members of Council had received a letter from the President and the Hon. Treasurer drawing

attention to the very grave financial difficulties facing the Association in that it had incurred losses in both 1972 and 1973 and it seemed certain that a further deficit would have to be faced in 1974. The primary causes of this were the escalation in publishing costs and the effect on advertisers and exhibitors of the shortage of raw materials, since many had drastically cut back their advertising and many regular participants in former exhibitions were not taking space in 1974. Council reflected that it could well be argued that the Association had performed a splendid service at low cost to exhibitors in bringing visitors from 50 countries to the Exhibition but that the time had come to take stock of the situation and to review the policy of free admission to non-members and the issuing of copies of the *Official Guide* without charge to non-members, both before the Exhibition and at the entrance to the Hall.

The prices charged for the Association publications, such as the *Journal*, *Introduction to Paint Technology* and the *Works Practice Manual*, the capitation fee to OCCA Australia and the Association's advertising rates were considered well below current rates and were adjusted accordingly. Other suggestions for improving the Association's income were also considered by Council.

It was appreciated by Council that a very heavy burden had been imposed upon the Director & Secretary as a result of moving the offices at the end of 1972 from London to the suburbs, in that no member of his staff was able to continue in the Association's employ. Faced with the difficulty of recruiting and training an entirely new staff, the Director & Secretary at present was working without either an Assistant Secretary or an Administrative Assistant and was dependent to a large degree upon part-time staff. Council accepted that this state of affairs could not, of course, continue indefinitely and were

concerned that Section Committees should do all in their power, particularly in respect of circulars which are duplicated and dispatched by the staff at Priory House, to ease the burden. The co-operation of members in notifying changes of addresses and sending in subscriptions promptly without the necessity of further reminders was requested. In this connection, Council resolved in accordance with Article 14 that any member whose subscription had not been received by the end of March would not be sent the April and subsequent issues of the *Journal* until payment had been received.

Council noted with pleasure the appointment of Dr S. H. Bell (President 1965-67) as an Officer of the Most Excellent Order of the British Empire in the New Year's Honour List and decided to commemorate this distinction by the presentation of a scroll to Dr Bell at the Exhibition Dinner.

Arrangements were made for the production of an additional Association tie with a maroon background and a single woven under-knot motif. These would be sold through the Sections at £1.85 (including VAT) and the blue background ties would continue to be issued as at present by Thresher and Glenny Ltd. It was expected that the ties would be available in about three months' time (May or June). Orders would only be accepted against prepayment.

Council asked the Director & Secretary to prepare a memorandum for its next meeting setting out the present procedure for the election of candidates to Ordinary and Associate Membership and as Registered Students and to suggest ways in which the procedure could be streamlined to avoid criticism of delays and unnecessary expenditure. Council received a report from the Professional Grade Committee and accepted its recommendation that the obligatory periods of service as Ordinary Members before application could be

*See notice on page 177

made for admission to the Professional Grade should be reduced as follows:

For Fellowship two years
For Associateship (both routes) two years
Licentiatehip to remain at one year.

The President reported that, at the Annual Convention of the Federation of Societies for Paint Technology at Chicago in November, he had presented the retiring Executive Vice President, Mr R. W. Matlack, with a tankard on behalf of the Association. Details of Section activities since the last meeting were received and Council noted with pleasure that the Cape Branch of the South African Section had

been re-started. It also gave its approval to changes in Section rules proposed by the South African Section Committee for presentation to that Section's Annual General Meeting.

The Hon. Editor reported that during the emergency conditions, which had naturally affected the printing industry, it had been found necessary to saddle stitch the "Journal" in order to save time. Even so, with the three-day working week, the "Journal" was being produced later in the month than usual though attempts were being made to rectify this.

Council received a report giving the thanks of the Society of Dyers and

Colourists' Terms and Definitions Committee to the Associations representatives (Mr A. E. Honiball and Dr J. Toole) for their work on the publication "Colour Terms and Definitions". Council was saddened to learn of the death of a former Council member Dr V. T. Crowl who, at the time of his death in October 1973, was Hon. Publications Secretary of the London Section. A wreath had been sent on behalf of the Association, which was represented at the funeral by Dr S. H. Bell and members of the London Section Committee.

The meeting closed at 4.15 p.m. and the President thanked members for their attendance.

THE SYMPOSIUM ORIGINALLY SCHEDULED FOR 26 JUNE HAS BEEN POSTPONED UNTIL 17 SEPTEMBER 1974. FULL DETAILS WILL BE CIRCULATED LATER

Register of Members

The following elections to Membership have been approved by Council. The Section to which new Members are attached is given in italics.

