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**OCCA 26 EXHIBITION**  
FIRST LIST OF EXHIBITORS—PAGE 55

# JOURNAL OF THE IL & COLOUR CHEMISTS' ASSOCIATION

Ecological consideration in the biodeterioration of interior surface coatings

*D. G. McGlown and G. Old*

Accelerated testing of durable coatings

*E. Oakley and J. J. Marron*

A study of microfouling on antifouling coatings using electron microscopy

*J. H. Bishop*

Some physical properties of pigments and their importance in relation to dispersion in plastics

*M. J. Smith*



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OLYMPIA LONDON APRIL 1974

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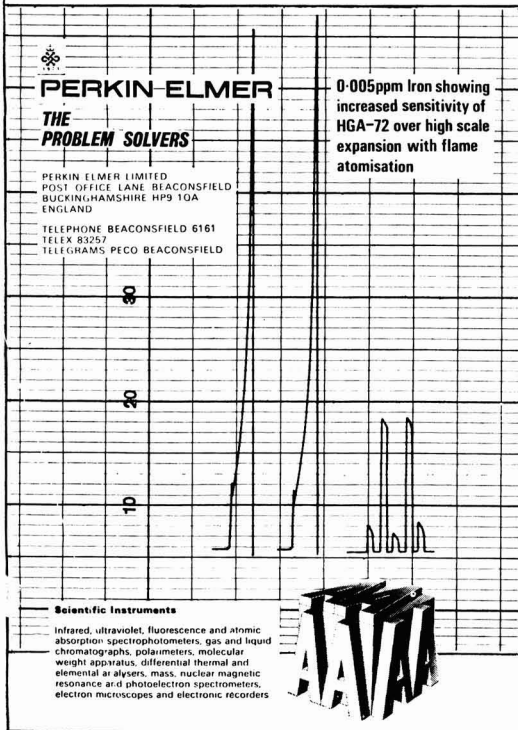
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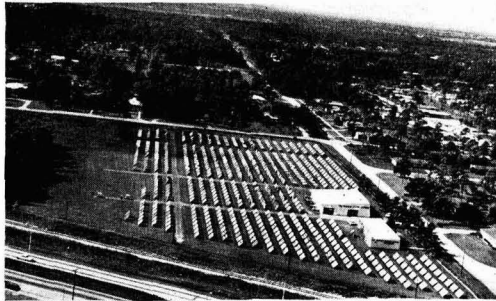
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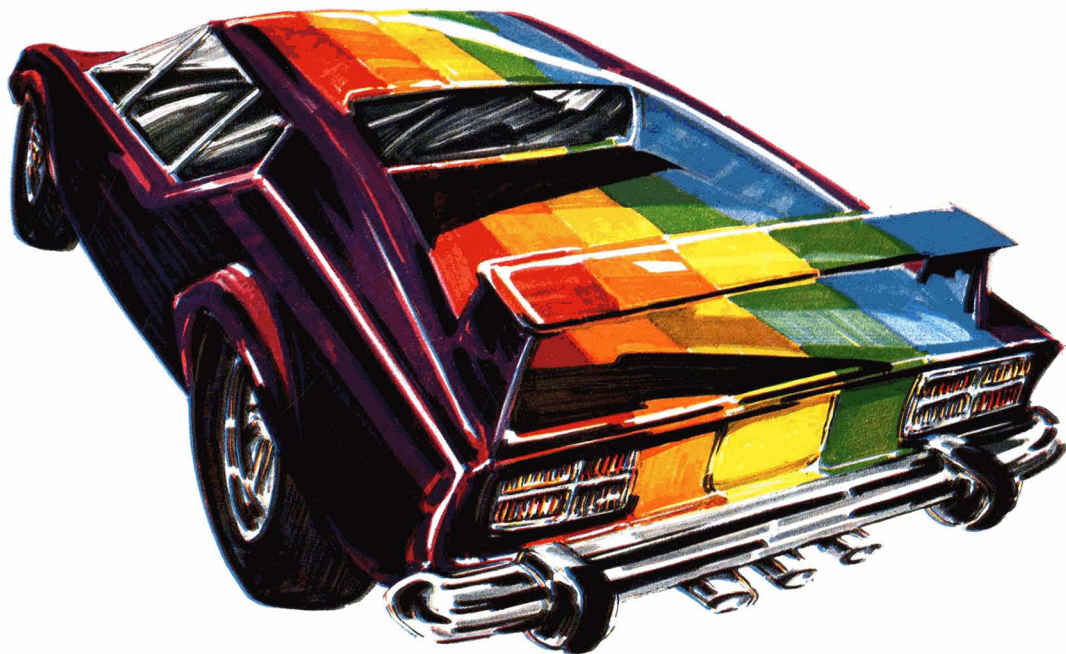
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# Ecological considerations in the biodeterioration of interior surface coatings\*

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### Summary

This paper seeks to promote an "ecological" viewpoint and approach to the problem of biodeterioration of interior surface coatings; a recognition of the fact that in biodeterioration there is involved a complex eco-system of organisms, substrate factors and environmental conditions, which must be understood before inter-disciplinary efforts can be made to ameliorate or prevent microbiological growth.

