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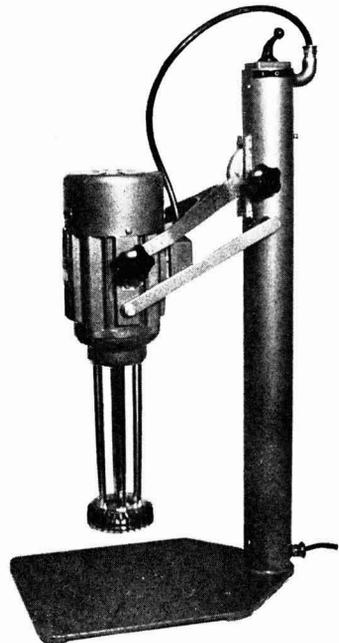
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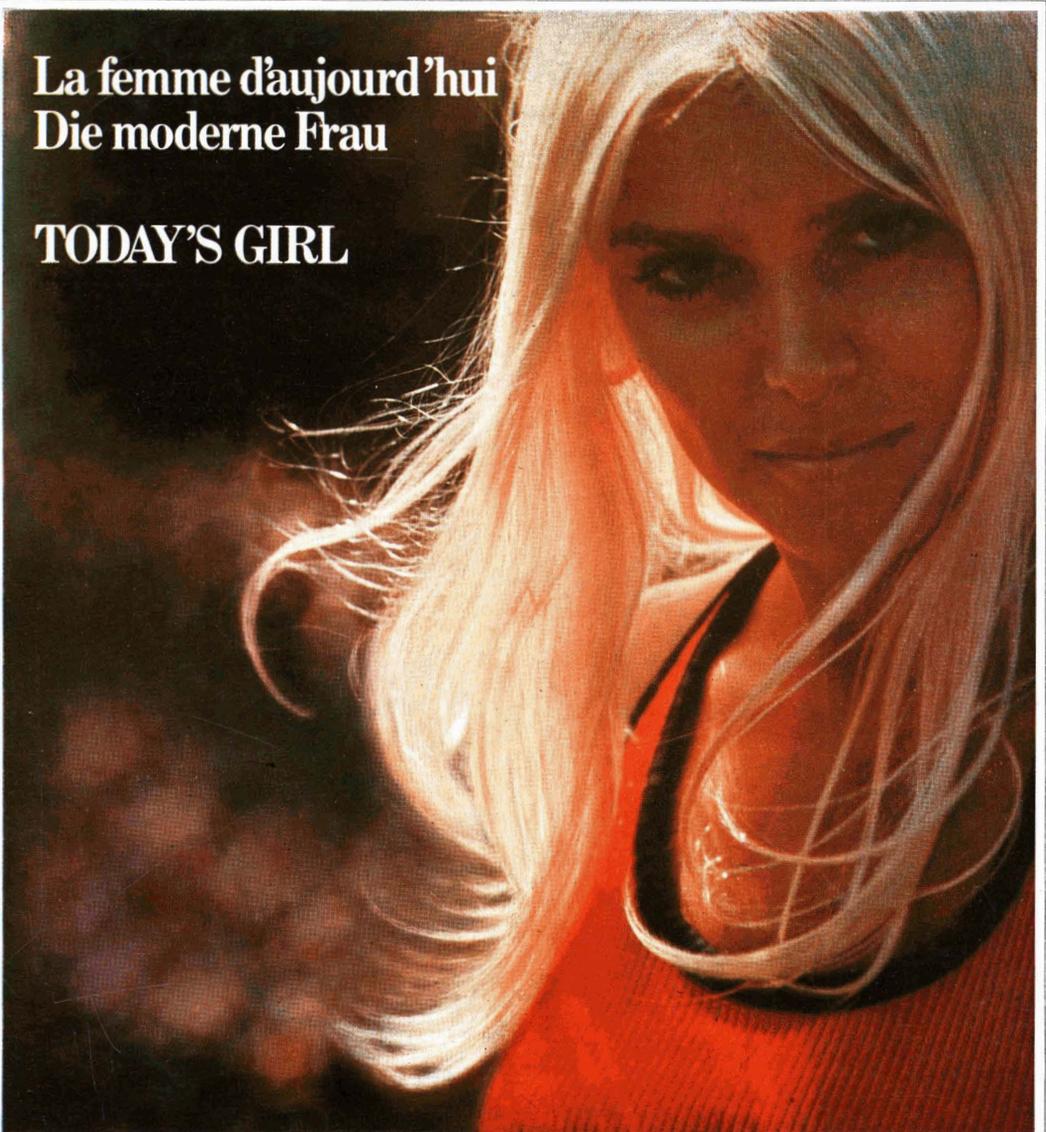
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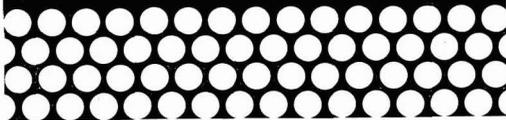
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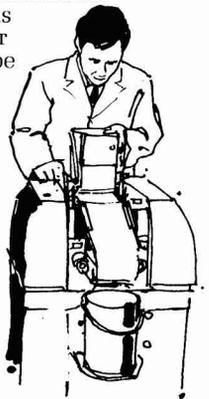
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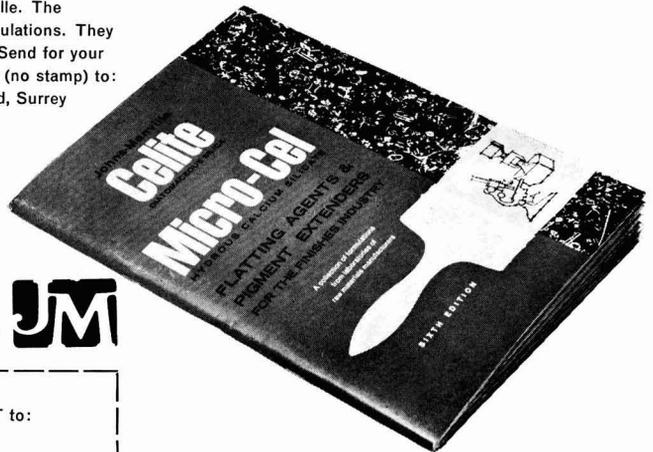
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# The prevention of gelation during the maleinisation of dehydrated castor oil

By **A. M. Naser** Chemistry Department, Faculty of Science, Al-Azhar University, Cairo

**N. I. Sadik** Chemical Department, Ministry of Industry, Cairo

**N. A. Ghanem** National Research Centre, Dokki, Cairo

## Summary

Gel formation during the maleinisation of dehydrated castor oil (DCO) is explained and attributed to the residual hydroxyl groups of the oil. The maleinisation of DCO is made possible without any sign of gelation by treating the oil prior to maleinisation with phosphorus pentachloride. Maleic adducts containing 15 per cent (by weight) or more maleic anhydride are found to be

water-soluble as ammonium salts and their preliminary evaluation as paint binders are performed. The differences between the theoretical acid values of the adducts and those found in practice are ascribed in part to carbon dioxide evolved during the reaction. The viscosity reduction on thinning with a water/ethanol mixture is studied.

## Keywords

*Raw materials for coatings: binders—oils*  
dehydrated castor oil

*Processes and methods primarily associated with manufacture or synthesis*

maleinisation  
gelation

## L'empêchement de gélification pendant la maléinisation de l'huile de ricin déshydratée

### Résumé

On donne une explication de la formation de gel pendant la maléinisation de l'huile de ricin déshydratée (D.C.O.) et l'on l'attribue aux groupements hydroxyle résiduelles de l'huile. On peut effectuer la maléinisation de D.C.O. sans risquer aucune trace de gel en traitant l'huile avec le pentachlorure de phosphore avant de commencer la maléinisation. On a trouvé que les sels d'ammoniaque des adducts maléique renfermant au moins 15 pour cent (par poids)

d'anhydride maléique sont soluble dans l'eau, et l'on a mis au point une évaluation préliminaire. On peut attribuer en partie les différences entre les valeurs théoriques de l'indice d'acide et celles que l'on obtient en pratique au dégagement de l'acide carbonique au cours de la réaction. On étudie le taux de diminution de la viscosité en se servant d'un mélange eau/éthanol en tant que diluant.

## Die Verhütung des Gelierens während der Maleinisierung von dehydratisiertem Rizinusöl

### Zusammenfassung

Es wird erklärt, dass das Gelieren von dehydratisiertem Rizinusöl (DCO) im Verlauf der Maleinisierung auf verbliebene Hydroxylgruppen des Öls zurückzuführen ist. Es ist möglich, DCO durch Vorbehandlung mit Phosphorpentachlorid ohne irgendwelche Anzeichen von Gelierung zu maleinisieren. Man kann feststellen, dass Maleinaddukte mit 15 oder mehr Gew. Prozent Maleinsäureanhydrid als Ammoniumsalze wasserlöslich sind, und sich als

solche zur Vorprüfung auf ihre Eignung als Anstrichbinder eignen. Die durch die Unterschiede in den zwischen den theoretischen und den in der Praxis gefundenen Säurezahlen bestehenden Schwierigkeiten werden zum Teil auf die während der Reaktion entwickelte Kohlensäure zurückgeführt. Die bei Verdünnung mit einem Wasser/Alkoholgemisch eintretende Verringerung der Viskosität wurde untersucht.

## Предотвращение желатинизации во время маленизирования касторового дегидратированного масла

### Резюме

Объясняется процесс образования геля во время маленизирования дегидратированного касторового масла и приписывается остаточным гидроксильным группам в масле. Маленизирование дегидратированного касторового масла возможно без наличия желатинизации путем обработки масла пентхлористым фосфором до маленизирования. Малениновые примеси, содержащие 15% (по весу, или более маленинового

ангидрида, растворимы в воде в виде солей аммония, и проводится их предварительная оценка как связующих для красок. Разница между теоретическими и практическими кислотными числами примесей приписывается частью двуокиси углерода выделяемой в течение реакции. Изучается понижение вязкости при разбавлении в смеси воды и этана.

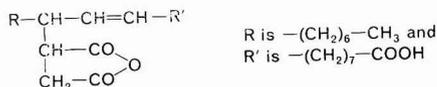
## Introduction

Ref: 1-8

The maleinisation of unsaturated vegetable oils is one of the common modifying processes applied in the field of organic surface coatings, especially with water-thinnable paints. This process permits the introduction of the hydrophilic carboxylic group in the middle of the fatty chain and consequently will aid solubility, and provides sites for subsequent curing of the film. The modified oil must be both

uncrosslinked and sufficiently hydrophilic to be soluble in bulk; it must also be capable of producing a coherent and water-insoluble film after drying. These adducts give water-soluble salts upon neutralisation with a suitable base. The introduction of these groups extends the range of application of the oils considerably; they become compatible with cellulose varnishes, useful in the preparation of resins and for improving the drying properties of non-conjugated oils, in addition to their common use as binders for water-thinnable paints.<sup>1</sup>

The reaction between maleic anhydride and unsaturated oils depends on the type of unsaturation present. In the case of non-conjugated oils, such as linseed oil, the reaction proceeds by the formation of a succinic anhydride derivative (addition by substitution) of the type given below in case of oleic acid:



This reaction is usually carried out<sup>2-1</sup> at temperatures above 200°C. On the other hand, the reaction with conjugated oils proceeds smoothly at 80°C by Diels-Alder addition and most of the mechanisms suggested involve the formation of a six-membered ring. Water-soluble linseed and tung oils, as examples of non-conjugated and conjugated oils, respectively, were prepared with as high as 15 per cent maleic anhydride by weight<sup>5</sup> without gelation.

There is, to the best of the authors' knowledge, no publication describing the use of dehydrated castor oil alone in water-thinnable paints, since its maleinisation leads to gel formation even with a maleic anhydride concentration as low as 5 per cent by weight; a situation which does not occur with other conjugated oils. The reasons for gel formation were discussed by Bolley<sup>6</sup> but no conclusions were reached; in one instance, the gel formation was attributed to polymerisation of maleic anhydride with DCO in a manner similar to the reaction of maleic anhydride with *cis*- and *trans*-piperylene.<sup>7, 8</sup>

This paper investigates the reasons why gel formation occurs during the reaction of DCO with maleic anhydride; gives new methods for preventing it, and facilitates the production of uncrosslinked water-soluble DCO. A preliminary evaluation of the product as a paint binder is made, and its viscosity in several water/ethanol mixtures is investigated.

## Experimental

Ref. 9-11, 2, 11-14

All chemicals used were, at least, of the grade "chemically pure."

### Preparation

DCO was obtained by the dehydration of Egyptian castor oil using Dowex-50 ion exchange resin as a catalyst.<sup>9</sup>

An acrylonitrile adduct of DCO was prepared by refluxing the oil (100g) with excess acrylonitrile (50ml) and hydroquinone (0.5g) for 96 hours. Unreacted acrylonitrile was removed by successive washing with distilled water, followed by vacuum conditions to remove the last traces.<sup>10</sup>

The acetylated derivative was obtained by refluxing DCO with acetic anhydride in the presence of *p*-toluene sulfonic acid. The ethereal solution of the product was purified by successive washing with distilled water, sodium carbonate (3 per cent) and again distilled water until neutral to methyl red. The material was dried over anhydrous sodium sulfate and the ether was removed in vacuo.<sup>11</sup>

The chlorinated derivative of DCO was prepared by refluxing the oil with phosphorus pentachloride in carbon tetra-

chloride. A mixture of DCO (50g), phosphorus pentachloride (10g) and carbon tetrachloride (100ml) was refluxed for 10 hours. Removal of excess phosphorus pentachloride left a dark red oil which was washed in ethereal solution with sodium carbonate (3 per cent) and water. The ethereal solution was dried over anhydrous sodium sulfate and the ether was removed by evaporation under vacuum.<sup>2, 11, 12</sup>

### Maleinisation

The maleinisation of DCO, acetylated and chlorinated DCO was carried out by charging the material to be maleinised in a dry test tube with the required amount of maleic anhydride and heating on a water-bath with occasional shaking; the time was recorded when gelation occurred. If no gelation occurred, the tube was transferred to a paraffin bath kept at 150°C and this was checked periodically for the appearance of gel.

For the purpose of evaluation, preparation of the maleic adduct of chlorinated DCO was carried out in a 250ml triple-necked flask equipped with a thermometer, inert gas inlet and stirrer. The temperature was adjusted to 100°C and the adduct was tested for unreacted anhydride by washing small portions with hot distilled water followed by titration against standard alkali. The reaction was considered complete when the unreacted anhydride was less than 5 per cent of the original amount.

Estimation of the carbon dioxide evolved during maleinisation was determined gravimetrically using an apparatus involving Nesbitt absorption tube.<sup>13</sup>

### Water solubility

The water solubility of the adducts was determined by dissolving them in dilute ammonium hydroxide to give a solution of pH 7-8, followed by dilution with more water. Addition of alcohols was sometimes necessary to remove any cloudiness in the solution.<sup>14</sup>

All methods of adduct testing and evaluation were carried out according to standard specifications, except where otherwise stated.

## Results and discussion

### Reaction with acrylonitrile

To explore the vexed problem of gelation, it was attempted to find out whether or not the carboxyl group (or the anhydride) was responsible for it. Maleic anhydride was replaced by acrylonitrile to avoid any possible participation of the carboxyl groups in gel formation. The acrylonitrile adduct was then hydrolysed in concentrated hydrochloric acid/acetone. This was preferred to alkaline hydrolysis because it avoids saponification.

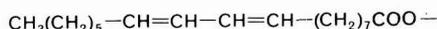
The physical constants and characteristics of DCO, the acrylonitrile adduct and the hydrolysed adduct were determined, and these are shown in Table I.

The following equation represents the addition of acrylonitrile to 9, 11-linoleic acid ester, obtained in the dehydration of ricinoleic acid of the castor oil.

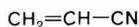
Table 1

Physical constants and characteristics of DCO, acrylonitrile adduct and acid-hydrolysed adduct

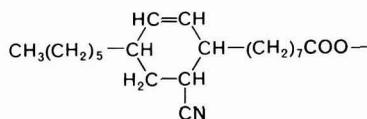
	Acrylonitrile adduct of DCO:		
	DCO	before hydrolysis	after hydrolysis
Colour (Gardner)	.. .. 4-5	5	5
Viscosity (Gardner)	.. .. 1	1	1
Water solubility	.. .. insoluble	insoluble	disperses
Viscosity increase after 3 hours heating at 150°C	.. .. none	none	slight
Acid Value mg KOH g <sup>-1</sup>	.. .. 10.9	10.0	28.6
Saponification value mg KOH g <sup>-1</sup>	.. .. 188.4	210.0	207.3
Hydroxyl value mg KOH g <sup>-1</sup>	.. .. 30.9	30.9	30.3
Iodine value g I <sub>2</sub> g <sup>-1</sup>	.. .. 1.425	0.681	0.68
Diene value per cent	.. .. 23.3	12.7	12.7



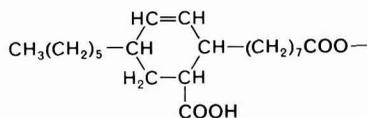
+



(Hydroquinone)



Hydrolysis



The above equations are supported by the facts that:

- There is an increase in viscosity of the hydrolysed adduct when heated at 150°C. This is probably due to the condensation of the carboxyl groups with the residual hydroxyl groups of DCO.
- The hydrolysed adduct has a higher saponification value than that of the parent oil. The acid value is also higher in a proportion corresponding to the initial double bond conjugation in the oil.
- The unsaturation of the adduct is less than that of the parent oil, as indicated by the iodine and diene values.

It may be concluded, therefore, that the replacement of maleic anhydride by acrylonitrile in performing the Diels-Alder reaction has reduced the tendency of the adduct to gel.

#### Reaction with acetic anhydride and phosphorus pentachloride

Since it was established that the carboxylic group is a reactive site for gelation, tests with the DCO molecule were made in such a way that any residual hydroxyl groups—probably responsible for gelation by reacting with carboxyl groups—

would be eliminated. Replacement of these hydroxyl groups by acetate groups and chlorine atoms was made, prior to maleinisation, in the ways already indicated. The results obtained are given in Table 2 and 3.