Ordinary Members

ADAMS, IAN GEORGE, BSc, 4 Tower Road, Darwen, Lancashire, BB3 2DV. (*Manchester*)
ALLEN, DAVID FRANK, 25 Queens Close, Boothstown, Manchester M28 4BQ. (*Manchester*)
ALLEN, JEFFREY DAVID, BSc, Kingsley & Keith (Chemicals) Ltd, Suffolk House, George Street, Croydon CR9 3QL. (*London*)
BERGER, MARTIN NORBERT, BSc, PhD, FRIC, 112 Park Lane, Whitefield, Manchester M25 7PT. (*Manchester*)
CANNON, DAVID EDWARD, 8 Geneva Gardens, Chadwell Heath, Romford RM6 6SP, Essex. (*London*)
CROUCHMAN, PHILIP JOHN, 15 West Road, Sawbridgeworth, Hertfordshire. (*London*)
DEAN, TUAN KAMAL, 21 Sagara Mawatha, Panadura, Sri Lanka. (*General Overseas*)
FREUND, KURT ALFREDO, PhD, DIC, PO Box 611, Ouito, Ecuador. (*General Overseas*)
FRY, ERIC SYDNEY JOHN, BSc, 11 Eslington Terrace, Jesmond, Newcastle-on-Tyne 2. (*Newcastle*)
GISBEY, SHIRLEY E., 8 Earn Avenue, Kessington, Bearsden, Glasgow. (*Scottish*)
GUSH, JOHN RICHARD, BSc, 14 School Road, Amanzimoti, Natal, South Africa. (*South African*)
HAMPSHIRE, GEORGE ROBERT THOMAS, 101 Horns Road, Barking-side, Ilford, Essex. (*Thames Valley*)
HAVARD, BERNADETTE, 39 Rue Carves, 92120 Montrouge, France. (*General Overseas*)
HEYES, STUART GRAHAM, BSc, 34 Somerset Road, Rishton, Nr. Blackburn, Lancashire. (*Manchester*)
HOLLAND, ROY LESLIE, 563 Lytham Road, Blackpool, Lancashire. (*Manchester*)
HORWITZ, DAVID, BSc, 99 Mountain Rise, Carrington Heights, Durban, South Africa. (*South African*)
HOUSE, WILLIAM FRANK, 14 Elm House, East Ferry Road, Poplar, London E14. (*London*)
JALLI, SUBHI MOHAMMED HASEN, International Paint Co, Stony-gate Lane, Felling, Gateshead NE10 0JY, Co. Durham. (*Newcastle*)
KHAN, MOHAMED BASHIR, BSc, PhD, ARIC, API, 25 Ashton Road, Darwen, Lancashire BB3 2DX. (*Manchester*)
LODGE, DAVID WILLIAM, 30 Holland Road, Chatham, Kent ME5 9TW. (*London*)
LOVE, DAVID JOHN, LRIC, 6 Pine Grove, Ashton-on-Mersey, Sale, Cheshire. (*Manchester*)

LOYE, BRIAN ANTHONY, 61 Coleridge Road, Maldon, Essex. (*London*)
MCQUEEN, IAN, BSc, 14 Hazel Dene, Bishopbriggs, Glasgow G64 1TZ. (*Scottish*)
NEWELL, BARRIE ARTHUR, LRIC, 19 Hordern Crescent, Brierley Hill, Staffordshire DY5 2NR. (*Midlands*)
PRINCE, JANET MARY, 30 Wellington Road, Grove Park, Sittingbourne, Kent. (*London*)
RAYNER, GRAHAM ARTHUR, BSc, 29 Belgrave Road, Ilford, Essex. (*London*)
SANSUM, ANDREW JAMES, BSc, MSc, ARIC, BIE (Anti-Corrosion) Ltd, 63a Stanley Grove, London SW8. (*London*)
THORNLEY, JOHN MICHAEL, Park Villa, 5 Victoria Road, Fulwood, Preston, Lancashire. (*Manchester*)
TOMLINSON, JOHN PARKER, PhD, GradRIC, 26 Ellerbeck Road, Darwen, Lancashire. (*Manchester*)
TONGE, CHRISTOPHER JAMES, BSc, PhD, 4a Rock Street, Yasingden, Rossendale, Lancashire. (*Manchester*)
TOOVEY, JOHN, BSc, 37 Meadowbank Avenue, Strathaven, Lanarkshire. (*Scottish*)
YOUNG, CRAIG, 13 Darwin Road, Westwood, East Kilbride. (*Scottish*)
WATSON, JOHN MILNER, PO Box 1212, Wellington, New Zealand. (*Wellington*)