The factors necessary for microbiological growth are discussed, namely moisture, temperature, oxygen, pH and food requirements, and it is shown that these factors must be in balance to promote optimum growth. From a consideration of practical findings in their observation of instances of biodeterioration, the authors re-assess this balance of requirements in terms of the micro-organisms concerned, substrate factors, surface treatment con-

siderations and environmental conditions of high relative humidity, low ventilation and high thermal conductivity of building materials.

Recent changes in occupant behaviour and in architectural and building practices are discussed, and this consideration leads to a possible explanation of why the problem of biodeterioration is increasing.

The paper concludes with an account of the authors' practical experience of prevention and treatment, in which specific suggestions for treatment are outlined. It is emphasised throughout that prevention presents a complex and multi-factorial problem, calling for an inter-disciplinary approach for its understanding and practical implementation.

### Keywords

*Raw materials: biologically active agents  
fungicide*

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

### *The environment*

*humidity  
ambient temperature  
geographical location*

*Miscellaneous  
biodeterioration*

## Considérations écologiques dans le domaine de la biodégradation de revêtements superficiels à l'intérieur

### Résumé

Cet exposé a pour but l'avancement d'un point de vue et d'un abord "écologique" au problème de la biodégradation des revêtements superficiels à l'intérieur, de la reconnaissance du fait que la biodégradation est provoquée par un système complexe d'organismes, des aspects du support, et des conditions de l'environnement, que l'on doit comprendre avant que l'on puisse faire les efforts interdisciplinaires vers l'amélioration ou la prévention de la croissance de micro-organismes.

On discute les facteurs nécessaires pour cette croissance, tels que l'humidité, la température, l'oxygène, le pH, et la nourriture, et l'on montre que ces facteurs doivent être dans un certain équilibre afin d'assurer la croissance optimale. A partir d'une considération des conclusions mises en évidence lors de leurs observations des exemples de la biodégradation en pratique, les auteurs font une réappréciation de cet équilibre de besoins au point de vue des

micro-organismes dont il s'agit, les facteurs du support, le traitement de sa surface, ainsi que les conditions de l'environnement, telles que l'humidité relative élevée, la faible ventilation, et la conductivité thermique élevée des matériaux de construction.

On discute les changements récents à l'égard de la mode de vie des locataires et des techniques de l'industrie de construction, et cette considération parvient à une explication éventuelle de la raison pour l'élargissement du problème de biodégradation.

L'exposé se termine par un compte rendu de l'expérience pratique des auteurs dans le domaine de prévention et de traitement, où ils mentionnent des suggestions spécifiques de traitement. On souligne partout que la prévention présente un problème complexe à plusieurs facteurs, qui fait appel à un abord interdisciplinaire afin de le comprendre et de l'accomplir en pratique.

## Ecologische Betrachtungen über den biologischen Abbau von für Innen bestimmten Anstrichmitteln

### Zusammenfassung

Diese Arbeit bemüht sich, einen "ecologischen" Gesichtspunkt und Weg zum Problem des biologischen Abbaus von Anstrichmitteln für Innen zu suchen; eine Erkennung der Tatsache, dass ein komplexes Eco-System von Organismen, Faktoren betreffend das Substrat und durch die Umgebung bedingte Verhältnisse in biologische Verschlechterung verwickelt sind. Dies muss zunächst verstanden werden, ehe interdisziplinäre Anstrengungen zur Verringerung oder Verhütung mikrobiologischen Wachstums gemacht werden können.

Die für das mikrobiologische Wachstum notwendigen Faktoren werden besprochen, und zwar Feuchtigkeit, Temperatur, Sauer-

stoff, pH und Bedarf an Nahrung, wobei auch aufgezeigt wird, dass diese Faktoren für optimales Wachstum in einem Gleichgewicht stehen müssen. Als Folge praktischer bei Beobachtung von Fällen biologischen Abbaus gemachter Feststellungen bewerten die Autoren dieses Gleichgewicht in den Erfordernissen mit den Begriffen: mit Mikroorganismen zusammenhängende Substrat-Faktoren, Oberflächenbehandlung und Umweltverhältnisse hoher relativen Feuchtigkeit, schlechte Ventilation und hohe Wärmeleitfähigkeit von Baustoffen.

Besprochen werden ebenfalls in neuerer Zeit vor sich gegangene Veränderungen im Verhalten von Bewohnern, sowie in den

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Bräuchen von Architekten und Bauleuten, woraus möglicherweise Schlüsse gezogen werden können, warum das Problem der biologischen Verschlechterung im Ansteigen begriffen ist.

Die Arbeit wird mit einer Schilderung der bei Massnahmen zur Verhütung und Behandlung von den Autoren gesammelten Erfah-

runge abgeschlossen, wobei spezifische Vorschläge für Behandlungsmethoden skizziert werden. Es wird immer wieder betont, dass Verhütung ein kompliziertes und vielseitiges Problem darstellt, welches für sein Verständnis und praktische Verwirklichung eine interdisziplinäre Behandlung erfordert.