Table 2  
Physical characteristics of acetylated and chlorinated DCO

	Acetylated DCO	Chlorinated DCO
Colour (Gardner)	.. .. 5	5
Viscosity (Gardner)	.. .. 1	1
Water solubility	.. .. insoluble	insoluble
Acid value mg KOH g <sup>-1</sup>	.. .. 12.0	12.0
Saponification value mg KOH g <sup>-1</sup>	.. .. 215.6	220.3
Hydroxyl value mg KOH g <sup>-1</sup>	.. .. 2.0	2.7
Iodine value g I <sub>2</sub> g <sup>-1</sup>	.. .. 1.401	1.405
Diene value (%)	.. .. 23.3	23.3

Table 3  
Gelation during the maleinisation of treated DCO

Maleic anhydride (%)	Acetylated DCO	Chlorinated DCO
5	no gelation	no gelation
10	gel formation	" "
15	" "	" "
20	" "	" "
25	" "	" "

It can be shown that chlorinating DCO does not alter to any appreciable extent the physical characteristics of the oil, but that the chlorinated oil does not show any sign of gelation when treated with as much as 25 per cent maleic anhydride.

#### Characteristics of maleinised chlorinated DCO

Ref: 14-16

The maleic adducts of chlorinated DCO were tested for water solubility and for other physical characteristics and the results are shown in Table 4.

Table 4  
Characteristics of different maleic adducts of chlorinated DCO

	Maleic anhydride (Per cent)			
	10	15	20	25
Colour (Gardner)	.. .. 11	12	13	13
Viscosity	.. .. viscous	viscous	viscous	gel-like
Water solubility	.. .. disperses	soluble	soluble	soluble
Aniline number <sup>15</sup> mg KOH g <sup>-1</sup>	.. .. 97.2	138.7	182.4	225.7
Saponification value mg KOH g <sup>-1</sup>	.. .. 260.0	291.9	326.4	360.0
Hydroxyl value mg KOH g <sup>-1</sup>	.. .. 3.0	1.2	1.8	1.5
Iodine value g I <sub>2</sub> g <sup>-1</sup>	.. .. 92.0	74.0	57.0	34.0
Diene value (%) spectrophotometry	.. .. 2.4	1.1	0.7	0.7

The residual hydroxyl groups in the oil seem to play a very important part in promoting gelation, since it has now been shown that blocking most of these groups leads to the possibility of producing non-gelling adducts from DCO alone, without having to blend it with other non-conjugated oils.<sup>14, 16</sup>

#### Discrepancies in acid and saponification values

Ref: 14

The adducts of maleinised chlorinated DCO were found to have lower acid and saponification values than the theoretical

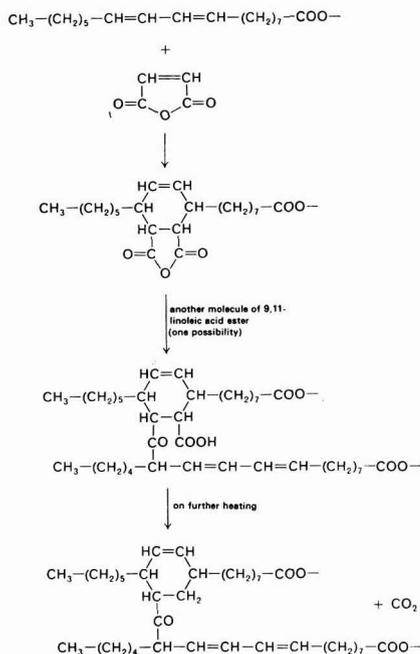
values, even allowing for the amount of carbon dioxide evolved during the reaction. These discrepancies are shown in Table 5.

Table 5

Acid and saponification values of maleic adducts of chlorinated DCO

Maleic anhydride (per cent)	Acid value mg KOH g <sup>-1</sup>		Saponification value mg KOH g <sup>-1</sup>	
	Calculated	Found	Calculated	Found
10	121.2	97.2	283.2	260.0
15	176.8	138.7	329.8	291.9
20	232.4	182.4	376.4	326.4
25	288.0	225.7	423.0	360.1

The total loss in acidity represents about 20 per cent of the theoretical value. This loss was found to be equivalent to twice the amount of carbon dioxide evolved, which agrees with the results obtained in previous work.<sup>14</sup> The loss in acidity can be illustrated by the following equations taking 9,11-linoleic acid ester as an example:



### Viscosity reduction studies

Maleic adducts of chlorinated DCO containing 15 per cent maleic anhydride or more, were water solubilised by neutralising with dilute ammonium hydroxide and thinning with water to the required solids content. The pH of the solution was adjusted to be in the range 7 to 8. These aqueous solutions are very viscous and can not be used in this condition. It was necessary to reduce the viscosity to within the range suitable for application, whilst keeping a suitable solids content. Ethyl alcohol was chosen as a viscosity reducing agent. The viscosity was followed using the Ford Cup No. 4 and the

results are shown in Figs. 1 and 2. The conclusions drawn from these Figs. are:

(a) The viscosity is rapidly reduced when a content as low as 15 per cent ethyl alcohol is present in the aqueous solvent (Fig. 1).

(b) The solid content in the aqueous mixture has a significant role. Whilst it is possible to reduce sharply the viscosity of a solution with a solid content of about 45 per cent with 10 per cent ethyl alcohol, it is not possible to do so when the solid content is higher than this (Fig. 2).

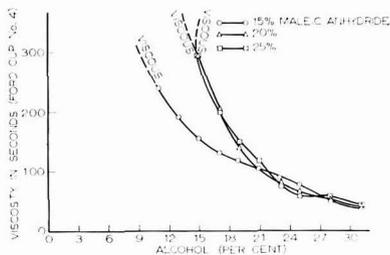


Fig. 1. The effect of ethyl alcohol on viscosity

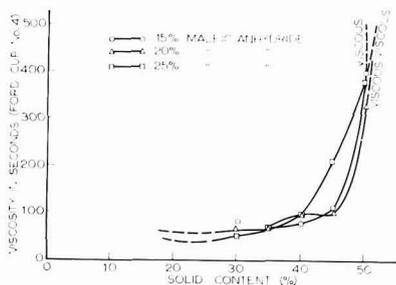


Fig. 2. Viscosity reduction with 10 per cent ethyl alcohol solution

### Film properties

Films of these aqueous solutions dried in air in less than 24 hours with tackiness, even when manganese acetate was used as a drier. Further evaluation of this water-soluble oil as a plasticiser for hard water-soluble resins is in progress.

### Conclusions

A simple treatment of dehydrated castor oil with phosphorus pentachloride removes the residual hydroxyl groups and allows smooth maleinisation of the treated oil with up to 25 per cent maleic anhydride without any gelation. These oil/maleic adducts are soluble in aqueous ammonia and their solutions are film-formers. The high viscosity of the solutions can be reduced to application viscosity by small additions of ethyl alcohol; for example, the viscosity of the 45 per cent solids content solution can be reduced to about 100 seconds (in Ford Cup No 4) when 10 per cent aqueous ethyl alcohol is used instead of pure water.

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# Painting concrete\*

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## Summary

When painting a concrete surface, its condition or character is of the highest importance for the final result. The influence of different mechanical and chemical surface treatments on the strength of the concrete and on the adhesion of paints has been studied. The surface strength of different concrete surfaces and also the adhesion of paints was measured by the "pull-off" method. Concrete with low water-cement ratio (wcr) and with a smooth surface cast against a mould had high surface strength. On free horizontal concrete surfaces, a laitance is formed especially if the wcr is high (much water). This layer is carbonated continuously by carbon dioxide from the air.

The alkaline character of concrete surfaces was studied. The pH

## Keywords

*Types and classes of structures or surfaces to be coated*  
concrete

was found to be from 10 to 12, depending on the age of the surface. Two-component paints gave good adhesion (3.5–4.0 Newtons  $\text{mm}^{-2}$ ) and latex paints somewhat lower values (1.0–2.0 Newtons  $\text{mm}^{-2}$ ). Good adhesion was achieved even when the paints were applied to fresh concrete (20 hours), with the exception of an alkyd paint.

The penetration of different paints and varnishes into concrete was studied on cross-sections from painted concrete blocks. The best penetration (1–3mm) was obtained with diluted binders consisting of low molecular and polar resins (for example, liquid grades of epoxy resins). Pigments and latex particles are too large to penetrate into concrete.

Moisture transport and water loss from painted concrete and the problem of efflorescence has been investigated.

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

adhesion  
permeability

*structures or surfaces being coated*

efflorescence  
paintability

## La peinture de béton

### Résumé

En ce qui concerne le peignage d'une surface de béton, sa condition et sa nature ont une importance primordiale au point de vue du rendement ultime d'une couche de peinture appliquée là-dessus. On a étudié l'influence qu'exercent quelques divers traitements mécanique ou chimique de la surface de béton sur la résistance de cette surface et également sur l'adhérence de peintures. La résistance des surfaces de divers types de béton ainsi que l'adhérence de peintures ont été mesurées par une méthode d'arrachement. Dans le cas où l'on coule un béton de faible rapport eau-ciment, la surface attenante au moule possède une résistance élevée. Sur les surfaces libres et horizontales de béton une laitance se forme, surtout s'il s'agit d'un béton de rapport eau-ciment élevé (beaucoup d'eau). Cette couche de laitance se carbonate continuellement grâce à l'acide carbonique dans l'air.

On a étudié la nature alcaline des surfaces de béton. On a trouvé que le pH est de 10 à 12, selon l'âge de la surface. Les peintures à

deux emballages donnent une bonne adhérence (3.5–4.0 Newtons  $\text{mm}^{-2}$ ) et les peintures-latex donnent les valeurs un peu plus basses (1.0–2.0 Newtons  $\text{mm}^{-2}$ ). On a obtenu de bonne adhérence même dans le cas où les peintures ont été appliquées au béton frais (20 heures), sauf où il s'agit de peintures alkydes.

On a étudié la pénétration du béton par de diverse peintures et vernis à l'aide des sections transversales découpées des blocs de béton peint. Le meilleur niveau de pénétration (1–3mm.) s'est produit par les liants dilués à base des résines polaires de bas poids moléculaire, (par exemple les variétés liquides des résines époxydes). Les particules de pigments ou de latices sont trop grandes pour pénétrer le béton.

On a étudié le transport de l'humidité et la perte de l'eau à partir du béton peint ainsi que le problème de l'efflorescence.

## Betonanstrich

### Zusammenfassung

Beim Anstrich von Betonoberflächen ist deren Zustand und Charakter von höchster Bedeutung für das Endresultat. Die Einwirkungen verschiedener mechanischer und chemischer Oberflächenbehandlungen auf die Festigkeit des Betons und die Haftung von Anstrichmitteln wurden untersucht. Die Oberflächenfestigkeit verschiedener Betonoberflächen und ebenfalls die Haftung von Anstrichfarben wurde unter Benutzung der "pull-off" (Abzugs-) Methode gemessen. Beton mit einem niedrigen Wasser-Zementverhältnis (wcr) und glattem Oberflächenguss nach Berührung der Formwandung besass hohe Oberflächenfestigkeit. An horizontalen Betonoberflächen im Freien bildet sich eine Art Zementschaum besonders, wenn das wcr hoch ist (viel Wasser). Diese Schicht wird fortgesetzt durch Kohlensäure aus der Luft karbonisiert.

Der alkalische Charakter von Beton wurde untersucht. Es wurde festgestellt, dass je nach dem Alter der Oberfläche, der pH von

10 bis zu 12 reichte. Zweikomponentenlacke zeigten gute Haftung (3.5–4 Newtons  $\text{mm}^{-2}$ ), Latexfarben etwas niedrigere Werte (1.0–2.0 Newtons  $\text{mm}^{-2}$ ). Mit Ausnahme von Alkydharzfarben konnte, selbst wenn auf frischen Beton (20 Std.) aufgemalt, mit anderen Prüflacken, gute Haftung erzielt werden.

An Querschnitten gestrichener Betonklötzer wurde die Eindringung verschiedener Anstrichfarben und Klarlacke in den Beton untersucht. Die beste Eindringungstiefe (1–3mm) wurde mit verdünnten Bindemitteln aus niedermolekularen und polaren Harzen erhalten (z.B. flüssigen Epoxyharzen). Pigmente und Latexpartikel sind zu gross, um in den Beton eindringen zu können.

Untersucht wurden ebenfalls Wassertransport und Wasserverlust aus gestrichenem Beton und das Problem der Ausblühungen.

\*European Liaison Lecture presented to the London Section on 8 November 1972.



deterioration mechanisms occur in the pore structure. The gel pores are so small, however, that ice crystals cannot form since no more than 10-15 molecules of water can occupy a gel pore; the water is supercooled. Capillary pores, on the other hand, are sufficiently large to accommodate ice crystals and the water will freeze. Capillary pores are more numerous and larger, the higher the wcr and the shorter the period of curing. This immediately suggests the benefit from a low wcr in reducing the capillary cavities. Bubbles of entrained air are not generally filled with water unless the concrete becomes saturated by means of vacuum or pressure.

In spite of its porous structure, a cement paste is nearly impermeable to water. It is reported in the literature that a cement paste with wcr of 0.40 is as water impermeable as marble. For higher wcrs, the permeability increases exponentially with the wcr. The measured water permeability into concrete (cement paste + aggregates) is higher than that for pure cement paste due to shrinkage between paste and aggregates, and imperfect packing of the plastic concrete mixture during vibration.

#### Laitance layer

When a concrete mixture is vibrated, water is forced up to the horizontal surface of the concrete. This phenomenon is called "water separation" or "bleeding" and increases with the wcr and time of vibration. This water-rich top layer forms during the drying-out of a mechanically weak layer called "laitance", which also may contain some dehydrated cement and small badly wetted aggregate particles. In addition to poor mechanical properties, the laitance has a chemical composition different from the rest of the concrete because the wet concrete surface is alkaline and reacts with the carbon dioxide in the air.

The fresh alkaline laitance is also attacked by sulfur oxides in the air when voluminous calcium sulfoaluminates are formed, causing scaling. Concrete surfaces with this laitance are obviously bad substrates to paint and some of the problems concerning painting concrete are connected with removal of the laitance, obtaining good adhesion or anchoring, and with which types of paints are to be chosen. The problems are more severe when the concrete is cast horizontally rather than against vertical surfaces in a mould. Vertical surfaces, in general, are smooth and have no typical laitance; on the other hand, painting problems may occur due to residues of "mould oil" (release agent). In this investigation, the more difficult horizontally cast concrete surfaces have received most attention.

#### Types of finished concrete surfaces

A concrete surface which is to be painted may have been finished or treated in many different ways, depending on whether the surface is a floor, wall, ceiling or whether the concrete is fresh or old. Some methods are summarised below:

##### Mechanical methods

###### Plastic concrete

striking off  
floating  
steel floating  
power floating  
vacuum treated  
surface vibration  
brushing (bass broom)

###### Hardened concrete

brushing (wire brush)  
mechanical grinding  
sand blasting  
cutting with concrete saw

##### Chemical methods

surface reinforcing agent  
exposing of aggregates  
etching with e.g. 3 per cent HCl  
fluorinating with fluorosilicates  
impregnation with organic binder  
impregnation with hydrophobic agent

##### Thermal methods

flame cleaning with  
gas burner

Not all the types of concrete surfaces mentioned have been tested in this investigation, but most of them have.

## Experimental

### The concrete surface

The aim of this part of the investigation was to study how the surface strength of concrete was influenced by such parameters as type or method of finishing, curing time, humidity and wcr. The concrete was prepared at the Institute. A "standard" concrete was specified with w-c ratio 0.60 and weight ratio cement to aggregate 1:3.5. A Portland cement type called "Rapid" was used and the aggregates had a particle diameter of up to 8mm. The concrete was prepared in a mechanical kneader, cast in shallow plastics moulds (height 3cm) and vibrated on a vibration table. After casting, the concrete was allowed to cure for nine days at 50 per cent RH and 23°C, unless otherwise stated.

### The pull-off method

The principle of this technique is to glue a cylindrical piece of metal (dolly) directly to the surface to be examined and pull it off. A tensile strength fracture will occur where the system is weakest. Dollies with a contact surface of 3.14 cm<sup>2</sup> were used and the two-component glue was Epikote 815/Versamid 140 (1:1). By treating the epoxy glue at approximately 50°C, the curing time could be reduced considerably. After 24 hours of curing the "dollies" were pulled off with a pneumatic adhesion tester, recently developed at the Institute (Fig. 3). Some of the advantages of the pneumatic adhesion

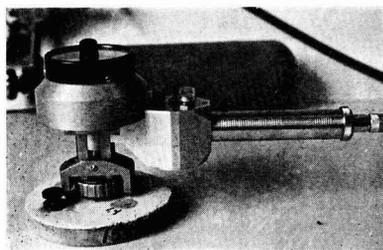


Fig. 3. Pneumatic tensile tester; compressed air is connected to the handle

instrument are that there is no risk of disturbing forces and it is a convenient working procedure. The maximum tensile strength for the instrument is 2200 N and the accuracy is  $\pm 0.05$  N mm<sup>-2</sup>. In most cases, especially when measuring on concrete, it was necessary to carry out ten measurements for a system to obtain a good statistical average of the tensile strength. In this investigation, the statistical *t*-test was used to evaluate the significance of the effects of various parameters on a pull-off measurement.

Table 1

Finishing methods and surface strength ( $N\text{ mm}^{-2}$ ) of concrete. Concrete: wcr = 0.60; cured nine days at 50% RH and 23°C

Finishing method	1 Steel floated	2 Wood floated	3 Steel brushed 18h after casting	4 Vacuum treated steel floated	5 Steel floated etched with 3% HCl	6 Plane mould surface
M*	2.02 ± 0.26	1.60 ± 0.30	2.10 ± 0.47	2.12 ± 0.55	1.57 ± 0.43	2.90 ± 0.37
n**	10	10	10	10	9	10
s†	0.37	0.41	0.65	0.77	0.58	0.52

\* Mean with 95% confidence interval.

\*\* Number of observations.

† Standard deviation at 95% level of significance.