Associate Members

BOARDMAN, DENNIS CLYDE, PO Box 175, 4930 Stutterheim, South Africa. (*South African*)
GILL, TRALOCHAN SINGH, PO Box 555, Blantyre, Malawi. (*General Overseas*)
LEWIS, CRAIG ROBERT, c/o Hoechst (NZ) Ltd, PO Box 38169, Petone, New Zealand. (*Wellington*)
SMITH, BARRY KEITH, 36 Chawson Pleck, Droitwich, Worcester-shire. (*Midlands*)
TITTERTON, RICHARD JOHN HERWALD, 12 John Street, Birchleigh Ext. 4, Kempton Park, Transvaal, South Africa. (*South African*)

Registered Students

BUCK, PHILIP ANTHONY, AIST, 9 Kestrel Close, Caldicot, Newport. (*Bristol*)
DUNBAR, JOHN MARTIN, 27 Brownlie Street, Glasgow G42 9BT. (*Scottish*)
MUNRO, DUNCAN BELL, 63 Arduthie Road, Drumoyne, Glasgow G41 1EH. (*Scottish*)
VALLANCE, DAVID ALEXANDER, 46 Lennox Drive, Faifley, Clydebank G81 5JX. (*Scottish*)
WALKER, MARTIN RICHARD, 31 Ancaster Avenue, Bricknell Avenue, Hull HU5 4QP. (*Hull*)

News of Members

Mr R. C. Somerville, an Ordinary Member attached to the Irish Section, and a founder member and past Chairman of that Section, has retired as Operations Director of Berger Paints Ireland Limited after 50 years and the Berger Group both in Ireland and the UK.



Mr R. C. Somerville

His successor is Mr D. Pountain, an Ordinary Member attached to the Manchester Section, who was formerly Chief Industrial Chemist with Crown Paints.



Mr D. Pountain

Erratum

It was incorrectly reported in the February issue that Miss M. Searle was attached to the Trent Valley Branch of the Midland Section. Miss Searle is, in fact, attached to the Thames Valley Section.

Newcastle Section

Ladies' night



Mr C. N. Finlay, Chairman of the Newcastle Section (left), and Mr L. H. Silver, President of the Association, with their ladies

The Section held its annual Ladies' Night at the Five Bridges Hotel, Gateshead, on Friday 22 February, and about 210 people attended. Those present included the immediate Past President, Mr A. W. Blenkinsop and his wife, and the Chairman with the ladies from the Hull and Manchester Sections and the Midlands Section, including the Trent Valley Branch. Mr J. K. Keminsky, President of the Newcastle and Gateshead branch of the British Decorators Association, and his wife also attended. Guests of Honour were the President, Mr L. H. Silver, and his lady.

The Ballroom Five provided music for dancing after dinner, and the evening was considered to be highly successful.

Auckland and Wellington Sections

Eleventh New Zealand Convention

The Association notes with interest that two papers presented at the Eleventh Annual Conference held in Wairakei last year were published in *Chemistry and Industry in New Zealand*. The President of the Federation of Labour, Mr T. E. Skinner, addressed delegates on "Industrial relations and the future" and a paper by Dr T. Yamaguchi and J. J. Magill reported on "Novel vinyl resins".

Professional Grade

Amendments to regulations for admission to the optional professional grade for Ordinary Members

At its meeting on 26 February 1974 the Council accepted the recommendation of the Professional Grade Committee in respect of the periods of Ordinary Membership to be completed before admission to the professional grade.

The amendments, which take place immediately, reduce the obligatory periods as Ordinary Members for candidates for Associateship and Fellowship to two years *in all cases*, i.e. amending the regulations C9 and D2 as last printed in the *Journal* (page 463, September 1973).

Forms of application for admission to the professional grade may be obtained from the Association's offices.

Admission

Thomas Nelson Mills, an Ordinary Member attached to the South African Section, has been admitted to the Professional Grade as an Associate in the Technology of Surface Coatings.