## Экологические соображения в биологической дегерации внутренних поверхностных покрытий

### Резюме

Настоящая статья пытается выдвинуть «экологическую» точку зрения и подход к задаче биологического разрушения внутренних поверхностных покрытий и подчеркнуть тот факт что биодегерация включает в себе сложную экологическую систему организмов, субстратных факторов и условий окружающей среды, которые должны быть выяснены прежде чем междисциплинарные усилия могут быть приложены для улучшения или предотвращения микро-биологического роста.

Обсуждаются факторы необходимые для микро-биологического роста как например влажность, температура, кислород, pH и пищевые требования и показано что эти факторы должны быть в состоянии равновесия для обеспечения оптимального роста. Из практических соображений и выводов авторы, наблюдая случаи биологического разрушения, проводят оценку этого равновесия, учитывая данные микроорганизмы, субстратные факторы, поверхност-

ную обработку и условия окружающей среды – высокой относительной влажности, плохой вентиляции и высокой теплопроводности строительных материалов.

Обсуждаются происшедшие за последнее время изменения в поведении жильцов и в архитектуре и строительной практике и эти соображения приводят к возможному выяснению почему проблема биологического разрушения становится более интенсивной.

В заключение статья даст отчет практического опыта авторов в предохранении и обработке, в котором даются специальные предложения для обработки. Особо подчеркивается что предохранение является сложной и много-факторной проблемой, требующей междисциплинарного подхода для ее полного понимания и практического применения.

## Introduction

### Ref: 1

The biological literature in the field of paint technology displays a peculiar tendency towards a fragmentation of its concern. Thus, bacteria are normally considered in relation to the spoilage of raw materials and the storage of the liquid product; moulds and fungi are regarded as organisms causing deterioration and spoiling the appearance of the final dry film; and, in this latter context, a high proportion of the literature is devoted to the consideration of exterior surface problems, as opposed to those occurring in interior locations. Preoccupation with the conditions of use of a surface coating is understandable and must remain an important factor for consideration, but the result of the trend towards specificity of concern is to obscure the inter-dependence and inter-relatedness of a host of variables involved in the biodeterioration of surface coatings. Moulds can, and do, attack the liquid product; bacteria are involved in the initial attack on dry paint films, irrespective of the substrate; and a combination or succession of types of micro-organisms are normally involved in the biodeterioration of a surface coating. Moreover, the problem is more complex than it appears not only because of the variety of organisms involved, but also by virtue of the variety of other factors contributing to biodeterioration. Such factors include not only the variation in the composition of the coating, its adhesiveness, hardness, chalking characteristics, PVC etc, but also environmental considerations, such as the nature of the substrate, moisture build-up in the coating, temperature, relative humidity, thermal conductivity of the substrate, changes in building practice and architectural specification, and even the habits and behaviour of the persons occupying the buildings. Accordingly, to appreciate the extent and ramifications of the biodeterioration of surface coatings, it is necessary to take a more generalised and "ecological" view of the problems.

It is recognised that in the prevailing popular scientific climate the term "ecological" has acquired fashionable connotations. However, in this paper the intention is to demonstrate an important aspect of the problem of biodeterioration which has for a long time either gone completely unnoticed, or has received too little attention. This aspect

is that in the biodeterioration of surface coatings a complex eco-system is involved, which includes a delicate balance between the varieties of micro-organisms involved, the environmental variables of conditions, substrates and coatings and the human elements of the behaviour of the architect, builder and occupant. The problem is, therefore, a global one and hence not solely the concern of the surface coatings manufacturer. Since the experiences and opinions presented in this paper reflect work in the fields of paints and wall-papers, and for the most part, the biodeterioration of interior decorative systems, it is concerned with the interests and problems of the surface coatings' manufacturer, but an attempt is made to illustrate the extent of the problem, and to show that many more "experts" should be involved in a collective appraisal and study of the problems.

Superficially, the inter-relationships of the multiple variables involved in the biodeterioration of surface coatings appear conceptually difficult. This paper seeks to provide a clearer perspective on why, and precisely how, biodeterioration occurs, and why it is important that the problem should be seen in a broader context, requiring an inter-disciplinary approach to prevention and treatment. At the present time, the problem is largely left to the surface coating industry to be dealt with by the expedient of attempting adequately to "preserve" its products, irrespective of the conditions to which they are likely to be exposed. This is, perhaps, the reason why the problem of biodeterioration increases yearly, for whilst the biologist concerns himself with the micro-organisms, the paint technologist with the surface coatings and the architect with building fabrics and systems, few studies are made of the contributions which each could make towards a solution of the total environmental problem. Ross<sup>1</sup> has suggested that the role of the biologist in the paint industry is analogous to that of "an insurance salesman and life insurance." He argues, "The paint chemist, unable to predict where his coatings will be used, how they will be applied, or to what environmental conditions they may be subjected, employs a microbicide for insurance." The practical outcome of the "insurance mentality" foisted upon the industry, is rather to cast the biologist into the role of a "troubleshooter." When times are good he assumes his "insurance" role and promotes good housekeeping; when serious biodeterioration occurs, he sets out to apportion

blame for the occurrence. For when the worst happens in a given location, much ingenuity goes into each participant's (architect, builder, building fabric manufacturer, surface coating manufacturer, painter, occupant) disclaiming of responsibility.