*t*-test between M for surfaces 1 to 6:

1 2 3 4 5 6

1	+	-	-	-	+
2		+	+	-	+
3			+	-	+
4				+	+
5					+
6					

+ = Significant (95%) difference

- = No significant (95%) difference

The surface tensile strength for different types of finished concrete surfaces is given in Table 1. The most significant tensile strength measurement ( $2.90\text{ N mm}^{-2}$ ) was recorded for a smooth surface cast against a plane mould (of plastic). Steel-floated, wire-brushed and vacuum-treated concrete gave values around  $2.0\text{ N mm}^{-2}$ . The lowest surface strength was measured for wood-floated and HCl-etched surfaces (both around  $1.5\text{ N mm}^{-2}$ ).

The reason for low surface strength for the two last-mentioned methods of finishing may be explained by the fact that during the floating (with wood float), aggregates are raised up to the surface and by an etching process, the cement paste is attacked and dissolved; thus, the aggregates near the concrete surface are no longer so well anchored in the cement paste.

Changes in the surface strength of concrete according to the curing time was studied on steel floated "standard" concrete. The experiment indicated that the surface strength increased from  $0.8\text{ N mm}^{-2}$  to  $1.6\text{ N mm}^{-2}$  when the curing time was increased from two to seven days. The influence of carbon dioxide was studied by curing similar concrete blocks for nine days in pure carbon dioxide and in pure nitrogen atmospheres. The result was a rather drastic change in

surface strength from  $\text{CO}_2$  to  $4.1\text{ N mm}^{-2}$  for  $\text{N}_2$ . The concrete cured in carbon dioxide was covered by a mechanically weak, powder-like, carbonated layer which could be removed very easily.

Furthermore, it was found that a decrease in the wcr gave rise to a higher surface strength in accordance with the hypothesis that lower wcrs means stronger concrete (Table 2).

The compressive strength of the "standard" concrete investigated was approximately  $36\text{ N mm}^{-2}$  and the measured tensile strength for this concrete varied between 1.0 and  $4.0\text{ N mm}^{-2}$ . The surface strength is thus roughly 5-10 per cent of the compressive strength. An exact relationship between compressive strength and surface strength cannot exist due to the very varying character and properties of the surfaces of different types of concrete.

When measuring the tensile strength by the pull-off method, most of the fractures occurred at a depth of 1-3mm in the concrete depending on the type of surface and the concrete mixture. Fig. 4 shows the material pulled off from an unpainted concrete surface by a "dolly". A higher value of tensile strength means, generally, that more material is pulled from the substrate.

Table 2

Influence of wcr on concrete surface strength ( $N\text{ mm}^{-2}$ ). Concrete: steel floated, 25 days curing at 50% RH and 23°C

Water-cement ratio	0.50	0.55	0.60	0.65	0.70	0.75
M	2.30 ± 0.50	2.39 ± 0.35	2.04 ± 0.32	1.86 ± 0.24	1.17 ± 0.29	0.82 ± 0.22
n	8	8	10	10	9	9
s	0.60	0.42	0.37	0.34	0.37	0.30

*t*-test between means:

0.50 0.55 0.60 0.65 0.70 0.75

0.50	-	-	+	+	+
0.55		+	+	+	+
0.60			-	+	+
0.65				+	+
0.70					+

+ = Significant (95%) difference

- = No significant (95%) difference

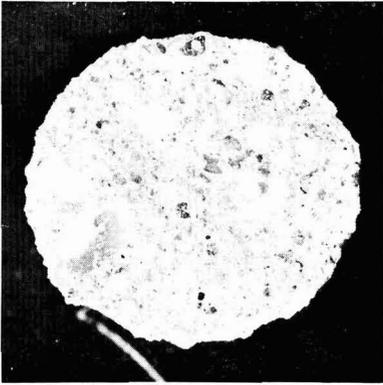
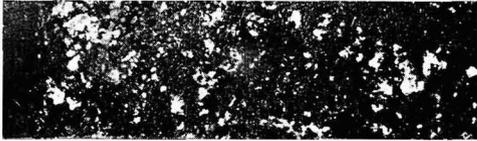


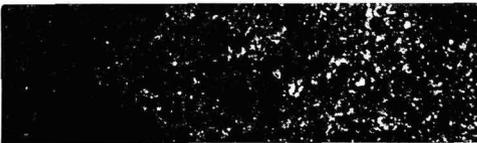
Fig. 4. The back of a pulled-off dolly with materials from the concrete



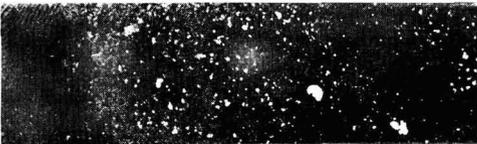
Float treated, wcr = 0.60



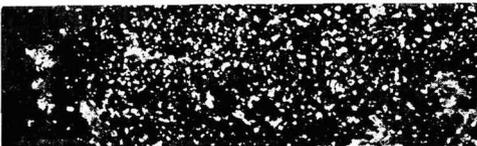
Vacuum treated, wcr = 0.60



Etched with 3% HCl, wcr = 0.60



Steel floated, wcr = 0.50



Steel floated, wcr = 0.60



Steel floated, wcr = 0.70

Fig. 5. Pull-off with black tape from different treated concrete surfaces. Approximately 1.5X magnification

### *The tape method*

The pull-off method can have one disadvantage when measuring tensile strength directly on a concrete surface, and this is due to the penetration of the epoxy glue into the concrete so that a reinforcing effect is achieved. The pull-off value is then no longer representative for the concrete surface itself. This problem is especially pronounced for concrete with a porous laitance. To avoid this problem, some measurements were carried out using a black elastic pvc-tape, which was peeled off by hand. The amount of loosely anchored particles peeled off by the tape was judged visually.

Some of the results are shown in Figs. 5 and 6. The same concrete samples were chosen as those investigated by the pull-off method. Little material from the substrate was peeled off the plane concrete surfaces moulded against a plane plastic mould. On the other hand, an increase in wcr caused more material to be peeled off.



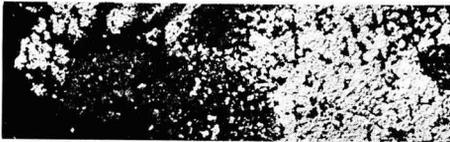
Mould surface from plastic foil coated board



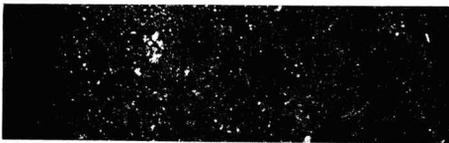
Mould surface from a plane plastic mould



Steel floated surface. The concrete cured in 0% RH during the first nine days



Steel floated surface. The concrete cured in 100% RH during the first nine days



Steel floated surface. The concrete cured in pure N<sup>2</sup> during the first nine days



Steel floated surface. The concrete cured in pure CO<sup>2</sup> during the first nine days

Fig. 6. Pull-off with black tape from different treated concrete surfaces. Approximately 1.5X magnification

It could be stated that the tape method is valuable in giving an idea of the amount of loosely anchored particles on the concrete surface, whilst the pull-off method gives some idea of the mechanical strength of the concrete surface.

The alkaline character of concrete surfaces is due to the formation of calcium hydroxide during the hydration reaction between cement and water. If a phenolphthalein pH-indicator is dripped on to a surface of fresh concrete (some days old), a colour change to red ( $\text{pH} > 10$ ) will occur. Older concrete (more than three months) will give very little or no red colour, depending on the degree of carbonation. If much indicator liquid is applied to the surface, alkaline salts deeper down in the concrete may be leached out and effect the alkalinity of the surface. On fresh concrete, an increasingly thick layer of carbonates is built up as the alkalinity decreases. If this thin layer is scraped off, the surface is again highly alkaline, so that intense mechanical treatment (eg grinding, steel brushing, blasting etc.) of the concrete surface will increase its alkalinity.

In addition to the investigation with a liquid indicator, alkalinity was studied by an electrical method. A specified volume (areas  $12\text{cm}^2$ , depth 1mm) of material was scraped from test specimens cast in pure cement after periods of from one day up to three months. The powder removed was dispersed in a specified amount of water and after one day the pH was measured. The results are given in Table 3. The pH decreased from 11.7 to 10.4 during the three months. As a reference, it can be mentioned that a saturated solution of calcium hydroxide has a pH of 12.4.

The neutralised carbonated layer must, therefore, be very thin and paint binders penetrating 1-2mm into the concrete will always be affected by alkali. Non-penetrating products will not be affected to the same degree, unless alkaline water inside the concrete is transported to the surface.

## Painted concrete

### Adhesion

The adhesion of five laboratory prepared paints (Table 4) to concrete, cast and surface treated in different ways, was studied. The adhesion was measured with the new automatic pull-off instrument. It is difficult to give an exact figure for the lowest acceptable adhesion, but a realistic figure is  $0.8\text{ N mm}^{-2}$  for architectural wall paints and  $1.2\text{ N mm}^{-2}$  for concrete floor paints.

The importance of different pretreatment methods on adhesion was studied on standard concrete for the acrylic latex paint (exterior wall paint) and the 2-component epoxy/urethane paint (floor paint).

The paints were applied 18 hours after the concrete had been cast, at a thickness of  $80\text{ }\mu\text{m}$  dry paint film. After nine days of curing ( $23^\circ\text{C}$ , 50 per cent RH), the adhesion was measured. The results are given in Table 5. For both paints, the best adhesion (pull-off values) was obtained on smooth concrete surfaces cast against a plastic mould. Steel-floated, acid-etched (3 per cent HCl) surfaces also gave good adhesion with both paints.

In pulling off the "dollies" attached to the paint films, the fractures occurred 0.1-0.2mm down in the concrete. In fact, the cohesive strength of the concrete surface itself had been measured in this case, but the pull-off value was also influenced by the type of paint and surface character. Higher pull-off values caused more material to be pulled off from the concrete.

Extensive studies concerning the effect of the age of the concrete on adhesion gave no significant effect for the paints investigated. The age of the concrete varied from

Table 3  
pH determination on scrapings from cement specimens

Curing time	20h	2 days	3 days	6 days	10 days	3 months
pH	11.7	11.4	11.0	10.8	10.6	10.4

Table 4  
Data for NIF-prepared paints

	Pva latex	Acrylic latex	Chlorinated rubber	Epoxy/urethane	Alkyd
Binder	Borvimal H10	Rhoplex AC-34	Pergut S10	Epikote 1009/ Desmodur L	Plexal P68
TiO <sub>2</sub>	RNCX	RN-47	RNCX	RNCX	RNCX
PVC	33	40	44	20	27
Non-volatile content %	53	58	57	60	77
Viscosity	92 KU	81-87 KU	70-75 KU	—	85-90 KU

Table 5  
Influence of pretreatments on adhesion ( $\text{N mm}^{-2}$ ) for two paints. Concrete: wcr 0.60, curing time eight days at 50% RH and  $23^\circ\text{C}$

		1 Plane mould surface	2 Steel floated	3 Wood floated	4 Levelling with board	5 Vacuum treated steel floated	6 Steel floated acid etched
Epoxy urethane	M	$3.20 \pm 0.56$	$1.12 \pm 0.25$	$0.39 \pm 0.12$	$0.78 \pm 0.31$	$2.11 \pm 0.30$	$3.59 \pm 0.29$
	n	9	10	9	9	10	10
	s	0.73	0.35	0.15	0.40	0.35	0.37
Acrylic latex	M	$2.76 \pm 0.42$	$0.77 \pm 0.13$	$0.64 \pm 0.13$	$0.85 \pm 0.20$	$0.99 \pm 0.24$	$1.72 \pm 0.31$
	n	10	13	12	12	13	10
	s	0.59	0.22	0.21	0.32	0.37	0.43

one day to three years. In most experiments, it was proved that good adhesion was achieved, even if the concrete was painted as soon as 18-20 hours after casting, provided that the binder in the paint was alkali resistant.

Early painting prevents too rapid drying of the concrete and the formation of a mechanically weak layer of carbonates, and gives rise to good curing conditions for the concrete. Due to the risk of saponification, the alkyd paint was not used on very freshly cast concrete.

Paint applied to concrete can penetrate only into the porous cement paste and not into the mineral aggregates. In order to study the effect of diluting and priming on adhesion, a more simplified system was used, namely blocks of pure cement paste. For each of the four paints (I pva latex, II acrylic latex, III chlorinated rubber and IV epoxy urethane), the following two-coat systems were examined:

System	I	II	III	IV
1st coat	und	d	unp	P
2nd coat	und	und	und	und

P = vinyl copolymer lacquer.  
 und = undiluted paint.  
 d = diluted paint (20 per cent for latex paints, 10 per cent for solvent based paints.)  
 unp = the paint without pigments or extenders.

The first coat was applied 18 hours after casting. The wcr for the pure cement paste was 0.45 and the painted surfaces were all formed by plane plastic moulds. After drying for ten days, the adhesion was measured and Figs. 7, 8, 9 and 10 are representative of fractures and mean pull-off values for each paint in the four systems shown. From these tests the following conclusions may be drawn:

1. A pure cement paste substrate gave, generally, higher pull-off figures than a concrete one. The epoxy/urethane value was about  $4.0 \text{ N mm}^{-2}$  and those for the two latex paints and the chlorinated rubber paint between 2.8 and  $3.1 \text{ N mm}^{-2}$ .
2. Diluting the paint or priming with a lacquer gave no measurable increase in adhesion, but the type of fracture (system IV) was similar to that for relatively good adhesion.
3. The low value ( $1.0 \text{ N mm}^{-2}$ ) for the chlorinated rubber in system IV was due to non-compatibility between vinyl lacquer and chlorinated rubber.
4. Only the epoxy/urethane showed such good adhesion that material was pulled off from the cement blocks. The pure cement blocks have a higher tensile strength than concrete.
5. Cross-sections of the painted cement blocks examined at a magnification of X 200 showed that no penetration occurred with any of the systems studied.

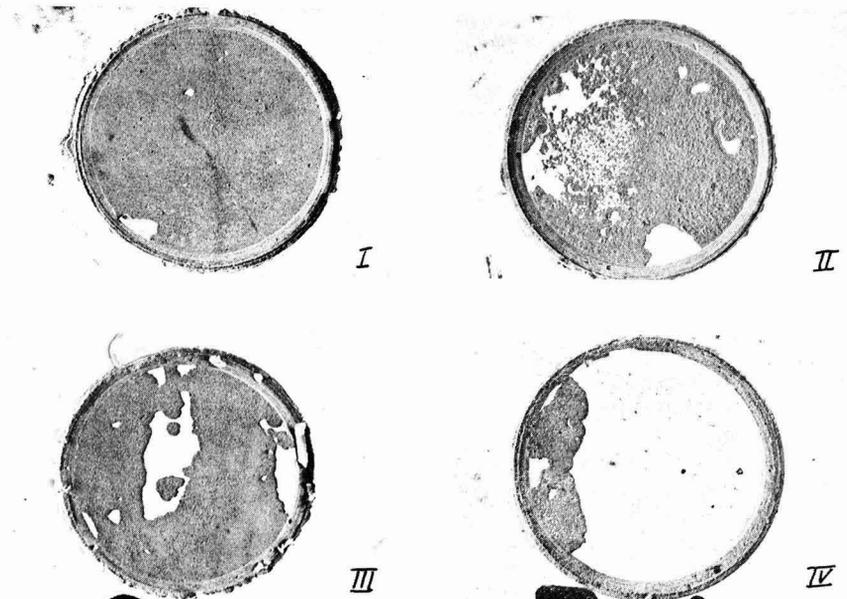


Fig. 7. Pva latex systems I-IV on cement specimens. 2X magnification. Mean tensile strength in  $\text{N mm}^{-2}$ : I = 2.8, II = 3.0, III = 2.1, IV = 3.1

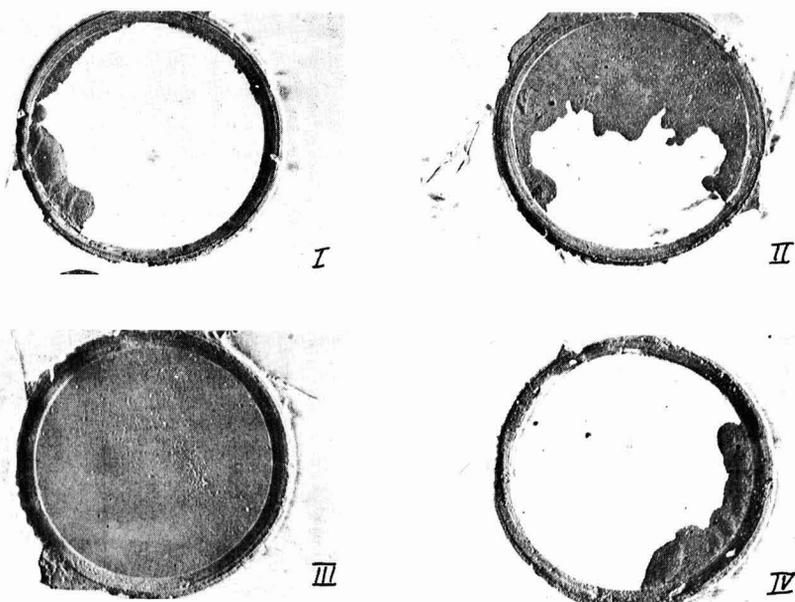


Fig. 8. Acrylic latex systems I-IV on cement specimens. 2X magnification. Mean tensile strength in  $N\ mm^{-2}$ :  
 I = 2.8, II = 3.0, III = 2.3, IV = 2.7