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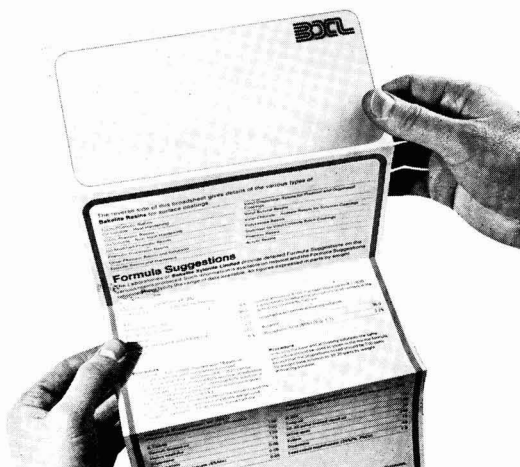
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OCCA 26 EXHIBITION REVIEW

A special feature reviewing the OCCA Exhibition at Olympia last month will appear in the June issue of this *Journal*. Anyone who was not able to visit the Exhibition this year and who wishes to receive a copy of the 112-page *Official Guide* for reference purposes should write immediately to the Association's Offices enclosing a remittance of £1.10 (including postage and VAT) per copy required.

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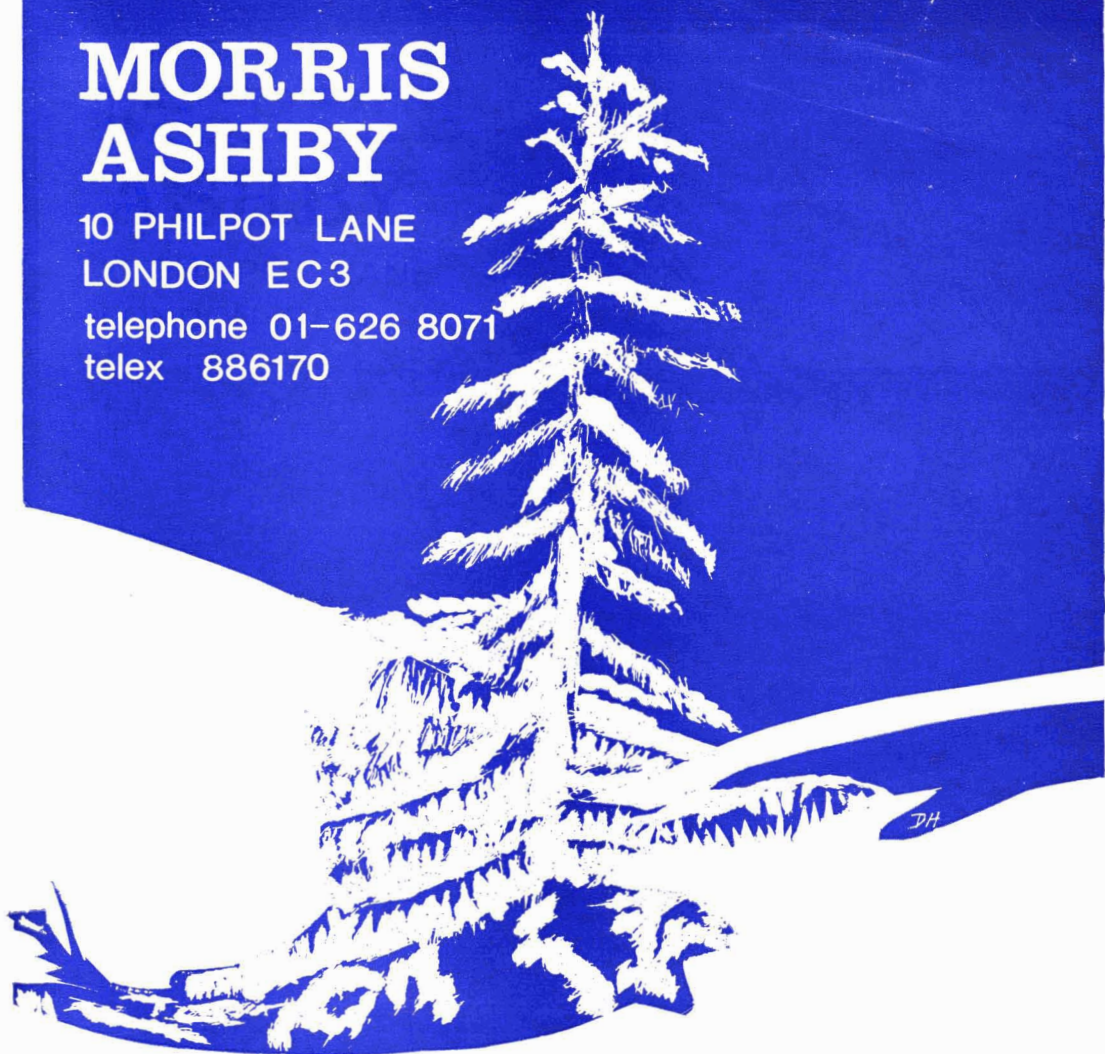
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