Perhaps the time has arrived, therefore, to look more closely and more broadly at the problem of the biodeterioration of surface treatments. Some 12 to 15 years ago the authors' experience indicated that the problem in the UK was mainly confined to the south of England; that is, south of a line drawn from Plymouth to the Wash. In recent years, the locations of serious problems have ranged from the south-west of England to the north of Scotland and, in fact, now cover the entire British Isles. It will suffice at this stage to point out that there are indications that with the increased use of industrialised building systems and the trend towards high-rise flats, the problem is likely to increase still further in the future. This paper, therefore, seeks to ascertain the conditions suitable for microbiological growth, and from a consideration of the basic data, to elicit why the problem of biodeterioration is increasing, and how it might prove possible to treat and/or prevent it.

### Factors necessary for microbiological growth

Many of the factors involved have been considered in other papers given at this symposium, so that a brief outline only will be given and sufficient to encourage the ecological perspective advocated.

#### Moisture requirements

In relation to micro-organisms, growth is connected with the available moisture, which is normally considered in relation to the "water activity" (AW) of any particular source of nutrient. Water activity is usually calculated as the vapour pressure of the solution (there are usually solutes present in the water) as a percentage of the vapour pressure of the solvent (usually water). A figure of 20 per cent moisture is often cited as the minimum moisture level necessary to permit fungi to grow in wood, and in the food industry it is known that in the case of flour and some dried fruits, if the product can be maintained at a total moisture content below 14 to 15 per cent then mould growth will be greatly delayed.

In general, bacteria require more available moisture than do moulds, but some moulds can exist and grow at an AW approaching that of pure water. Fungi on the other hand, require relatively low levels of moisture and grow best on a solid or semi-solid substrate.

As regards surface coatings, water environment in this sense can mean as little as trapped condensate, or continuous interchange between substrate/coating and an environment in which the relative humidity is high. Certain minimum quantities are sharply limiting to microbiological growth, but the presence of moisture predisposes surface coatings to biodeterioration and/or habitation, and this may be taken as the greatest single factor relevant to microbiological growth.

#### Temperature requirements

Temperature, which might appear to be vital, is concerned only in determining the rate of growth in a given environment. In general, micro-organisms can exist over a very

wide range of temperature. Individual types of organisms obviously have their individual optimum temperature ranges and this fact assists in classifying organisms according to their temperature requirements as cryophilic, mesophilic and thermophilic; but this is only a generalisation for the full field of micro-organisms. Some organisms fall outside this general classification, for example anthrax *bacillus* which can exist between 100°C and -269°C, the temperature of liquid helium. The point is that for practical purposes, micro-organisms thrive more readily in warm conditions, and their growth is retarded by cold conditions, but they are not killed by any of the temperatures likely to be encountered in buildings.

#### Oxygen requirements

Most moulds require free oxygen. As is commonly known, bacteria are usually classified as being aerobic, when they require the presence of free oxygen, or anaerobic. When they exist only in the absence of oxygen, they are termed "obligate anaerobes," but others which can either tolerate or dispense with oxygen are "facultative anaerobes." Still other micro-organisms, the micro-aerophils, thrive best in the presence of traces of oxygen.

#### pH requirements

Moulds grow over a fairly wide range of pH values from 2.0 to 8.5, although they tend to favour an acidic medium. Most bacteria grow best at a pH approaching neutrality, although some favour an acidic condition and a few grow well in alkaline media.

#### Food requirements

Generally speaking, bacteria have a definite range of food requirements. Some bacteria have a fairly wide range, but many individual types are very specific in their food requirements. Moulds, generally, possess a wide range of food requirements varying from very simple to the complex.

In either case, a source of energy is a pre-requisite for the life process to continue. In nature, the energy source is usually carbon—certain specialised organisms can utilise directly elemental sulfur or iron. In surface coatings, carbon-containing energy sources vary from paper, cellulosic and proteinaceous thickeners, portions of the molecule of the binding medium itself, to contaminants fouling the surface, organic and/or mineral impurities in water coming into contact with the coating, and to the remains of preceding generations of micro-organisms. Some organisms survive at the expense of the surface coating; that is, its vehicle component most often serves as a food source for the organisms, and since such organisms have the ability to concentrate certain elements from a dilute environment, the levels of nutrition required can be amazingly low.

Thus, it can be seen that the requisite minimal amounts of water to sustain a microbiological population are frequently encountered in the micro-environments of surface coatings. Likewise, most of the temperatures, pH values and oxygen levels encountered in the utilisation of such coatings are within the range of tolerance of micro-organisms and indeed, are frequently within the optimum range. Essential nutritional elements are almost always present in ample amounts, thus the chances of not having an acceptable environment for microbiological growth are practically nil, once water is provided in sufficient quantity.