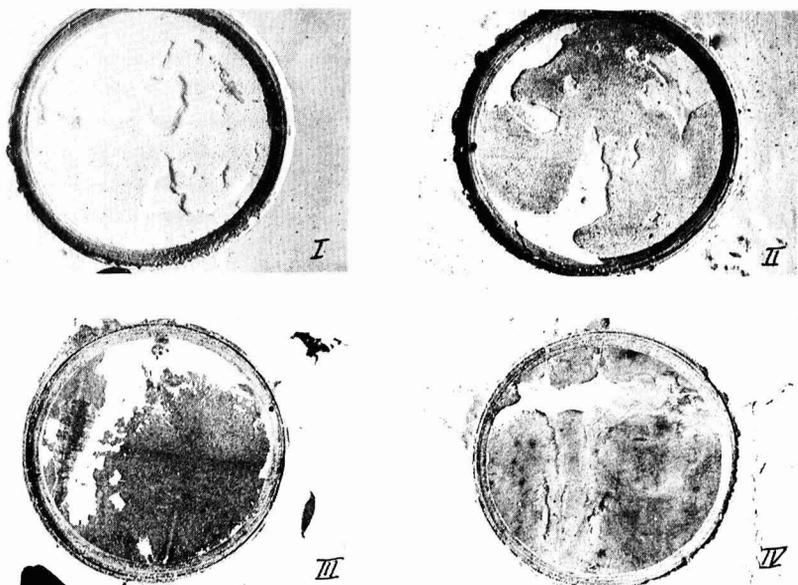


Fig. 9. Chlorinated rubber systems I-IV on cement specimens. 2X magnification. Mean tensile strength in  $N\ mm^{-2}$ :  
 I = 2.7, II = 2.8, III = 3.5, IV = 1.0

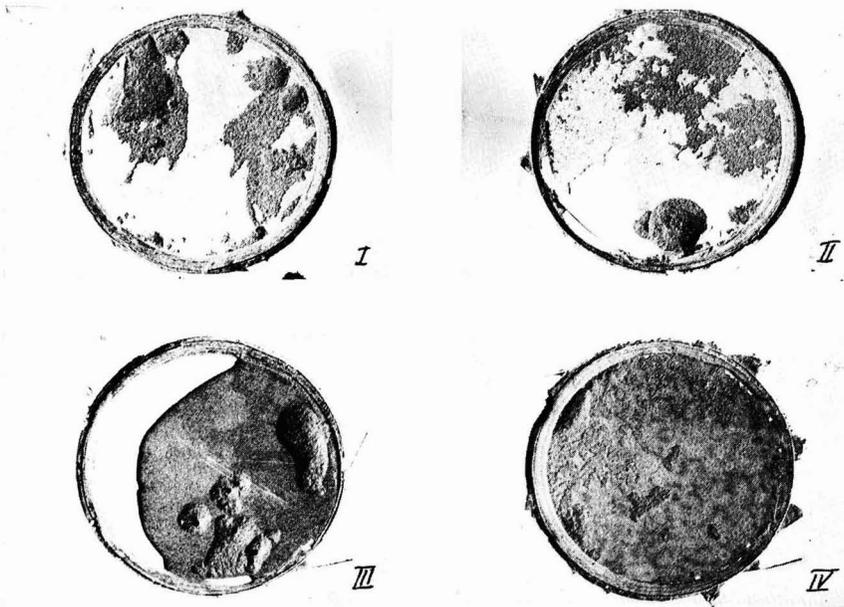


Fig. 10. Epoxy urethane systems I-IV on cement specimens. 2X magnification. Mean tensile strength in  $\text{N mm}^{-2}$ : I = 4.5, II = 4.4, III = 3.8, IV = 3.7

#### Penetration

The penetration of paints, lacquers and varnishes into concrete substrates was studied by cutting out cross-sections with a special concrete sawing-machine. In order to protect the painted concrete surface during cutting, a 2-3mm thick epoxy/amide resin was cast on the top. The cross-sections were examined under a stereo microscope with a maximum magnification of  $\times 200$ . The system under test and the protecting coating of epoxy/amide were suitably dyed to obtain a good visual contrast. The concrete was steel floated and standard concrete composition was used. The paints or lacquers were applied, in general, by brushing 18-20 hours after casting the concrete.

The experiments mentioned in the last section had proved that normally pigmented paints do not penetrate into the cement paste, so unpigmented systems were studied in the first instance. Deep penetration (2-3mm) was obtained with diluted binders of low molecular weight (e.g. epoxy resins). Highly polar (hydrophilic) binders give better penetration. As an example of this, Fig. 11 shows the system Epikote 815/Versamid 140 with 33 per cent solvents (the scale is in mm). The molecular weight is 330 for Epikote 815. About 20 per cent by weight titanium dioxide (Kronos RNCX) with a mean particle size of  $0.3 \mu\text{m}$  was added to the above system. It could now be clearly seen that the pigment particles did not penetrate into the concrete but were filtered on the top of the concrete surface whilst the binder system was absorbed (see Fig. 12: the white line is pigment and above this there is a dark protecting layer of epoxy resin).

The influence of dilution and viscosity was studied by diluting a vinyl-copolymer lacquer with ethanol to 25, 20 and 17 per cent binder. In all cases studied, approximately that theoretical film thickness calculated from the non-volatile content was formed on the top of the concrete

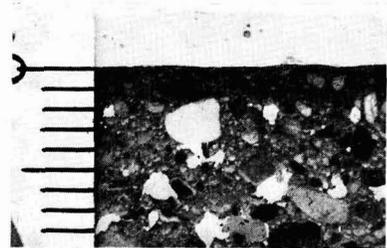


Fig. 11. Penetration of Epikote 815/Versamid 140 diluted with 33% by weight solvent

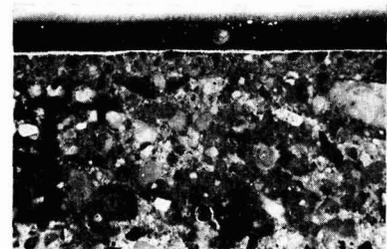


Fig. 12. As for Fig. 11 but with the addition of 2% by weight titanium dioxide

surface so that no penetration had occurred. The molecular weight of the vinyl binder was about 36,000. It should be remembered here that the molecular size of the binder is not even slightly influenced by diluting the system.

Another type of lacquer was also studied, namely chlorinated rubber in three grades of molecular weight: 60,000, 135,000 and 185,000. As with vinyl lacquer, no real penetration was obtained even for the lowest molecular weight system.

The viscosity in all three cases was 3.9 P. An unpigmented alkyd varnish and an acrylic emulsion (Rhoplex AC-34) plus 5 per cent by weight micronised iron oxide were also examined but no penetration was observed.

In one experiment, the latex emulsion (AC-34) was dyed with water-soluble Rhodamine and pigmented with phthalocyanine pigment (Fig. 13). The water phase dyed by the

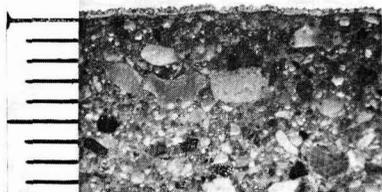


Fig. 13. Penetration of acrylic latex emulsion dyed with Rhodamine FB and pigmented with phthalocyanine blue

Rhodamine was absorbed by the concrete whilst the latex particles and the phthalocyanine particles (both with a diameter range of 0.1-0.4  $\mu\text{m}$ ) had been filtered at the concrete surface forming a dark-blue film. Some of the red spots in this dark-blue film were due to Rhodamine which was retained when some of the water evaporated into the air.

In addition to penetration, there is the possibility of mechanical anchorage of paint in micro cracks formed by the shrinkage of the older concrete, either in the cement paste or between the cement paste and the aggregates. In the small and freshly cast concrete blocks at the author's laboratory, very few such micro cracks were formed.

#### Moisture transport in painted concrete

Moisture transport and movement in porous building materials may be expressed by many complex mathematical expressions representing different phenomena. In this paper, some fundamental principles only will be discussed rather briefly.

When painting a facade of cement plastering, asbestos, concrete brick wall etc, it is important that the paint be permeable for water vapour coming from the inside, but the paint must, at the same time, prevent the wall from absorbing water during rain. If the paint is not sufficiently permeable to water vapour, there is a risk of water condensation in the wall or behind the paint film. The results are decreased heat insulation and paint scaling due to freezing-thawing cycles.

A normal fresh concrete contains an excess of about 80kg water per  $\text{m}^3$  concrete, which will evaporate until the concrete reaches an equilibrium with the surrounding humid air. A fresh concrete, with a weight composition of water/cement/aggregates equal to 0.6:1:5, contains 9 per cent water and 3.5 per cent will be chemically bound to the cement. Such a concrete will contain 1.5 per cent moisture when in equilibrium with air at 50 per cent RH and the rest (4 per

cent) must evaporate. The water in equilibrium with that in the air is called hygroscopic water and changes with the humidity (Fig. 14).

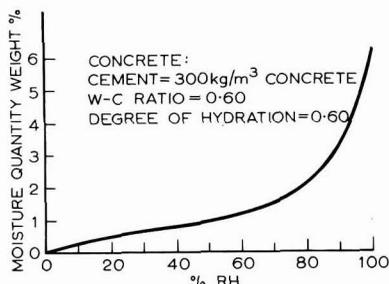


Fig. 14. Absorption isotherm for concrete

Some equations for describing water movements are:

Ficks' first law:

$$G = D (dc/dx)$$

where  $G$  = diffused amount of water vapour per time unit

$x$  = length of diffusion path

$dc/dx$  = concentration gradient

$D$  = diffusion constant

This equation can be transformed to an equation, where a material of thickness  $d$  divides air masses of water of partial pressure  $p_1$  and  $p_2$  respectively

$$G = k_d (p_1 - p_2) d$$

$k_d$  = diffusion constant for the actual material

The value  $k_d$  is expressed in  $\text{g s}^{-1}$ ,  $\text{m}$ ,  $\text{mm H}_g$  and if  $d$  is constant as for thin foils or even paint films, then  $k_d/d$  also is a constant and is expressed in  $(\text{g m}^{-2}, \text{m}, \text{mm H}_g)$ . When free water (e.g. rain and ground water) is in contact with a porous material, such as cement paste, a capillary transport also occurs. For stationary conditions in a cylindrical capillary, the maximum height of water transport is expressed by:

$$H = 2 \sigma \cos \theta / (r \rho g)$$

$H$  = height of capillary transport

$\sigma$  = surface tension of the liquid

$\theta$  = contact angle

$\rho$  = density of the liquid

$g$  = earth acceleration due to gravity

$r$  = capillary radius

If the known data for water is put into the equation and if complete wetting takes place the height of capillary transport is  $1.5 \cdot 10^{-4}/r$ .

The height of capillary transport is inversely proportional to the capillary radius of the material.

#### Water loss of painted concrete blocks

Concrete blocks (diameter 14cm and height 3cm) were cast in plastic moulds and painted to 80  $\mu\text{m}$  dry film thickness with a brush. Seven laboratory manufactured paints were applied 18 hours after the casting. The concrete blocks were kept in the plastic moulds so that the water was forced to evaporate through the paint films.

The painted concrete blocks weighed around 0.6kg so it was possible to follow the evaporation by direct weighing. The balance had an accuracy of 0.1g. Seven different laboratory prepared paints were examined. The evaporation was to some extent accelerated by holding the temperature at 50°C and the air humidity between 18 and 20 per cent RH. The results are given in Fig. 15. Little variation in moisture

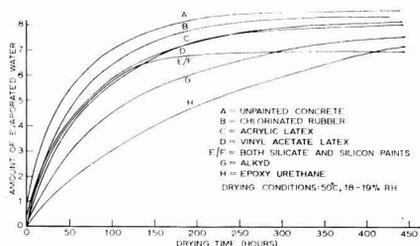


Fig. 15. Drying rate as loss of water from painted concrete blocks at 50°C

loss was registered between unpainted concrete blocks and blocks painted with latex paints. Measurements on concrete blocks painted with a non-porous coating, as for example the two-component epoxy/urethane, gave a drying time approximately four times that of latex paints. A notably high water permeability was observed for the chlorinated rubber paint; on the other hand, the pvc was rather high for this type of paint, namely 44 per cent. Scanning electron micrographs later confirmed that the pigments were surrounded by the binder to a rather low extent. During evaporation, the moisture was also measured with an AUCON moisture indicator, which measures electrically the moisture content 1-2cm down in the concrete directly, when the instrument is placed on the painted concrete surface.

With the indicator instrument, greater differences were noticed between the paints (Fig. 16). The instrument seemed to be valid for an indication of whether water had accumulated under the paint film. When the indicator is used directly on concrete surfaces, there is a risk of obtaining values which are too low because water evaporates rather quickly at the surface but remains for a long period inside the material.

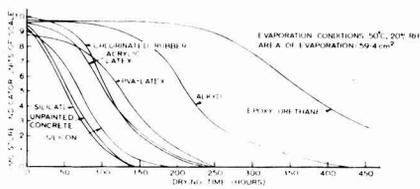


Fig. 16. Drying rates for painted concrete blocks at 50°C measured by the moisture indicator

Moisture transport through painted concrete

The moisture permeability for the combination paint-and-concrete substrate was studied. Test pieces of painted round concrete blocks were attached to light aluminium cups containing distilled water (Fig. 17). The amount of paint applied to the concrete blocks was controlled by weighing.

The moisture transport through concrete and paint was followed by weighing the cups. After an initial period, the

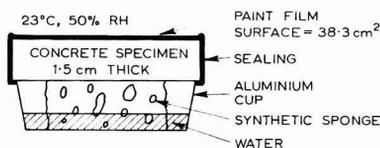


Fig. 17. Method for determining water permeability through painted concrete

weight loss per unit time was constant and the water permeability could be compared for the different systems. The water vapour permeability for the same paints and paint systems is shown in Fig. 18. In this case, a great difference was noticed between painted and unpainted concrete. Notably

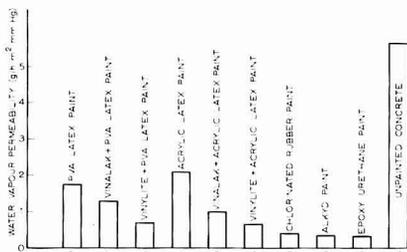


Fig. 18. Water vapour permeability of concrete blocks (1.5cm) coated with paint films of 100µm thickness

that the chlorinated rubber in this case showed low water permeability. It was further confirmed that latex paints, compared to chlorinated rubber, alkyd and epoxy/urethane, have a relatively high moisture permeability. When the concrete surface was primed with vinyl lacquer before application of the two latex paints investigated, the moisture permeability was reduced by nearly half. Studies of moisture transport in larger and thicker painted concrete blocks have been carried out in cooperation with the Technical University of Lund (Sweden). Painted concrete blocks of 15cm thickness were built up as a wall construction between a warm room and a cold one (23°C, 50 per cent RH and 2°C, 70 per cent RH). The moisture content was measured by advanced electronics (resistance principle) at three points for each block. At the beginning of the experiment, the moisture content was 6.2 per cent evenly distributed in all blocks. Paints with very different water vapour permeability were chosen. After six months of testing under the conditions mentioned, the moisture content was measured 1cm behind the paints and in the middle of the 15cm thick concrete block (Table 6).

Table 6  
Moisture content in painted concrete blocks

System (warm side/cold side)	Moisture content (%) warm side/middle/cold side	
1. silicate/unpainted	1.3	5.1
2. chlorinated rubber/acrylic latex	4.6	5.2
3. acrylic latex/silicate	4.2	4.6
4. unpainted/unpainted	1.3	5.5
5. unpainted/acrylic latex	1.3	4.9
6. silicate/acrylic latex	4.4	4.9
7. acrylic latex/epoxy urethane	3.6	4.7
8. epoxy urethane/unpainted	3.2	5.0
9. silicate/epoxy urethane	2.7	5.7
10. unpainted/silicate	1.3	4.8
11. unpainted/unpainted	1.3	5.3

From this Table, it can be concluded that it takes a long time for the moisture to evaporate from the deeper parts of the concrete blocks. The drying out effect seems to be more dominating than the gradient in water vapour (6.3mm Hg), because in most cases the moisture content is lower on the warm side than the cold side.

The experiment was completed after 37 days of freezing and thawing cycles down to  $-15^{\circ}\text{C}$ . It was thought that the cycles should cause scaling of the paint or deterioration of the outer parts of the concrete but this did not occur. The concrete surfaces had been formed against plane varnished wallboard. Any drastic loss in adhesion could not be measured for the paints. Though the paints had been applied to fresh concrete with a high moisture content, no efflorescence salts were noticed on the surfaces.

### Efflorescence

A phenomenon on concrete, bricks, rendering, etc, is the so-called "efflorescence" or "salt deposit," which causes white hazing on facades. The salts consist mainly of calcium hydroxide from the cement. By the time the cement has reacted with carbon dioxide in the air to form calcium carbonate, these efflorescence salts can migrate through paint films. The efflorescence was studied according to two different testing methods. One was the ASTM-standard (D 2831-69) where painted concrete blocks are placed above a water bath with the painted surface facing the water so the specimens are affected by high humidity and also condensed water.

The other method was developed at the Institute and meant simply that test pieces of painted concrete were half immersed in distilled water in a glass dish of known weight. The test block was water insulated so that water transport could take place only through the paint or coating. After 24 hours of leaching, the water has evaporated and the amount of water soluble salts is measured on an analytical balance. If the paint is suspected of containing water soluble components the paint itself may also be tested on the back of a plastic or aluminium dish (same amount of paint on the same area) to obtain a reference value. The amount of salt from the substrate can now be calculated. Some results from different paint systems are shown in Fig. 19. The amount of salts from the concrete itself has been calculated.

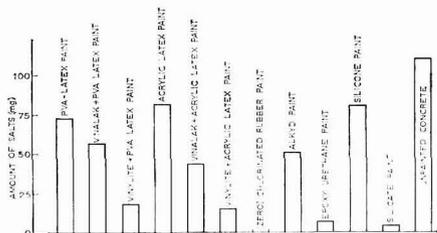


Fig. 19. Amounts of salts leached from the concrete blocks

The test surface was  $38\text{cm}^2$ . From concrete blocks painted with solvent-based paints, very little salt is leached out. For latex paints, the efflorescence is relatively high but can be reduced by priming with, for example, a vinyl lacquer.

### Conclusion

The investigation has shown that the smooth, mould-cast, concrete surface gives good adhesion for most types of paints.

Two-component paints (eg, epoxy) give better adhesion to concrete than latex paints. The alkalinity of a concrete surface just after casting is pH 12 and is reduced by carbonation during three months to approximately pH 10. The surface strength of concrete 1-3mm down in the material can be measured by a pull-off technique. The amount of loosely fixed particles at the concrete surface can be estimated by peeling off a black tape strip.