The theoretical considerations relevant to the growth of micro-organisms have been summarised above. The effects of such growth are very far reaching indeed, and have occupied the attention of numerous surface coating technologists for a considerable time. It is evident that for microbial development, a suitable balance of conditions is necessary, and that this balance provides the key to adequate prevention and control.

It has been stated that because of the tremendous latitude in both the amount and type of growth requirements which can be tolerated by micro-organisms, the chances are extremely high that a suitable environment for growth will be provided by any surface coating. However, in the course of laboratory investigations the following practical findings have emerged, which must be set against the theoretical considerations to provide insight into how to deal with the problem.

### Practical findings

Experience has shown that:

1. Although the porous texture of plaster provides an anchorage for developing mould spores, many troublesome species of mould will not grow over plaster until it has become contaminated with a layer of dust and has been subjected to very high relative humidities (85-95 per cent).

2. The presence of a surface coating of some kind, be it glue-size treatment or a conventional paint system, is absolutely necessary for the growth of moulds over plaster samples held at moderate levels of relative humidity (75-80 per cent).

3. In cases of severe biodeterioration and/or habitation of surface coatings *in situ*, moulds have been isolated irrespective of whether the substrate was paint, wallpaper or the plaster lining of the building fabric. Thus, the mould infection when present has been distributed throughout the surface coating and the plaster lining. Infections have usually comprised surface growing species, which may be wiped or brushed off without leaving disfiguring stains, together with dark-walled, deeply-penetrating species, which produce a discoloration which cannot be washed off.

4. When inspected, surfaces which have been seriously affected have, nevertheless, appeared in good, sound condition and could not normally be described as damp, although in every case there has been evidence that condensation has occurred. Moisture meter readings have varied considerably, depending on the time of the day when they were taken.

5. Under laboratory conditions, moulds do not thrive on oleoresinous or alkyd-based painted surfaces unless the coatings have become fouled, greased or otherwise seriously soiled.

6. Factors common to the inspection of serious infection in a range of interior materials, treatments, geographical locations etc, have been found to be the prevalence of a combination of high relative humidity, low ventilation, and high conductivity of building materials.

The above findings enable a reformulation of the factors necessary for mould growth to arise in buildings as:

- (a) The presence of spores of the moulds in question.
- (b) The presence of suitable material on which the moulds may thrive.

(c) The prevalence of high relative humidity, low ventilation, and high conductivity of the building materials.

Moreover, this indicates that a complex balance is involved in which the contribution of the surface coating is only one factor, and where the crucial factor appears to be the water environment provided by high relative humidity.

It may seem to be labouring the obvious to reiterate these practical findings, but they do serve to stress the point that a balance of factors is involved in the conditions leading to the biodeterioration of surface coatings. Only by a consideration of these facts, allied to a study of the conditions under which biodeterioration does not occur, can a perspective be gained of the true nature of the problem. A conscious decision might be involved to the effect that a surface coating manufacturer's product must be capable of application and service under the worst possible conditions conducive to microbiological growth. What happens in the case where this simple expedient fails? Unless there is a clear picture of the relevance of each environmental factor concerned in the case, there is no indication as to which alternative variable is to be altered to enable the surface coating to give satisfactory service. A study of the eco-system concerned should provide valuable insight into (a) alternative approaches to a solution of the problem and (b) the requisite remedial action when a surface coating system has failed in use.

Accordingly, it might prove useful to integrate the previous practical findings with the basic data concerning the factors necessary for microbiological growth, and to broaden the consideration of the variables present in typical instances of biodeterioration of interior surface coatings; for this purpose, such variables as the organisms concerned, substrate factors, surface coating factors and environmental factors will be considered, and an attempt made to elicit the part played by each set of factors leading to the total problem of biodeterioration.

### Organisms

Ref: 2-7

It is generally realised that whenever decaying organic matter is exposed to the atmosphere, there is a plentiful source of micro-organisms; that dust and soil particles carried about by air currents, in fact, carry with them various micro-organisms. What is not so often realised is the extent of this potential source of infection, or the great number of organisms involved. It has been estimated that the number of individual micro-organisms in soil is probably in the region of  $10^8$  g<sup>-1</sup>, and in this context it is not surprising, therefore, that the majority of micro-organisms which are eventually involved in the attack of surface coatings are common soil organisms.

The literature of paint technology abounds with accounts of surveys of the types of micro-organisms associated with paint films, most of them relating to exterior conditions, and frequently implicating the predominant exterior fungal organism *Pullularia pullulans*. Perhaps the most complete surveys of this type are represented by those of Klens and Long<sup>2</sup> and Rothwell.<sup>3</sup> On the other hand, surveys of the microflora on interior paint films, notably those by Galloway,<sup>4</sup> Krumperman<sup>5</sup> and Koda<sup>6</sup> have shown that the number of different fungal genera isolated are numerous and overlap each other in frequency; and include extensive growth of many fungi rarely found on exterior surfaces. Furthermore, significant work emerging from the welter of literature includes that by Ross and Wienert<sup>7</sup> which established the part

played by bacteria at the paint/substrate interface in the biodegradation of oil- or alkyd-based primers, and the importance of this to the adhesion of paint films.

Similar surveys in the course of the authors' laboratory investigations of numerous instances of biodeterioration of interior surface coatings (including a variety of "treatments" and materials other than paints) have revealed a surprising range of micro-organisms involved. Unpublished work in 1968, for example, on water-paint coated asbestos flocking revealed an instance of severe biodeterioration of the coating in which the organisms isolated included (a) bacteria, (b) encysted amebae and other unicellular animalcules, and (c) mould species including *Monilia grisea*, *Gonytrichum* sp. and *Penicillium* spp., which suggested a complex "succession" of microbiological types as the condition of biodeterioration was established and progressed. Subsequent experience has led to the conjecture that a similar succession of types of micro-organisms is involved in most instances of biodeterioration of surface coatings, the specific type of "end point" or final predominant fungus, depending on a variety of local factors. Some supporting evidence for a succession of micro-organisms or some kind of symbiotic state of affairs, is provided by the practical observation that two broad classes of fungi occurred at the same time in cases of mould growth over coated surfaces. Such cases have involved a combination of (a) superficial vari-coloured (blue, green, pink, etc) mould growths, predominantly *Aspergillus*, *Penicillium*, *Cephalosporium*, *Actinomyces* and *Stemphylium* species and (b) deeply penetrating dark mould growths, predominantly *Cladosporium herbarum*, *Alternaria tenuis*, *Aspergillus niger*, *Paecilomyces* sp., *Scopulariopsis* sp. and *Gliomastix convoluta* species. The former group provides stains capable of removal, if conditions are improved; whereas the latter group results in intractable disfiguring stains.

The types of organism mentioned above are relatively easy to identify and much is known about their geographical distribution. The important point is that, even in the presence of fungicides in the paint, such organisms play an important role in the long-term durability of the paint film. As a liquid paint converts to a dry film, so an environmental change occurs in that it is sometimes possible for the film to provide a suitable substrate for the growth of bacteria and fungi. Microbial growth is greatly influenced by the nature of the resultant film, the surface being painted and the general environment of the painted surface.

### Substrate factors

Ref: 8

The growth of fungi, on the whole, occurs more frequently on paint films applied over wooden surfaces than on those over metal or masonry substrates. However, Ross<sup>8</sup> has stated that "Paint films on exterior surfaces represent the greatest number of microbiological problems, yet the severest disfigurement of painted surfaces by fungi occurs on the interior walls and ceilings of breweries, dairies, food processing plants etc, where the conditions of temperature and humidity are ideal for maximum growth." In recent years, "certain domestic dwellings" should be added to this list and it is worthy of note that in all of these locations, the greatest area of difficulty is concerned with the growth of mould over the surface of coated plaster.

In the authors' experience, when such mould growth occurs over plaster, the vegetative structure of the mould is frequently distributed throughout the plaster, and often in the

underlying brickwork as well. This introduces the point that plaster is a porous material capable of providing an anchorage for developing mould spores, and also capable of taking up appreciable quantities of water. In laboratory trials, in which dried plaster samples (particularly of the lightweight Carlite type) have been exposed in a closed system operating at a relative humidity of 80 per cent, a definite and consistent uptake of water has been demonstrated. Hence, in the minute non-continuous voids which are present throughout the structure of plaster, the micro-climate is often highly saturated with water and conducive to the germination of mould spores; and under certain conditions plaster may behave in a somewhat sponge-like manner, taking in and giving out moisture as the surrounding conditions change. Plaster is, therefore, a special substrate worthy of particular consideration.

In practice, if conditions are favourable, the domestic consumer often prefers to decorate plaster surfaces with wallpaper. If the conditions change towards those conducive to microbiological growth, he might have recourse to paint, usually emulsion, which does not seal the substrate. Under even wetter conditions of the plaster, the painter applies a certain water paint to allow the plaster to "breathe." In the worst possible conditions, such as occur in some breweries, old churches, cellars etc, the only practicable "decoration" of the surface may be whitewash. Such, then, is the practice which has developed over generations; a practice which still reflects an intelligent appraisal of present conditions. However, it also represents the inevitable sequence of events from the application of the wet plaster to the obtaining of a decorative surface. Conversely, when conditions have been favourable and the plaster is sound, there may be instances where one practical approach is to seal off the plaster by means of a fungicidal coating.

### Surface coating factors

Ref: 1, 9

Wallpaper, by its very nature, is particularly prone to microbiological attack. Moreover, it is commonly attached to plaster by means of starch or cellulose adhesives and is applied over a glue-size or cellulose pretreatment. It provides, therefore, nutrient materials (of both nitrogen and carbon) for micro-organisms, and is also particularly vulnerable to dampness, condensation and consequent loss of adhesion. However, it is not impossible to protect such a decorative system, as evidenced by the provision in recent years of coated, water-resistant, "washable" wallpapers and fungicidal adhesives; materials which offer the possibility of a continued provision of wallpapers and vinyl wall-coverings capable of use in all but the worst of environments. Certainly, such materials now compete favourably with emulsion paints for application in kitchens and bathrooms.