Good penetration in concrete is best achieved by polar (hydrophilic) and low molecular weight binders (eg, epoxy without pigment). Pigments such as titanium dioxide and phthalocyanine blue do not penetrate into the concrete. Compared to low molecular weight epoxy resin, vinyl and chlorinated rubber resins gave poor penetration. Latex particles in paint dispersions were also too large to penetrate into concrete.

Paints with different capabilities for water permeability will influence the moisture content in a painted concrete wall. The drying out mechanism takes a very long time, so if a concrete surface looks dry it may still contain a lot of moisture in its deeper parts.

The phenomenon called efflorescence (lime deposit) could be studied by a new rapid technique where the salts are leached out by pure water. Latex paints gave more efflorescence than solvent based paints.

### Acknowledgment

The author expresses his gratitude to the National Swedish Building Research, which sponsored this investigation.

[Received 13 June 1973]

### Discussion at London Section

DR V. T. CROWL asked whether the concrete sections shown had been polished.

MR B. LINDBERG said that the surface photographed was as produced by the cutting operation, but to get photographs with sharper contrasts, the cross-sections had been treated with linseed oil.

MR G. V. G. HILL asked what dyes were used to colour the paint media.

MR LINDBERG replied that they had used "Sundanblau II" and also "Neozaponfeuerrot G" as dyes (soluble in organic systems) for registration of penetration. When they tested the latex dispersion, a mixture of phthalocyanine blue pigment and water soluble "Rhodamin FB" dye had been used.

MR T. R. BULLETT asked whether the chlorinated rubber paint used in the salt leaching tests was the same as that which showed a high water-vapour permeability, and if so, how the inhibition of leaching was explained.

MR LINDBERG said that the same chlorinated rubber paint had been used in the two experiments. The drying out experiment had been carried out at an elevated temperature (500hrs at  $50^{\circ}\text{C}$ ), whilst the salt leaching test lasted for only three days. It was possible that checking had occurred during the drying out period. A similar chlorinated rubber paint stored for six months in a room at constant temperature

and humidity showed micro cracking, which was visible only in a scanning electron microscope at a magnification of x 7,000. The PVC of the chlorinated rubber paint was rather high (44 per cent) and if the paint film were hydrophilic, it might swell during the salt leaching test, so that micro cracks were eliminated and only a small amount of the salts was leached out. At higher temperatures (eg 50°C), a lot of micro cracks might be formed.

MR B. L. WOOD commented that a chlorinated rubber coating would show a high permeability to water vapour and still prevent efflorescence because it would have a low permeability to solution.

MR LINDBERG said that both the paint plus the concrete substrate and the paint film itself (on an inert substrate) had been tested by the salt leaching technique. Thus, the amount of water soluble material from the concrete itself had been calculated.

A SPEAKER referred to the penetration of resins into the concrete, as shown in the coloured slides. Solvents were used in these systems. He suggested that the dyestuff might be taken into the concrete by the solvent, rather than by the resin.

MR LINDBERG said that he had been aware of this problem. A solution would be to use radiochemical methods, but this had not been possible.

The non-volatile content of the lacquers investigated and also the amount of material applied were known, and thus it had been possible to estimate the film thickness if no binder had penetrated into the concrete. The film thickness was measured under a microscope. If for example only 50 per cent of the theoretical film thickness were measured on the concrete, then 50 per cent of the binder must penetrate into the concrete.

MR J. BURKETT said that the first cost of painting a building might be less than any exposed aggregate finishing, but over the life of a building it was probably more. He asked why the common concrete surfaces cast against plywood and steel shuttering, and coated with mould oil, had not been considered. He also enquired what was the solution to the problem of choosing a paint which would allow the concrete to dry out, and yet protect it against an aggressive atmosphere.

MR LINDBERG said that one of the major problems considered in the investigation was the effect of the laitance layer on concrete surfaces, which was most pronounced on horizontally cast concrete. All concrete blocks had been cast at the Institute and, therefore, the author had had to work with relatively small concrete blocks.

The second question raised an interesting problem. One solution might be a porous silicate paint, which as a result of the reaction with the carbon dioxide in the atmosphere gradually hardened, thereby forming a protective layer on the concrete.

# Wood varnishes from wattle bark extract

By H. M. Saayman

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## Summary

Wattle bark extract has been used as raw material in the production of polyurethane and epoxy resin surface finishes for wood, the inherent high reactivity of tannins in the extract being modified by partial substitution of reactive groups. These wattle derivatives have been reacted further with isocyanates to produce polyurethane varnishes, or with epichlorhydrin to give epoxy resin surface coatings.

Such coatings prove to be extremely durable and after prolonged

outdoor exposure the surfaces are hard, resistant to abrasion, and retain a high proportion of their original gloss. The finishes would be suitable for application to furniture, exposed timber and wooden boats.

The use of relatively inexpensive wattle extract in the production of sophisticated surface veneers for wood is worthy of serious consideration by the industries concerned.

## Keywords

*Types and classes of coatings and allied products*

varnish  
wood finish

*Miscellaneous*

extract

## Vernis pour bois à partir de l'extrait de l'écorce des acacias

### Resumé

On a utilisé l'extrait de l'écorce des acacias en tant que matière de base pour produire des finitions polyuréthannes ou époxydes pour bois. La réactivité élevée qui est inhérente aux tannins présents dans l'extrait a été modifiée au moyens de la substitution partielle des groupements réactifs. Ces dérivés des acacias ont été encore réagies avec soit les isocyanates afin de rendre les vernis polyuréthannes, soit l'epichlorhydrin pour donner les revêtements époxydes.

Tels revêtements se montrent d'une durabilité extrême, et après

une période prolongée d'exposition aux intempéries, leurs surfaces sont dures, résistantes à l'abrasion et elles gardent une proportion importante de leur premier brillant. Les finitions seraient convenables pour être appliquées aux meubles, aux boiseries à l'extérieure et aux bateaux en bois.

L'emploi éventuel de l'extrait des acacias relativement pas cher pour la production de revêtements de bois avancés est digne d'être considéré sérieusement par les industries intéressées.

## Holzlacke aus Mimosenrindenextrakt

### Zusammenfassung

Mimosenrindenextrakt wurde als Rohstoff zur Erzeugung von Polyurethan- und Epoxyholzlacken verwandt. Die dem Extrakt eigene hohe Reaktivität von Tanninen wird hierfür durch Teilsubstitution reaktiver Gruppen modifiziert. Diese Mimosenrindenderivate wurden weiterhin umgesetzt und zwar mit Isozyanaten, um Polyurethanlacke oder mit Epichlorhydrin, um Epoxylacke zu erzeugen.

Solche Lacke erweisen sich als ungemein dauerhaft; nach längerer

Bewitterung sind die Oberflächen hart, abriebfest und halten einen hohen Prozentsatz ihres ursprünglichen Glanzes aufrecht. Solche Lacke dürften sich zum Lackieren von Möbeln, dem Wetter ausgesetzten Bauhölzern und Holzbooten eignen.

Die Verwendung des billigen Mimosenrindenextraktes für komplizierte Holzfurniere ist ernstlicher Betrachtung seitens der interessierten Industrien wert.

## Лаки для дерева из экстракта коры плетени

### Резюме

Экстракт из плетеной коры применялся в качестве сырого материала в производстве полиуретановых и эпоксидных смольных поверхностных покрытий для дерева, где существенная высокая реактивность танинов в экстракте модифицируется частичным замещением реактивных групп. Эти производные плетени подвергались далее реакции с изоциановыми эфирами для получения полиуретановых лаков или с эпихлоргидрином для получения эпоксидных смольных поверхностных покрытий.

Такие покрытия являются исключительно прочными и после

продолжительного выветривания на свежем воздухе поверхности остаются твердыми, устойчивыми против истирания и сохраняют высокую пропорцию их начального лоска. Покрытия пригодны для применения на мебели, обнаженного дерева и деревянных лодок.

Применение сравнительно дешевого экстракта плетени в производстве особых поверхностных фанер для дерева, заслуживает серьезного внимания со стороны заинтересованной отрасли промышленности.

## Introduction

*Ref. 1-7*

The bark extract of the black wattle tree (*Acacia mearnsii* De Wild.) contains 60 to 70 per cent of phenolic tannins, as

determined by the official hide powder method of tannin estimation.<sup>1</sup> Tannins are polymers of flavanoid constituents which possess a high proportion of hydroxyl groups.<sup>2,3</sup> These substituent phenolic hydroxyls are highly reactive and their presence provides reactive centres in the ortho- and para-positions of the benzenoid nuclei (Fig. 1).

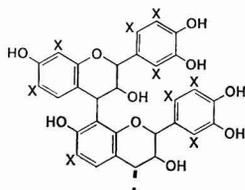


Fig. 1. Part of the tannin molecule, showing reactive centres (X) on the benzenoid nuclei

Such high reactivity is often undesirable and partial or complete substitution of the hydroxyl groups may be effected in order to deactivate these functional groups. Partially substituted wattle tannin derivatives have proved useful as intermediates in further reactions.

Benzoylation by the Schotten-Baumann method<sup>4</sup> and benzoylation of the tannin molecules were used to substitute certain reactive hydroxyls. Such derivatives were used as raw materials for the production of polyurethane surface finishes or lacquers for wood.

A urethane is the product of reaction between an alcohol or a phenol and an isocyanate:<sup>5</sup>



The linkage,  $-NHCO-$ , is similar to that which is present in ethyl carbamate, or urethane. Hence the polyurethanes are polymers which contain a number of such linkages.

Linear polymers are formed between diols and di-isocyanates, whilst triols and di-isocyanates may yield cross-linked network structures.

When small polymers are formed, the resultant products do not possess high tensile strength, hardness, elasticity or flexibility.<sup>6</sup> These properties only become significant when molecular weights above 5,000 are attained.

Isocyanates react with phenols to form urethanes:<sup>7</sup>



In the presence of water the isocyanates react very rapidly, with evolution of carbon dioxide:



Hence, reactions between isocyanates and phenols normally have to be carried out under anhydrous conditions.

Partially methylated wattle extract was used in the production of epoxy resins, which proved suitable as surface coatings for wood.

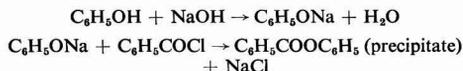
## Experimental

Ref. 8-12

### Benzoylation

In the original Schotten-Baumann acylation method, the phenolic hydroxyl groups of the tannins were reacted with an excess of dilute, aqueous caustic soda solution to yield phenoxides.

A small excess of benzoyl chloride was added and the mixture shaken vigorously. An exothermic reaction occurred spontaneously and the insoluble benzoyl derivative separated out readily. The reaction may be illustrated as follows:



Wattle extract was treated with varied quantities of 10 per cent aqueous caustic soda solution and benzoyl chloride in quick succession. The quantities of reagents were calculated to correspond to substitutions of 2, 3 and 4 hydroxyl groups, in turn, in the tannin monomer.

During recovery of the benzoylated tannins from water, it was noted that whereas the highly substituted derivatives yielded immediate flocculent precipitates, those samples having a lower degree of substitution became progressively more colloidal in nature. Products also exhibited a gradation in colour—from a dark-brown for the lower degree of benzoylation, to a cream coloured precipitate for the higher substituted derivatives. In addition, the yields of derivatives increased proportionately in the above order.

Powdered black wattle extract (3g) was dissolved in water (30ml) and benzoyl chloride (3ml) added. Potassium hydroxide (25ml, 20 per cent wt/vol) was added immediately and the reaction mixture vigorously stirred. An exothermic reaction ensued and a light brown solid settled out after a few minutes. The solid material was filtered off, washed and dried to give a light brown powder (3.5g). The reaction was repeated with 4.5ml and 6.0ml of benzoyl chloride and increased quantities of alkali. Yields in these cases were 5.8g and 6.0g, respectively, of light, yellow brown powders.

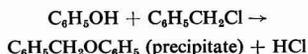
When calculated on the basis of  $C_{18}H_{14}O_6$  (mol wt = 290) for the basic wattle tannin molecule, and assuming an average of four hydroxyl groups per flavanoid unit, the three derivatives above should have had 50, 75 and 100 per cent of the available hydroxyls substituted. Carbon and hydrogen analyses showed that this assumption was reasonably accurate for the partially substituted derivatives, but it did not hold in the case where complete substitution was attempted.

Table 1  
Analysis of benzoylated derivatives of wattle extract

No. of groups substituted	Empirical formula	Calculated		Found	
		C	H	C	H
0	$C_{18}H_{14}O_6$	62.1	4.8	61.6	4.5
2	$C_{29}H_{22}O_8$	69.9	4.5	69.5	4.0
3	$C_{36}H_{26}O_9$	71.8	4.4	71.5	4.4
4	$C_{43}H_{30}O_{10}$	73.1	4.3	71.3	4.5

### Benzoylation

Reaction of benzyl chloride with phenols yields phenyl ethers with liberation of hydrochloric acid:



Dry, powdered wattle extract was dissolved in warm acetone and varied quantities of benzyl chloride added. Anhydrous

potassium carbonate and sodium iodide were added to neutralise the acid formed. The quantities of benzyl chloride were varied to give substitution of 2, 3 and 4 hydroxyl groups per tannin monomer.

After an initial reflux for 6hr, steam distillation was carried out for a further 1hr, when yellow brown products separated out. These were recovered from water.

Wattle tannins were benzylated by the modified method of Nakazawa and Miyata.<sup>8</sup>

To wattle extract (2g) was added anhydrous potassium carbonate (5g) and dimethylformamide (8ml). The mixture was warmed to assist solution of the extract. Benzyl chloride (2ml) was added and the solution refluxed for 2hr. After cooling, the solution was poured into a large volume of water and the precipitate left to harden overnight. The solid (3.3g) was filtered off and dried. By using 3 and 4ml of benzyl chloride, the tri- and tetra-substituted derivatives, respectively, were prepared. All these were analysed to assess the degrees of substitution.

Table 2  
Analysis of benzylated derivatives of wattle extract

No. of groups substituted	Empirical formula	Calculated		Found	
		C	H	C	H
0	C <sub>15</sub> H <sub>14</sub> O <sub>6</sub>	62.1	4.8	61.6	4.5
2	C <sub>25</sub> H <sub>20</sub> O <sub>6</sub>	74.0	5.6	74.6	5.8
3	C <sub>35</sub> H <sub>22</sub> O <sub>6</sub>	77.1	5.8	76.5	5.9
4	C <sub>45</sub> H <sub>24</sub> O <sub>6</sub>	79.4	5.9	77.8	5.9

### Epoxidation

When phenols are reacted with epichlorhydrin, the epoxide ring is not ruptured and an epoxy resin with a network structure results.



Caustic soda solution may be used to neutralise the liberated hydrochloric acid. Synthesis of an epoxy resin using wattle extract as the phenolic component was based on the method of Knowles and White<sup>9</sup> and involved methylation of the tannin before epoxidation.

Wattle extract (5g) was dissolved in dry methanol (25ml) and zinc dust (0.1g) and dimethyl sulfate (3.25ml) added. Aqueous potassium hydroxide solution (4.5ml, 50 per cent wt/vol) was slowly stirred in. Two more such additions of KOH and dimethyl sulfate were made at 30 min intervals. The solution was neutralised, when a precipitate formed. This was filtered off and dried.

The methylated wattle derivative was epoxidised with epichlorhydrin (4.5ml) and aqueous caustic soda solution (12ml, 10 per cent wt/vol) was added slowly and the mixture shaken. The epoxy resin separated out as a sticky mass which hardened in water. The resin was recovered and dried.

The epoxy equivalent of this resin was determined in order to ascertain the degree of substitution of the extract. Resin (50mg) was dissolved in pyridine hydrochloride (10ml) and the mixture heated for 30 min on a boiling water bath. The

excess pyridine hydrochloride was back-titrated with 0.1N NaOH solution, using phenolphthalein as an indicator. A blank determination was run concurrently. The difference between the volumes of caustic soda for these two determinations was used to calculate the epoxy equivalent, which was found to be 160-170. Lee and Neville<sup>10</sup> recommend the use of 14.5 parts of curing agent per 100 parts of resin having an epoxy equivalent in the range above. Hence, one part of curing agent was used for every 6 to 7 parts of resin.

### Preparation of surface finishes

#### From benzoylated derivatives

Benzoylated wattle derivatives were reacted with isocyanates to produce polyurethane finishes according to the method used by Drewes.<sup>11</sup> Partially benzoylated tannin was dissolved in the minimum volume of dry dioxane, and a calculated amount of poly-isocyanate added. After thorough stirring, the reaction mixture was applied to both wooden and mild steel surfaces. Application to steel was used to assess to what extent the reaction between the derivative and isocyanate was complete—any unreacted tannin would cause discoloration of the steel due to the formation of a chelation complex between the tannins and the ferric ions in steel.

The dibenzoylated wattle was reacted with twice its weight of a commercially available di-isocyanate ("Desmodur L", 75 per cent by volume in ethyl acetate, Farbenfabriken Bayer AG, Leverkusen), using an isocyanate/hydroxyl ratio of 1.1 to 1.0 (wt/vol). A range of surface finishes with varied R-NCO/OH ratios was prepared and applied to pine (*Pinus radiata*) and meranti (*Shorea* spp) panels.

Initially, the derivatives were dissolved in a solvent mixture consisting of equal volumes of butyl acetate and methyl ethyl ketone, but the flow properties of the finishes were poor and it was impossible to obtain very smooth surfaces. Mixtures of cyclohexanone and butyl acetate (1:4 by volume) or cyclohexanone: methyl ethyl ketone: acetone (1:1:1 by volume) were found to be more suitable solvents for the wattle derivatives. In some cases a small insoluble portion of the material, consisting mainly of gums, resins and sugars, remained. This could be filtered off conveniently through a coarse filter. However, by using suitable commercial wattle extracts, which have low gum contents, this additional filtration stage could be eliminated. The required weight of "Desmodur L" was added to a solution of the benzoylated extract. After thorough mixing the varnish was brushed on to wooden panels. The pot-life of the lacquer was approximately 2hr. Each panel received four coats of varnish, with light sanding between successive coatings.

A total of 52 panels were prepared with the benzoylated wattle derivatives.<sup>12</sup> Varied quantities of alkali, different wattle extracts and a range of di- and poly-isocyanates were incorporated in the resin mixtures. The ratio of isocyanate/wattle derivative was varied as well. Reference panels, prepared from several commercially available formulations, were used as controls.

The completed panels were exposed for 2 months in a metal frame which was tilted at an angle of 45° to the horizontal, on a north-facing roof site.

#### From benzylated derivatives

Benzyl derivatives of wattle extract having a low degree of substitution were found to be somewhat darker in colour

than the corresponding benzoyl derivatives. Benzyl products were, in general, less soluble in the normal solvents which were used.

Wooden test panels were coated with reaction mixtures of benzylated derivatives and "Desmodur L" as before and exposed with the benzoylated products on a roof site.

#### *From epoxy resin derivatives*

Diethylene triamine was used as curing agent for the epoxy varnishes. The resin was dissolved in the minimum volume of dry dioxane and a few drops of diethylene triamine added. After thorough mixing, the solution was applied to both wooden test panels and selected metal surfaces. To obtain a hard finish these films were heated at 60°C for 1 hr.

Mixtures of this epoxy resin were reacted, together with an equal weight of a commercial epoxy resin, using the same curing agent, to modify to some extent the properties of both resins.

## Results and discussion

*Ref. 12-15*

With the epoxy resin derivatives, excellent surface veneers were produced. The finishes were found to be hard, glossy and very resistant to abrasion, on both metal and wooden surfaces. As a direct result of the relatively high cost of commercial epoxy resins, however, the incorporation of small proportions of wattle based derivatives in such resin formulations was not considered worthwhile.

In the case of benzylated wattle derivatives, it was noticed that most of the polyurethane surface films started to crack soon after application. This crazing effect was evident also when films were applied to glass surfaces.

The only veneer based on benzyl derivatives which proved satisfactory was the one using a ratio of "Desmodur L"/wattle component of 2 : 1 and a 50 per cent substitution of the tannins. All other coatings showed dulling, bleaching and crazing of the surfaces after the 2 month exposure.

The low yields of products and laborious nature of the benzoylation of wattle tannins compared unfavourably with those of the benzoylated derivatives.

Since the ether linkage between the tannin and the benzyl part of the molecule is fairly resistant to hydrolysis, these derivatives may prove to be useful where stable intermediates are required.

The wooden test panels prepared from benzoylated wattle derivatives acquired hard, glossy finishes after the application of four coats. In general, these films were not abraded easily and resisted cracking when exposed to direct sunlight.

After exposure for 2 months, inspection of the panels revealed that those using 50 per cent and 75 per cent substituted derivatives and a ratio of "Desmodur L" to wattle component of 2 : 1 were best. Some darkening and dulling of the surfaces had occurred, but no cracks were visible in these cases. The flow properties of these surface finishes during application were better, in general, than those of other derivatives which were tested.

After exposure for a further period of 6 months, it was noted that the panels prepared from the di-benzoylated derivative gave superior results. These panels retained a higher gloss and the darkening or reddening effect was reduced as a result of the addition of a higher percentage of alkali. There was, however, evidence to suggest that the surface films prepared with less alkali in the formulation tended to weather slightly better than the others.

This series of panels showed that ratios of between 1.75 and 2.0 to 1.0 of di- or poly-isocyanate/wattle component were superior to all others prepared. These proportions represent approximately equimolar quantities of reactants. Panels with more wattle intermediate in the resin mixture tended to deteriorate faster during weathering. The weathering qualities of the superior wattle based formulations compared very favourably with commercial polyurethane finishes.

The remaining panels were inspected again after a total of 15 months' outdoor exposure. Some had been degraded completely, some showed partial breakdown, whilst a few still exhibited a satisfactory appearance.

Of the isocyanates tested, "Desmodur L" proved the best. Adhesion of surface coatings to the wooden substrates was good, although it suffered slightly in cases where higher proportions of alkali were used.

Apart from an isolated panel, all the controls, which were produced from commercial formulations showed separation of the veneer from the wood. Degradation of the cellulose fibres in the surface layers by ultra-violet light may be responsible for such separations. The generally superior adhesive properties of most of the wattle based films could be ascribed to possible ultra-violet absorption by the tannins.

Some degree of reddening was shown by all wattle based surface films. None of the reference panels exhibited this darkening, although all had yellowed to some extent. Usually, the reddening phenomenon occurred soon after initial exposure. After this very little intensification of colour was observed. The effect was least noticeable on panels coated with formulations prepared with more than the minimum amount of alkali.

Attempts were made by Drewes<sup>12</sup> to reduce the sensitivity to sunlight of the wattle based veneers. Methods included chemical modification of the basic formulation and incorporation of ultra-violet light absorbers in the coatings. It has been shown that simultaneous substitution of the 7- and 4- positions in the flavanoid unit of tannins tends to inhibit reddening.<sup>13, 14</sup> To this end, the extract was precondensed with formaldehyde, acetylated or bleached with sulfur dioxide, before benzoylation was carried out. Pre-reaction with formaldehyde was expected to block sites, responsible for reddening, on the tannin molecule by cross-linking certain units. Partial acetylation is known to substitute available 7- and 4- positions in flavanoids.

It has been shown that light of wavelengths between 300 and 400µm causes the most severe discoloration in polyurethane surface coatings.<sup>15</sup> Hence two commercial ultra-violet light stabilizers, with absorptions in the above range, were incorporated at 1 per cent levels in wattle based formulations. Panels prepared with formulations which included the above modifications were exposed as before. After 17 months' exposure these panels were taken down and inspected. Examination revealed that inclusion of ultra-violet inhibitors in wattle based surface coatings had little

effect on the reddening process. Bleaching with sulfur dioxide before benzylation produced light-coloured films initially; however, these coatings were very susceptible to subsequent reddening and were less durable.

Slight improvements in the degree of reddening were achieved by the production of mixed acetylated/benzyolated wattle derivatives. This effect was so small, however, that it hardly warranted the additional cost of acetylation.

Initial reddening of the coatings was reduced by pre-condensation of the wattle extract with formaldehyde. This was less pronounced after prolonged exposure. A distinct disadvantage of this method was the embrittlement of the surface films after formaldehyde treatment.

Hence none of the various measures, designed to counteract this reddening phenomenon, proved entirely successful. A considerable reduction in the degree of reddening was achieved, however, by a slight increase in the degree of benzyolation, so that more than 50 per cent of the reactive hydroxyl positions were substituted.

## Conclusions

Excellent wattle based polyurethane surface finishes can be produced by the use of spray dried black wattle stick bark extract as starting material. This extract contains a substantially lower proportion of gums, resins and sugars than normal fresh bark extract, thus largely obviating the necessity for subsequent removal of insoluble material either by filtration or by centrifugation. In addition, the extent of reddening of derivatives and films was limited with stick bark extract.

Since both the di- and tri-substituted wattle derivatives proved to be suitable for reaction with isocyanates, it was decided to use a benzyolated derivative which had been substituted in an average of 2.5 of the four possible phenolic hydroxyl positions per  $C_{16}$  unit of the tannin molecule. This degree of substitution still allows sufficient sites for subsequent polymerisation with isocyanates.

The final preparation of a suitable benzyolated wattle derivative was as follows: spray dried wattle stick bark extract (10g) was dissolved in water (100ml). To this solution was added benzoyl chloride (12.5ml), followed immediately by potassium hydroxide solution (24ml, 20 per cent wt/vol). The flask was shaken vigorously for 40 minutes until the exothermic reaction had subsided. The pale brown precipitate (16g) was filtered off, washed and dried.

The amount of alkali used in the formulation was found to be a significant factor in the performance of these finishes. The minimum amount of potassium hydroxide to substitute an average of 2.5 of the hydroxyl groups should be used.

Although "Desmodur L" (Bayer) was used extensively in these investigations—as a result of its slightly superior performance—other commercial di-isocyanates are almost equally suitable in this application.

For softwoods, a ratio of "Desmodur L"/wattle component of 1.75 to 2.0 : 1.0 proved most suitable. Small amounts of benzoic acid, which formed during the Schotten-Baumann

benzyolation, may be ignored. In the case of hardwood species, these main conclusions apply, except that the isocyanate content could be reduced to a ratio of 1.5 : 1.0, without serious deleterious effects in the varnish.

Recent studies have shown that solvent mixtures of cyclohexanol, methyl ethyl ketone, acetone, ethyl acetate and turpentine in ratios of 3 : 3 : 2 : 1 : 2 or 7 : 7 : 5 : 1 : 4 were far superior to the earlier solvents, both in ease of application and stability.

The two main components were mixed together shortly before use. Three coatings, with light sanding between coats, were usually applied.

Results of the outdoor exposure trials which lasted for a total of 2 years 3 months, showed conclusively that wattle extract may be used successfully as a raw material for the production of extremely durable and protective surface coatings for wood.

Although benzyolated derivatives impart slight reddening effects to polyurethane finishes, such effects occurred within the first few days of exposure. A bleaching process followed this initial discoloration and after the full period of exposure, the overall colour was a pleasant light brown. Surface cracking was not evident on the better panels, which still retained a certain amount of gloss after this extended period. The surfaces were hard, scratch-resistant and unaffected by organic solvents such as alcohols, acetone, benzene and chloroform.

These high-gloss, wattle based polyurethane surface veneers may find wide application in the finishing of furniture, wooden boats and exposed timber surfaces, such as laminated beams and outdoor cladding materials.

Stable wattle derivatives, having suitable solubility characteristics, could become useful substitute raw materials for expensive resins in the manufacture of paints and lacquers.

An investigation of the usefulness of low-cost wattle derivatives in the production of polyurethane and epoxy resin surface veneers, and possibly as extenders in paints and resins, should be worthwhile.

## Acknowledgments

The author wishes to thank Dr S. E. Drewes for the preparation and the long-term exposure trials of the series of varnished panels.

This work is supported by annual grants of the African Territories Wattle Industry Fund and the South African Council for Scientific and Industrial Research to the Leather Industries Research Institute.

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## Next month's issue

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

**Efflorescence on gypsum glass board** by *G. A. King, M. J. Ridge and G. S. Walker*

**Ion exchange behaviour of some anti-corrosive paints and paint media** by *W. U. Malik and L. Aggarwal*

**Oxidation of copper phthalocyanine blue pigment with potassium dichromate in acid solution** by *A. M. Islam, A. M. Naser, A. A. El-Mariah and A. A. Salman*

# Reviews

## **Pigment Handbook Vols I-III**

**Edited by T. C. Patton**

**John Wiley & Sons, New York, London, Sydney, Toronto, 1973. Pp. xxviii + 985; viii + 455; viii + 538. Price £65.00**

What a pity that this three-volume work is so expensive, because here is a handbook that every pigment technologist would like to have on his bookshelf. The need for the comprehensive approach that has been adopted has long been felt, but not previously attempted on the present lines. Indeed, even the concept of pigment technology as a distinct discipline in its own right has not always met a ready response in the UK as it has in other pigment-producing countries.

One hundred and thirty-five authorities have contributed to this work, which considers in turn: the properties and economics (Vol. I), applications and markets (Vol. II), and characterisation and physical relationships (Vol. III) of pigments. The reviewer finds this breakdown of the general field highly satisfactory; the editor is to be congratulated on the arrangement. An index to the whole work appears at the end of Vol. III.

Vol. I, the thickest one, provides information on the nature of individual pigments, their physical and chemical properties, relevant economic data, historical background, major reasons for use, method of manufacture, grades, specifications and manufacturers. A specific format has been followed by all authors contributing to Vol. I to ensure consistency in presentation.

Vol. II brings out the full scope of the applications of pigment technology, covering the pigmentation of paints, inks, paper goods, elastomers, plastics, cosmetics, magnetic tapes, foodstuffs, textiles, adhesives, ceramics, photochemical coatings, concrete, mortar etc.

Vol. III covers such matters as pigment particle size, surface characteristics of pigments, pigment/binder geometry, pigment optics, pigment dispersion, rheology, pigment electrochemistry, and colour aesthetics, from the viewpoint of pigments generally.

One or two English authors have contributed and there are ample literature references to English work, but one has to accept that the book is American. Thus, when one examines lists of manufacturers, these all turn out to be North American ones or if one examines specification references, they are found to be mainly ASTM, US Federal, FDA etc. as though ISO standards did not exist. And there are other criticisms that one might make, for example, the glossary of

definitions of common terms associated with pigment technology given in Appendix IV of Vol. III is rather limited. There is, however, so much good stuff in this book and one does not want to carp. Nevertheless, that a second edition were a more closely international pigment handbook would be a "consummation . . . to be wished"—that, and some easing of the price!

R. J. COLE

## **Functional Monomers, Vol. 1**

### **Their preparation, polymerisation and application**

**Edited by R. H. Yocum and E. B. Nyquist**

**Marcel Dekker, New York. August 1973. Pp. xii + 715. Price \$39.5**

A functional monomer is defined here as a monomer that maintains a reactive site after the monomer itself has been polymerised.

Functional monomers play an important role in the polymer industry, such as upgrading commodity type polymers (for example in the production of thermosetting coatings) as well as preparing speciality polymers such as polyacrylamide.

The volume deals with the following monomers:

1. Acrylamide and other alpha, beta unsaturated amides.
2. Reactive halogenated monomers.
3. Hydroxy monomers.
4. Sulfonic acid and sulfonate monomers.

The purpose of this book is to provide the chemist and technologist with a practical reference book dealing with the functional monomers. The authors, each dealing with a different functionality, attempt to present a broad background on a technical level with regard to the preparation, polymerisation and application of functional monomers in this particular area.

These topics have been dealt with in a very straightforward and readable manner, and the sections on applications are most interesting and useful.

This book would make a useful addition to any library.

N. PLUMMER

# Information Received

## Change of address

The Liverpool office of Morris Ashby Ltd. is now at 5 Castle Street, Liverpool L2 4SW.

## Desert Sunshine

Desert Sunshine Exposure Tests with exposure facilities in the Sonora Desert, Phoenix, Arizona, has been acquired by J. S. Robbins and G. A. Zerlaut and incorporated as a wholly independent testing laboratory.

## Rubber House instals Vacu-Blast equipment

Rubber House Limited, the major distribution company for the Allied Polymers Group, has set up a surface finishing facility in East London and installed a "Blastmaster" and "Hornermaster" manufactured by Vacu-Blast Limited of Slough.

## New products

### Black Seelfos

Jenolite has added "Black Seelfos" to its range of Seelfos phosphating lacquers. The new product is intended to perform a decorative function as well as to protect against corrosion.

### Filling machine for inflammable liquids

The latest adaptation of the Albro "Auto-line" is a flame-proofed four-head filling unit designed to handle aromatic coal-tar derivatives and thinners. The machine, which has an Albro vacuum-operated liquid-filling system specially modified to handle these products, has been built to the order of customers in Finland.

## Section Proceedings

### Hull

#### A review of the current and future trends in industrial finishing

A meeting was held at the Dorchester Hotel, Hull on Monday 3 December with Mr F. D. Robinson in the chair. Mr Robinson introduced Mr R. McD. Barrett, of BIP Chemicals Limited, who gave a lecture with the above title.

In the first part of his lecture, Mr Barrett presented an interesting collection of statistical data showing the present market with respect to paint type and end use. It was seen, for example, that alkyd amino coatings held 26 per cent of the market, acrylics 9.5 water based coatings 3 and polyurethanes 1 per cent. By far the largest outlet for industrial finishes lay in the car industry, with between 20 and 30 per cent and the overall annual expansion was about 1.5 to 2 per cent.

Mr Barrett considered in some detail the changes which had taken place in the painting of domestic appliances over recent years. This was a progressive industry which presently accounted for 5 per cent of industrial paint sales, though this was now declining because of more efficient application methods leading to savings in paint, the substitution as appropriate of self-coloured plastics and the use of pre-painted coil and strip. Whereas the use of alkyd amino coatings had declined in the years since 1965, there had been a tremendous upsurge in the use of acrylics. Epoxies also

## Bayer-organica

Bayer AG, Leverkusen, is now offering its organic intermediates under its new name of "Bayer-organica". There are approximately 800 products in the range.

## High colour furnace blacks from Cabot

Cabot Carbon Limited has introduced two high-colour, furnace process carbon blacks as replacements for channel process blacks to the most demanding pigment applications in plastics, paints and coatings. The new blacks are "Monarch 900" and "Monarch 1100" (fluffy form). Their equivalent pelleted versions are "Black Pearls 900" and "Black Pearls 1100."

## PAR dispersions

The New England Resins & Pigments Corporation, Wakefield, Mass., has introduced a range of pigment dispersions in water, DOP, and various other vehicles. These products, marketed under the trade name "PAR," can be used to colour a wide range of polymer systems. Technical information and laboratory samples are available.

## Courses and symposia

### Purchasing and supply

The IRI Commercial Group is organising a two-day conference on "Purchasing and Supply in a production orientated industry" to be held on 17-18 April 1974 at the St. Ermin's Hotel, London. The conference is intended to be a major event for the rubber industry during 1974. Prospective delegates should contact the IRI at 4 Kensington Palace Gardens, London W8 4QR.

## Heterophase and blended polymers

The Polymer Properties Group Committee of the Plastics Institute is calling for papers for its research meeting on "Structure and properties of heterophase and blended polymers" to be held at the University of Bristol in April 1975.

## The life of structural steel work

A one-day Symposium to be held on 8 April 1974 and with the theme "The life of structural steelwork" is being organised by B. Marshall, Department of Chemistry and Materials Science, Derby College of Art and Technology, Kedleston Road, Derby.

## US courses in colour technology

A summer programme of three intensive courses in colour technology is being offered for the 9th consecutive year by the Rensselaer Polytechnic Institute, New York. The courses offered are: colouring of plastics (2-4 April), principles of colour technology (8-12 July), colour technology of management (18-19 July), and advances in colour technology (22-26 July).

## Literature

### Synthetic Resins catalogue

A comprehensive manual of synthetic resin products for paint and ink formulations and surface coating treatments has been published by Synthetic Resins Limited of Speke, Liverpool.

The catalogue contains full data on the very broad range of non-drying, pure drying oil modified and thixotropic alkyd thermosetting acrylics, phenolic and epoxy resins available from SRL.

had made ground because of their superior durability in adverse conditions, for example as coatings on deep freezers stored in garages.