Many water-based paints contain casein or cellulose derivatives as thickening agents, and the inclusion of emulsifiers, surfactants and other additives, which counteract the improved bacterial and mould resistance of the synthetic, vinyl-type binders. Hence, these paints are rendered akin to wallpaper systems in their liability to microbiological attack, for although their binders provide increased water resistance and a more resistant chemical structure, their remaining components are particularly vulnerable to attack. Their formulation, therefore, represents a delicate balance viewed from a biological angle, and an otherwise satisfactory product can be rendered valueless, unless well protected by a bactericidal and fungicidal additive.

Oil and alkyd binders when wetted absorb varying amounts of moisture. Since the amount of moisture retained within a paint film determines the degree to which it is attacked by micro-organisms, it follows that the greater the water resistance of paint binders, the less favourable is the environment for microbiological growth. In this context, the higher viscosity oil and alkyd resins, in general, provide greater bacterial and fungal resistance, and by including in them a chemical anti-microbial agent it is possible to provide truly bactericidal and/or fungicidal coatings.

Finally, one more factor which influences the overall resistance of the paint film to micro-organisms is the choice of pigments and of their volumetric proportions in the paint film. Lead pigments tend to decrease the water absorption of a paint film. Diatomaceous silica and other flattening pigments tend to increase the water contact with the binder; moreover, any binder not directly in contact with the pigment is more easily hydrated and, hence, more readily accessible to microbiological attack.

If the foregoing considerations relating to surface coatings were carried to their logical conclusion, they would suggest one approach to the solution of the problem of microbiological growth in the interiors of buildings: an attempt to eliminate nutrient materials from the surfaces of rooms. Unfortunately, such an attempt is successful only up to a point. Hence, for example, various workers have aimed at the substitution of a "plastic" basis for glue-size and casein, and of a "plastic" adhesive for paste; but at the present time all such materials would be too expensive compared to the conventional materials. Accordingly, therefore, current practice is concerned with making surface coatings as resistant as possible, and then by the addition of a microbicide attempting to render them microbiostatic or microbiodical. In this latter context, both Ross<sup>1</sup> in the USA and Hanan<sup>9</sup> in France have demonstrated that the durability of a paint film can be significantly increased by the inclusion of adequate fungicide in priming coats. Indeed, Hanan has established that "1 per cent fungicide placed in the top coat will not afford as much protection as 1 per cent of the same fungicide placed in the primer."

## Environmental factors

*Ref: 10, 10*

The importance of environment with respect to fungal behaviour and variability on a paint film has been recognised, but its importance to the total problem has received less attention. In recent years, few workers have dealt with the "physical" aspects of the problem, the effects of which appear to have been accepted with an air of resignation. Yet the physical aspects of the environments created in buildings are of vital importance to an understanding of the problem of biodeterioration, and perhaps hold the key to an alternative approach to prevention and control. Many years ago, Bastings<sup>10</sup> working in New Zealand produced a classic paper tracing the occurrence of mould growth in modern houses to the prevalence of high humidity indoors caused by insufficient ventilation and inadequate natural ventilation of rooms. Obviously, this is relevant to the authors' own more recent findings; the paper should not become lost in antiquity and should certainly be required reading for all architects. This paper described a report by one of the Auckland Corporation staff which threw significant light on the problem at that time, and is highly relevant to current conditions in modern dwellings in Britain today. It was reported that "a block of houses which had, in the early part of the year,

been completed with the exception of electrical and drainage services, and which, in consequence, had not been inhabited during the winter. Not one house had any fungal growth on the ceilings although in the neighbourhood, in houses already occupied, the trouble is very serious." Bastings, therefore, concluded that "it appeared probable that the humid conditions within the affected houses are created by the occupants themselves." This has certainly been the authors' experience in relation to modern high-rise flats and a variety of other industrialised buildings, and it could be added that the humidity is occupant-generated in the environments which are provided for them.

In recent years, appreciable changes have occurred in occupant behaviour. With the trend towards both sexes becoming family wage earners, the conditions in many dwellings consist of periods of high temperature and humidity (corresponding with the peak hours of electricity consumption, the morning rush, evening activity, bath night, out-of-hours housework and washing etc) interspersed with long periods of absence from home during which the building fabric cools rapidly, only to meet later a further period of high humidity. In such conditions condensation is inevitable, but is normally interpreted as "dampness." Moreover, in addition to this, radical changes have occurred in architectural and building practice. "Progress" has caused the demise in many dwellings of the conventional open-fire and chimney (providing natural ventilation to rooms), building materials are employed which do not approximate to the insulating properties of traditional housing, the widespread use of central heating systems and the avoidance of draughts provide a prevailing condition of warm still stagnant air. Evidence will be provided later of a survey of a large number of dwellings in a southern city in which there was a direct correlation between the occurrence of mould growth and the amount paid per occupant per quarter in gas-fired central heating—or more accurately how much the fan controlling air movement, which is tied to the production of heating, was allowed to operate.