In the second part of his lecture, Mr Barrett identified the availability of raw materials, and considered future legislation with respect to safety and pollution as the two main influences on future trends within the industry. He thought that non-aqueous dispersions would occupy an important position for an interim period. However, these materials, generally, were formulated with significant proportions of aromatic hydrocarbon solvents and he thought that the future would lie with either water- or powder-based coatings. The former had received a large impetus with the advent of electrodeposition but had not made the headway which had been expected, possibly because of the high cost of plant. Similarly, there existed only a qualified enthusiasm for powder coatings—they have so far made only a limited impact on the UK paint market—and Mr Barrett was not prepared to predict which of the two types would eventually assume the greater importance.

The lecture was followed by an interesting discussion period, which was finally closed with a vote of thanks to the lecturer from Mr J. R. Tinegate. The meeting was attended by 16 Members and six visitors.

J.A.H.

## Newcastle

### Acrylic emulsion in maintenance paints

A meeting was held on Thursday 1 November at the Turks Head Hotel, Newcastle upon Tyne. Dr R. N. Washburn, of Rohm & Haas Ltd., presented a paper on acrylic emulsions in maintenance paints.

Dr Washburn discussed the main advantages of aqueous acrylic systems, notably fast drying, easy clean up, low toxicity, good durability as well as good spraying properties. Against these were the need for multi-coat systems, flash rusting on metallic surfaces and relatively poor brushing properties.

The aqueous nature of the acrylic systems had resulted in scepticism by some on the ability of these coatings to protect metallic surfaces. This was reinforced by the flash rusting, which was thought to be caused by the presence of iron salts on the surfaces of the metal when coated, which caused unsightly rust spots but did not detract from the film properties and, finally, by indifferent results sometimes obtained by accelerated test methods, for example Kesternich.

Dr Washburn's paper concentrated on test results obtained on a wide range of substrates and surface conditions after up to five years exposure. Multi-coat acrylic emulsions were compared to epoxies, alkyds, chlorinated rubbers, vinyls and polyurethanes. Very good results, with and without anti-corrosive pigment, had been obtained on all substrates with the best acrylic systems. The need for correct polymer selection had been shown by the premature failure of unsatisfactory acrylics.

It was clear from the results that the acrylics compared very well with traditional high durability coatings and would continue to play an increasing role in the preparation of maintenance paints.

Dr Washburn's talk was well illustrated with colour slides and a number of interesting questions were raised. The meeting closed with a vote of thanks by Mr C. N. Finlay.

J.B.

### Determination of the dispersibility of organic pigments in paints

The third meeting of the session was held on 6 December 1973, when Herr Haffner, of Hoechst—Cassella Dyestuffs, gave a lecture on the determination of the dispersibility of organic pigments in paints.

The lecture was concerned with the determination of the most efficient mill-base for two specific organic pigments—a carbazole violet and a quinacridone violet. A sand-mill was

used throughout the series of experiments to disperse the pigments.

A certain number of variables were covered; these included pigment concentration, binder concentration, temperature of mill, type of pigment, type of binder and viscosity of mill-base.

It was found that increase of either pigment in the mill-base through the range 6-20 per cent gave an extremely large increase in thixotropy, particularly moving from 8 to 13.5 per cent. At 13.5 per cent both pigments gave a highly thixotropic mill-base.

Increase of binder (non-drying alkyd) through the range 25 to 45 per cent gave no increase in thixotropy.

Increase in temperature of mill-base gave large an increase in thixotropy.

Variation of the type of binder (non-drying alkyd, dco alkyd and non-drying alkyd-mf resin mixture) gave great differences in tinctorial strength. Improvement in tinctorial strength was directly related to the increase in thixotropy.

Herr Haffner suggested one lesson to be learnt from the results was that it was unwise to categorise organic pigments as "easily dispersible" or "difficult to disperse," as so many varying factors affected the ease of dispersion.

Herr Haffner then answered a considerable number of questions in an expert manner, particularly as English was not his natural tongue.

Thirty-four members and guests were present.

It is hoped to publish the paper in full in due course.

J.B.

### Student Group

The first meeting of the 1973-74 Session was held on Friday 14 December in the library of Berger Chemicals Ltd. when 26 members and visitors assembled to hear Dr A. F. Everard talk on "Applications of synthetic resins". Using slides and samples, Dr Everard covered many applications including surface coatings, printing inks, moulding compounds and laminating. With refreshments served during the course of the afternoon, this new style of meeting was considered a success by both students and organisers, following disappointing support in recent years.

T.H.

## Scottish

### Colour difference—the measure of the future

This was a joint meeting with the Society of Dyers and Colourists on 20 November 1973 when Mr K. McLaren (ICI) presented a full account of the development of the equations, techniques and equipment for colour difference measurement. Mr McLaren stressed that he was advocating the use of "colour difference" not of colour measurement *per se*. With the development of inexpensive computerised systems, quality control in dye-houses and paint shops could be vastly improved and standardised. In his vote of thanks Mr E. Burns, the Section Chairman, thanked Mr McLaren for his interesting talk. He also thanked the SDC for their invitation to the meeting and hoped that the joint meetings would continue.

### Review of plastics

Professor N. Grassie (University of Glasgow) presented a very entertaining lecture on the history, use and future of plastics to a meeting on 15 December. He began by tracing the history of plastics from the nitrocellulose-camphor mixture of Parks in 1861 through casein (c. 1900), phenol-formaldehyde i.e. Bakelite (1914) and cellulose acetate (1917) to the big upsurge of plastics after 1930.

The reason for this upsurge could be traced to general improvement in affluence, the advent of the motor car and Germany's desire for independence in strategically important materials. The technical factors involved were the application of free radical polymerisation to vinyl monomers and Her-

man Staudinger's elucidation of the reality of high molecular weight polymer chains.

Coal had been the initial raw material for vinyl polymers but this has now been replaced by oil. The continuing growth in the use of plastics in the post-war years had, over the last year (before the present oil crisis), placed considerable pressure on raw material supply. Although this situation could worsen with the present oil situation, Professor Grassie felt the important question was whether the long term (more than 100 years) supply of fossil materials as raw material for plastics could be maintained. The energy crisis could be solved, within the foreseeable future, with the advent of nuclear fission or fusion reactors. It was unlikely, however, that even this supply of energy could supply materials with carbon bonds (eg from atmospheric carbon dioxide), at a cost commensurate with their wide usage.

Although new plastics, such as polyimides and other heterocyclic polymers were being developed as high-grade

materials, the speaker considered that existing monomers as homo- and co-polymers would provide the plastics of the future. He felt that in both structural and fibre plastics the important development would result from new treatments and engineering techniques.

During a stimulating discussion period, Professor Grassie answered questions on various aspects of plastics use. He considered that toxicity was covered by suitable regulations but would always be under critical examination. Plastics degradable by ultra-violet light were now technically possible but he did not foresee a major use of such products. The burning of plastic waste would produce toxic gases (eg monomers and hydrogen chloride) and, therefore, plastic re-use would be a more satisfactory control on pollution. Following a discussion on carbon fibre, Professor Grassie concluded that composites from this material would fulfil their early promise as a structural material.

G.H.R.

## South African

### Transvaal Branch

#### Properties and functions of dry films

A meeting was held on Thursday 29 November 1973 when Mr R. J. Kinsman gave a lecture on paint and printing ink films. The lecturer first discussed relative functions of dry paint and printing ink films. He suggested that whereas protection was probably the most significant function of paint films, colour and image sharpness of printing ink films was a far more important function than the protective qualities of the film.

The lecturer went on to discuss, for the benefit of the majority of paint makers in the audience, the various printing processes and the thickness of films involved in printing. It was apparent that in order to achieve a particular colour with the films of two microns or less involved in offset and flexographic printing, much higher colour concentrations in the printing ink would be necessary than would be the case with a paint film of some 50 to 100 microns film thickness. Letterpress films were 4 or 5 microns thick, gravure films varied in thickness from 5 to 50 microns and silk screen films commonly varied between 25 and 75 microns and were much more comparable with paint films.

Properties of dry film of either printing inks or paint might require specific resistance properties for a particular application; for example a controlled coefficient of friction might be required either for floor paints, ship-deck paint or in certain packaging applications where it was undesirable that packs should slide excessively over one another whilst being filled. Adhesion of printing inks and paints to a wide variety of substrates was a mutual problem involving common technology and choice of film formers and solvents. Paints might be required to resist hot detergents as used in domestic or commercial washing machines and so also might the packs of the detergents themselves, because it would be undesirable that the pack of a detergent should lose its consumer appeal as a result of being handled by hot detergent covered hands. However, the exposure of a detergent pack was merely one or two washes, whereas the exposure of the paint film would be many years; thus the durability requirements were a matter of degree.

Some of the detailed rheological requirements involved in the printing process were then discussed. These mostly involved transfer between rollers, and they were contrasted with the requirements for dipping, spraying and brushing applications of paint.

The particle size of pigments used in printing inks which were to be distributed in thin film was obviously of para-

mount importance. With a film thickness of less than 4 microns, it was apparent that particles approaching this order of magnitude would be objectionable. This was the explanation for using smaller particles of bronze powders in printing ink applications than in paint applications, because larger particles would not transfer satisfactorily from one roller surface to the other. Similarly, extender pigments for printing inks were normally less than 1 micron in diameter in contrast with the larger particles used in paint extenders.

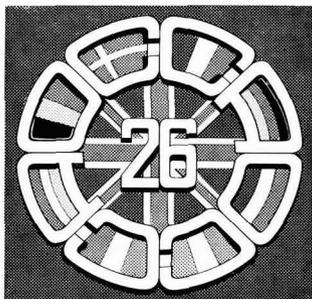
The lecturer displayed samples of typical printing inks and discussed the manufacturing processes for printing inks compared with those processes used for paint tinters as well as actual ready-to-use paints. It was apparent that similar plant could be used for both industries and the techniques involved in dispersion had very much in common. Because of the fine particle size requirements of printing inks, with most particles in the system substantially below one micron in size, the use of coated, easily dispersed pigments was particularly important to this industry.

Drying methods for the films of printing inks and paints had very much in common. The most substantial difference in drying technology was due to the fact that printing inks were commonly used on absorbent substrates such as paper and carton board, and in these cases use was frequently made of absorption of solvent and other thin liquid components of the printing ink into the substrates in order to achieve an initial set of the ink. It was particularly important that printing inks should have rapid setting in order that subsequent printed sheets may rest on top of sheets printed only seconds before or when the printed work was re-reeled, that the printed side of the sheet shall not set-off on the reverse side of a subsequent layer of the reel. Setting in printing inks was thus vaguely equivalent to touch-dry painting.

Mr Kinsman went on to discuss the various ingredients which might be used in paints and printing inks. He indicated the cross fertilisation of ideas between the two industries that might be of value in the formulation, for example, of nitrocellulose and acrylic finishes and also the considerable common technology involved in systems drying by oxidation and polymerisation. Recently developed methods of drying, such as microwave, electron beam and ultra-violet curing systems, were mentioned and the difficulties of focusing sufficiently high concentrations of radiation in the first two systems in thin printing ink films for the required speed of printing was noted. Practical ultra-violet curing systems had been developed by both industries.

P.A.J.G.

*Continued on page 126*



## Shortages of raw materials enhance the value of this unique forum for technical display and discussion

### OCCA 26 Exhibition

23-26 April 1974, Empire Hall, Olympia, London

Japanese delegation of paint manufacturers to visit OCCA 26

Never has the value of the Association's annual Technical Exhibition been so apparent as during the present shortage of raw materials for the surface coatings industries, since the Exhibition provides a unique forum for technical discussion between the personnel using these resources and the suppliers. Grasping this opportunity to explain to the technical personnel in the industries the optimum use of those resources available, many companies will be using this splendid platform to show how they are helping their clients during these critical times, and the Exhibition Committee is pleased to report that the countries represented at this year's Exhibition are widespread and include, for the first time, three more representatives from Eastern Europe. The full list of countries represented will be as follows: Australia, East Germany, Finland, France, Holland, Hungary, Italy, Poland, Rumania, Spain, Switzerland, USA and West Germany.

Applications for stand space can still be accepted, and any company wishing to exhibit should contact immediately the Director & Secretary of the Association at the address shown on the front cover of this *Journal* (or telephone 01-908 1086—telex 922670.)

#### International character

Arrangements have been made for a delegation of Japanese paint manufacturers to visit OCCA 26. They will also tour the

Paint Research Station at Teddington, Middlesex, and visit the SPL Group, Batley, Yorkshire, where the President, Mr L. H. Silver, will act as host.

The party will want to discuss licensing arrangements for the manufacture of specialised products in Japan and anyone wishing to approach them should contact, in the first instance, the Director and Secretary, R. H. Hamblin, at the Association's offices.

The Exhibition, which has long been known as the forum for technical display and discussion for the surface coatings industries, in 1973 attracted visitors from more than 50 overseas countries, and there was direct contribution by exhibitors from 14 overseas countries. The motif chosen for this year's Exhibition shows the flags of the enlarged European Economic Community and, by converging on the flag of the UK, symbolises the welcome extended for many years to exhibitors and visitors not only from these countries but from farther afield to the OCCA Exhibitions in London, one of the capital cities of the European Economic Community. To further the aim of a truly international character, the Exhibition is widely advertised in technical journals both at home and in many technical journals overseas. Furthermore, it has been the practice for many years to issue information cards in six languages (English, French, German,

Italian, Spanish and Russian) and these are widely distributed to firms and individuals in many countries. Interpreters are available at the Exhibition without charge to help both exhibitors and visitors alike.

#### Exhibition Dinner

Following the success of the Exhibition Dinner on the opening day in 1973, the Committee has decided to hold a Dinner at the Savoy Hotel, London, WC2 on Tuesday 23 April at 19.00 for 19.30hr. At the request of some exhibitors, a cash bar will be made available after the function for those visitors wishing to use this facility. Applications for tickets are now being accepted at the Association's offices.

#### Official Guide

As far as conditions permit, the *Official Guide* is prepared well in advance of the dates of the Exhibition so that copies can be circulated widely, both to members and non-members, thus allowing visitors an opportunity to plan their itineraries. As well as maintaining the Exhibition's unique position as a purely technical display, manned by technical personnel, the Exhibition Committee has for many years followed the successful policy of encouraging visitors in this way, and by making no charge for admission so that the maximum flow of communication between exhibitors and visitors can take place.

## 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, Potts 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 St Nicholas Hotel, Scarborough, where the Association's Conference was held in 1967. 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.

## Jordan Award

This award was instituted by the late Mrs M. R. Jordan in memory of her husband Dr L. A. Jordan, who was President of the Association 1947-49 and an Honorary Member, and who died in December 1964. The Committee invites applications for the third award of £100.

The rules of the Award are:

1. The Award will be made for the best contribution to the science or technology of surface coating by a Member of any nationality working in either the academic or industrial field who is under the age of 35 at the date of application.

2. The final date for submission of applications will on this occasion be 27 December 1974 and it is hoped to present the award at the Scarborough Conference in the following June.

3. The selection of the recipient of the Award will be made by a Committee

under the chairmanship of the Association's Hon. Research and Development Officer.

4. There will be two methods of application. First, by the submission of a paper describing original work by the candidate which is offered for publication in the Journal or has been so published during application. The alternative method will be by recommendation by a superior for work which for reasons of commercial secrecy cannot be published; in this case the candidate will be expected to submit a dissertation on a topic relating to his work and demonstrating his superior knowledge of the principles thereof. The Award is for individual merit and clear evidence of the candidate's own contribution will be required if a paper is offered under joint authorship.

5. Applications should be addressed to the Director & Secretary at the Association's offices.

## Association Dinner Dance 1974

Early in the New Year members attached to the United Kingdom, Irish and General Overseas Sections were sent the application form for the Association's Biennial Dinner and Dance to be held on Friday 31 May 1974 at the Savoy Hotel, London WC2R 0EU.

The Reception will take place at 7.00 p.m. for Dinner at 7.30 p.m. 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 a.m.

The price of single tickets is £7.00 plus 70p VAT each and applications should be made by Members as soon as possible after receipt of the form. 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.

## Exhibition Dinner

Forms can be obtained from the Association's offices for the Exhibition Dinner to be held at the Savoy Hotel, London WC2, on Tuesday 23 April 1974.

## News of Members

Mr F. Hellens, an Ordinary Member attached to the Newcastle Section, has left Universal Bedding & Upholstery Ltd. to take up an appointment with Thomas Swann & Co.

Mr R. G. Stichbury, an Ordinary Member attached to the London Section, was appointed Joint Managing Director of Wymouth Lehr & Fatoils Limited at the beginning of the year. The company is a member of the British Tar Products group. Mr Stichbury was previously Sales Director of both the Chemical Supply Co. Ltd. and Rex Campbell & Co. Limited in the Chemicals Division of Blagden & Noakes Holdings Limited.

# Forthcoming Events

Details are given of meetings in the United Kingdom up to the end of the month following publication, and South Africa and the Commonwealth up to the end of the second month.

## March

### Monday 4 March

*Hull Section:* "Etch and blast primers" by H. F. Clay of Cromford Colour Co. Ltd., to be held at the Dorchester Hotel, Beverley Road, Hull at 7.00 a.m.

### Thursday 7 March

*Newcastle Section:* "Coatings for plastics" by Dr Kut, E. Wood Ltd., to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne.

*Thames Valley—Student Group:* Works visit.

### Friday 8 March

*Bristol Section:* Annual Dinner Dance at the Mayfair Suite, New Bristol Centre.

*Manchester Section:* "Recent trends in wood finishing" by Mr K. Waddington of Donald Macpherson & Co. Ltd. to be held at the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1 at 6.30 p.m.

### Tuesday 12 March

*London Section:* "Additives in paint." Half-day symposium at the Polytechnic of the South Bank, Borough Road, SE1, commencing at 2.00 p.m.

*West Riding Section:* "The forensic examination of inks and paints" by Mr R. M. Kevern to be held at the Griffin Hotel, Leeds at 7.30 p.m.

### Thursday 14 March

*Midlands—Trent Valley Branch:* "Marketing" by Mr J. E. Fowles-Smith, Mebon Ltd., Sutton-in-Ashfield, Notts to be held at British Rail School of Transport, London Road, Derby at 6.30 p.m.

*Scottish Section:* "Paint and joinery" by A. Sherwood (Paint Research Association) to be held in the St Enoch Hotel, Glasgow at 6.00 p.m.

### Friday 15 March

*Irish Section:* "Dispersible pigments for printing inks" by Mr H. Bower BSc, Ciba Geigy (UK) Ltd. to be held at the Clarence Hotel, Dublin at 8.00 p.m.

### Friday 15 March

*Midlands Section:* Annual J. Newton Friend Lecture 7.30 p.m. "Wines" by D. J. Smith (Seligman & Co. Ltd.) to be held in the Birmingham Chamber of Commerce and Industry, PO Box 30, 75 Harborne Road, Birmingham B15 3DH.

### Saturday 16 March

*Scottish—Student Group:* "Pigment elaborations" by Mr A. G. Able of Hoechst Cassella to be held at St Enoch's Hotel, Glasgow at 10.15 a.m.

### Tuesday 18 March

*London—Southern Branch:* Meeting cancelled.

## Wednesday 20 March

*Scottish—Eastern Branch:* AGM at 7.00 p.m. followed by a lecture on coloured television by Mr L. Ashton, BBC, to be held in the Carlton Hotel, North Bridge, Edinburgh.

*Midlands Section:* Day Symposium 10.00 a.m. at Pavilion Suite, County Ground, Edgbaston, Birmingham "Atmospheric pollution—will the paint user overcome this problem."

## Thursday 21 March

*Thames Valley Section:* "Dispersymer two year's production experience" by Mr M. Waghorn of ICI to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks at 7.00 p.m.

## Friday 29 March

*Bristol Section:* "The new flashpoint regulations—flashpoint and combustibility tests" by Mr A. N. McKelvie of the Paint Research Association, to be held at the Angel Hotel, Cardiff.

## April

### Thursday 4 April

*Midlands—Trent Valley Branch:* Annual General Meeting followed by a lecture on "Colour television" by a speaker from Pye Television Ltd., Cambridge, to be held at British Rail School of Transport, London Road, Derby at 6.30 p.m.

*Newcastle Section:* Annual General Meeting to be held at the Royal Turks Head Hotel, Grey Street, Newcastle upon Tyne.

*Thames Valley Section:* Annual General Meeting to be followed by a talk on "Windsor Castle" by Mrs G. Grove.

### Friday 5 April

*Manchester Section:* Annual General Meeting, at the University of Manchester Institute of Science & Technology, Sackville Street, Manchester, at 6.30 p.m.

### Tuesday 9 April

*West Riding Section:* Annual General Meeting to be held at the Griffin Hotel, Leeds at 7.30 p.m.

### Wednesday 17 April

*Scottish-Eastern Branch:* "North sea oil" speaker from the Scottish Council of development and industry.

### Friday 19 April

*Irish Section:* Annual General Meeting to be held at the Clarence Hotel, Dublin, at 8.00 p.m.

*Midlands Section:* Annual General Meeting to be held at 6.30 p.m. in the Birmingham Chamber of Commerce and Industry, PO Box 30, 75 Harborne Road, Birmingham B15 3DH.

### Tuesday 23 to Friday 26 April

Annual Technical Exhibition, Olympia, London.

### Friday 26 April

*Bristol Section:* Annual General Meeting to be held at the Royal Hotel, Bristol, at 7.15 p.m.

## Scottish Section

### Wine and Cheese Party

The Scottish Section was joined by the Student Section and Eastern Branch at a very enjoyable Wine and Cheese Party in the Saints and Sinners Lounge in Glasgow on 6 December. A wine tasting session was followed by a lively dance session. It was the unanimous feeling of those attending that Mr MacLean and his social committee should be congratulated on the high standard and diversity of their social functions.

### Annual Dinner and Dance

The Annual Dinner and Dance took place in the St Andrew's Suite of the Albany Hotel, Glasgow on the 11 January 1974.

The Gaelic menu (with Scottish and

French translations) and haggis, piped in with a traditional ceremony, provided a distinctly Scottish flavour. Although the energy crisis depleted the numbers at the top table, the quality of the speeches showed no lack of power.

Mr E. Burns, Chairman of the Scottish Section, gave the Selkirk grace and the address to an impressive steaming "chieftain of the pudding race," which looked to be in good company with Mr Burns's undisclosed amber liquid. Mr Burns welcomed the President and Mrs Silver, several Section Chairmen and their wives, and proposed the toast on behalf of all members to their guests. In his address, Mr Silver thanked Mr Burns for the invitations and proceeded to relate, in the best of after-dinner fashion, some of his previous experiences at Scottish Section dances. There were, however, some people present who questioned his prediction of a

Leeds victory over Celtic in next year's European Cup. The St Mirren contingent doubted if even McQueen's presence could make such a change in the Yorkshire club!

Mr Burns also used the occasion to present Mr J. McVey with a suitcase from his friends within the Scottish Section. Mr McVey is a past Chairman of the Scottish Section and after 31 years with Tioxide International (BTP) is well known within the paint trade. All present wished him a long and happy retirement.

Following the excellent meal, the company took the floor for a most enjoyable dance session interspersed with novelty and spot prizes. At the end of the evening everyone joined with Mr Burns in expressing their appreciation to Mr McLean and his social committee for their selection of the menu and general organisation of a first-class evening at this new venue.

## 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

- BARBER, JOHN WILLIAM, 10 Meldaun Court, Sunbury, Victoria, Australia 3429. (*General Overseas*)
- CHAMBERS, JOHN MALCOM, BA, 30 Ardmeen Park, Black Rock, Co. Dublin. (*Irish*)
- CUNNINGHAM, ALAN, BSc, ARIC, 12 First Street, Pont Bungalows, Leadgate, Consett, Co. Durham. (*Newcastle*)
- HACKNEY, THOMAS, NZCS, 12 Tenterdon Avenue, Balmoral, New Zealand. (*Auckland*)
- HOMER, RAYMOND INGLEDIEFELD, LRIC, 285 Davenport Road, Glenwood, Durban, South Africa. (*South Africa*)
- KASSAS, MOUHAMAD TAYEZ, BSc, Osama eben al Monkez, Jeser-Salihieh, Damascus, Syria. (*General Overseas*)
- MCGREER, JAMES FREDERICK, BSc, 10 Bushy Park Road, Terenure, Dublin 6. (*Irish*)
- MOORE, PETER ENGLISH, BSc, AMBIM, 40 Edgecombe Drive, Darlington, Co. Durham DL3 9DQ. (*Newcastle*)

- MOUALLEM, MOUHAMAD, BSc, Halbonney-Phalestein, Str. 39, Damascus, Syria. (*General Overseas*)
- SWAILES, GEOFFREY NORMAN, LRIC, API, 30 Botany Road, Howick, New Zealand. (*Auckland*)
- TURNER, PAUL HERBERT, BTech, 7 Gloucester Close, Tytherington, Macclesfield. (*Manchester*)
- VAN PROOIJEN, WILLIAM, Keuser & Mackay, 114 Boulevard de Magenta, 75010, Paris, France. (*General Overseas*)

### Associate Members

- BRINCK, ROBIN, 6 Oakleigh Lodge, Oakleigh Drive, Berea, South Africa. (*South Africa*)
- SAVAS, ANDREW, 65 Pireos Street, Athens TT, 113, Greece. (*General Overseas*)

### Registered Students

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## Section Proceedings

Continued from page 123

### West Riding

#### Water based metal primers

A meeting was held on Tuesday 13 November 1973, at the Griffin Hotel, Leeds. Mr K. A. Safe, of Vinyl Products Limited, presented a lecture on the subject of water based metal primers.

A preliminary screening of a wide variety of anticorrosive pigments had been undertaken using a vinyl acetate homopolymer as binder. This had indicated that zinc chromate was worthy of further examination. Further work had confirmed that zinc chromate gave very good salt spray resistance but poor results on exterior exposure.

It was decided that a homopolymer emulsion was not really suitable and other types were screened. A styrene/acrylate emulsion had looked very promising and further work confirmed this initial conclusion. Several anticorrosive pigments had been examined using this binder and zinc chromate still gave the best performance.

Further tests had been conducted using either zinc phosphate or zinc chromate at 10 per cent on binder weight on

various substrates. It was found that a top coat of unpigmented latex gave a big improvement in protection and that zinc phosphate was less effective than the chromate.

A mixture of zinc chromate and red oxide had been examined using varying pigment volume concentrations (PVC) between 0 and 50 per cent. The sample without the pigments stood up quite well and those with between 5 and 20 per cent PVC gave about the right level of anticorrosive behaviour. Samples with 50 per cent PVC gave very poor results.

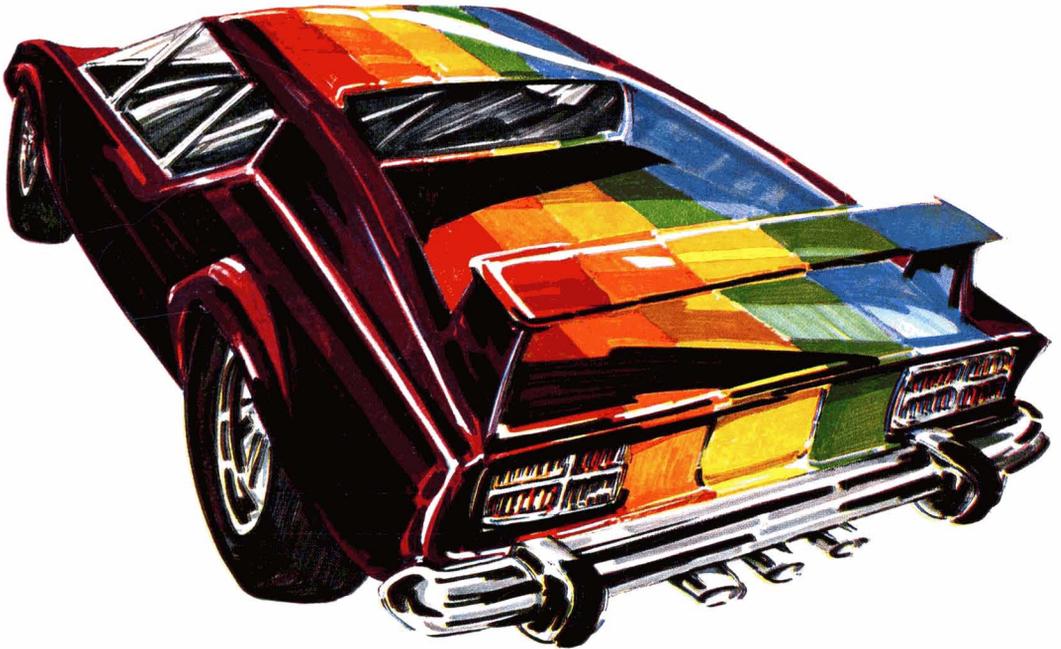
One drawback not yet overcome was a consequence of the fine particle size of the styrene/acrylate emulsion which caused poor flow. Spray application was satisfactory, but brushing was less so and two coats were essential to ensure adequate coverage.

Mr Safe's talk was well illustrated with colour slides and provoked a number of questions. A vote of thanks was proposed by Mr M. J. Cochrane.

R.A.C.

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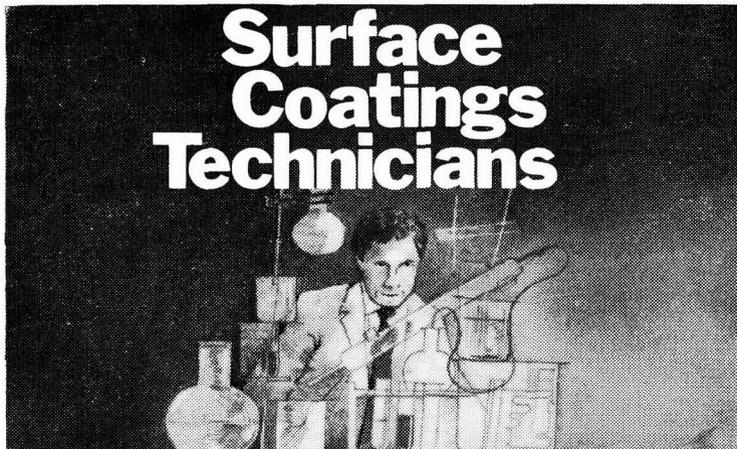
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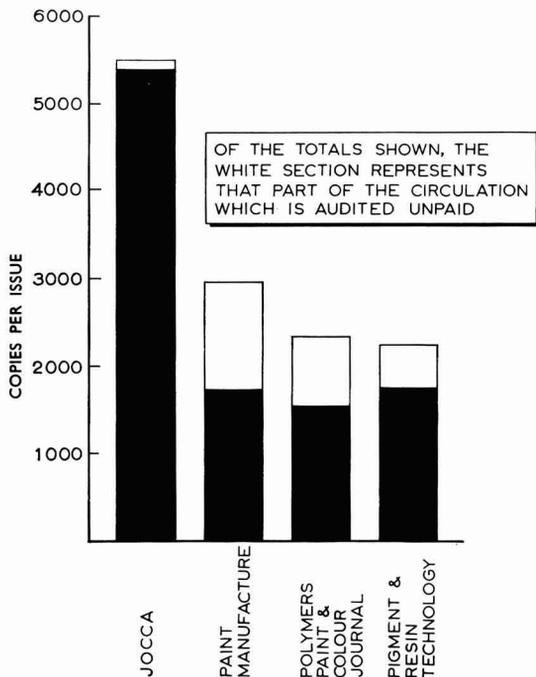
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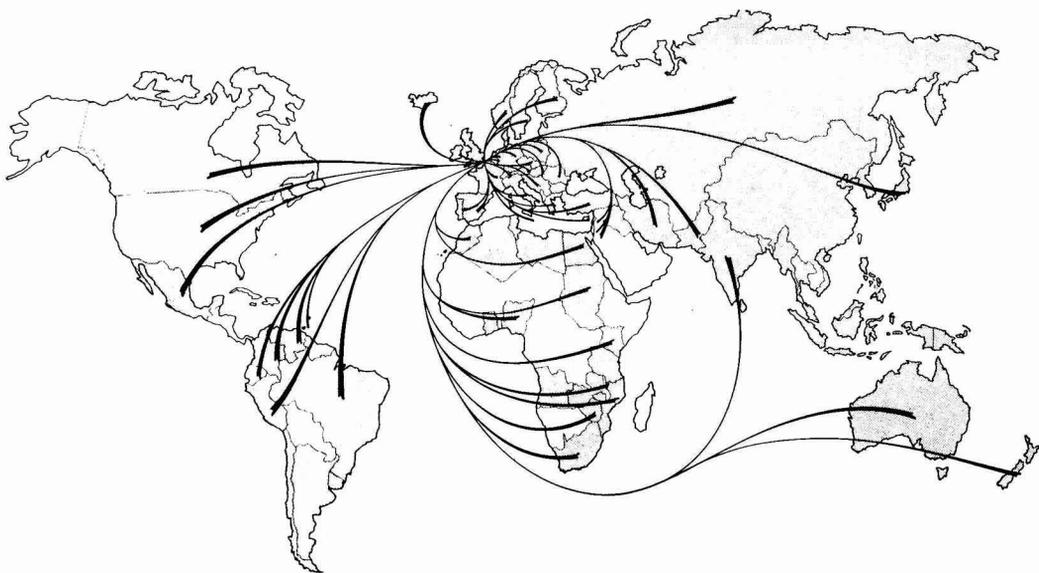
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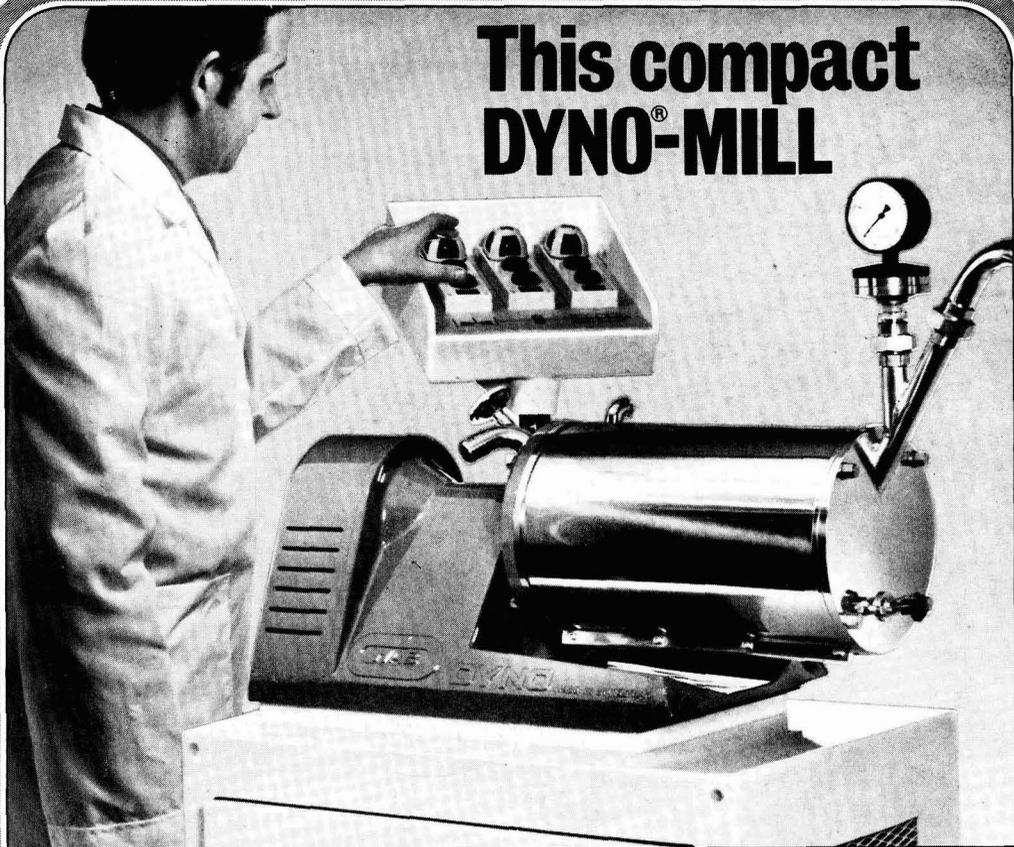
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