Such factors are relevant to the variation found in moisture-meter readings. These readings are usually taken during working hours when officials and technical staff are available and when the surfaces will most probably appear sound and free from dampness. However, the occupant's frequent comment is that "last night the wall was absolutely running with water," taken with the above observations and the sponge-like behaviour of certain treated plasters, should enable a broader perspective to be obtained about what is happening in such dwellings. The previous cited paper by Bastings<sup>10</sup> provides detailed methods for estimating the deficiencies in any given room with respect to insulation and ventilation, and offers suggestions as to how such defects may be overcome reasonably and economically, both in existing and new houses. In the course of an examination of instances of severe mould growth, Bastings made the following observations:

1. In 1943, after a survey of 671 houses in 16 districts throughout the country (New Zealand) he found a total of 53 per cent to be affected by severe mould growth.
2. Comparing the conditions prevalent in rooms seriously affected by mould growth with those occurring in rooms unaffected, Bastings found:
  - (i) high humidities and very low indoor temperatures occurred frequently in all the modern rooms, but the conditions were much less severe in the older types of house;
  - (ii) "in the traditional type of house, the mean relative humidity (RH) over the period of both surveys was found

to be appreciably below 80 per cent and considerably below the outside mean humidity. In most modern rooms, on the other hand, the mean RH was above 80 per cent, in some cases considerably above this figure, and in at least one case higher than outside;

(iii) "the data suggested the existence of a limiting value for the mean relative humidity, below which mould does not thrive in houses . . . we may, with a margin of safety, accept 80 per cent RH as the lower limit of mould incidence in houses;

(iv) "assuming a mean RH outside of 86 per cent and an optimum mean RH indoors of 80 per cent, we see that to ensure freedom from mould it is necessary to preserve the inside mean humidity at about 6 per cent below the outside value. Now, when air at 86 per cent RH is warmed by 2°F, its RH automatically falls to about 80 per cent. Hence it follows that, if no other disturbing factors intervene, it is only necessary to ensure a permanent rise of 2°F in the temperature of air entering a room from outside to avoid mould growth;

(v) in all modern rooms studied, ceiling temperatures were found to be below the air temperatures in the room. In the traditional type of house the ceiling temperature was above room temperature. This implies a continuous loss of heat from the room through the ceiling;

(vi) Wall temperatures at most sites were lower than the temperatures of the air in the room, again indicating a continuous and extensive drain of heat from the room;

(vii) in a great proportion of modern houses, windows and fanlights are usually kept closed at night. The rate of air change in typical modern rooms in calm weather is of the order of one per hour. In all modern houses investigated, the air speed in the cavity exceeded  $8\text{ft min}^{-1}$ , reaching as much as  $60\text{ft min}^{-1}$  in brick-veneer houses. The air moving in these cavities is outside air. "The effect of this large current of cold air upon the interior temperatures of rooms, separated as they are from it, in modern houses, by only 0.5in or less of some such poor insulator as fibrous-plaster or plaster-and-pumice wallboard is clearly an important factor in the chain of causes leading to the prevalence of mould".

Bastings gives a mathematical treatment of the balance between gains and losses of heat and moisture in rooms and argues that it is this balance which determines the RH and temperature conditions within a room. Applying this theory and mathematical data in a predictive manner, and thereby employing the controlled use of insulating materials and alterations of ventilation rates, he found that he could produce environments in which a final mean relative humidity not higher than 80 per cent was reached, and no further mould growth occurred in these locations. In some instances, the soundness of his theory was demonstrated by the provision of no greater an alteration in the room than the provision of a 100W electric heater.

Finally, Bastings' work strongly suggested that as a minimum the average ventilation rate of about three air changes per hour might apply reasonably well for all bedrooms and living rooms in modern houses. This, it will be noted, is considerably higher than the value which appears to be general in houses today. Bastings called for a system of natural ventilation to be installed in the ceilings of all modern rooms without a fireplace. Not only has this never become common practice; it is the common experience of many corporation housing managers that where means of

ventilation such as Expelair fans, or ventilation bricks, are provided, these are either not used, or frequently blocked off.

It therefore remains to be seen whether an economical solution of the problem of biodeterioration of interior surface coatings can be found by seeking the cause of high humidity in rooms and endeavouring to reduce it permanently to a satisfactory level. Certainly, it makes an alternative approach to the problem, and at the same time throws light on the fact that the problem should be seen as requiring an interdisciplinary, collaborative approach from all those concerned in its occurrence. The economics of microbiological control should not be thrown upon the shoulders of the surface coating manufacturer exclusively. He will always be called upon to produce speciality materials for special intractable locations, such as breweries, dairies, abattoirs, food manufacturing plants and so forth, but as the increase in the problems in dwellings in recent years has shown, there is clearly a large number of instances involving the human element of occupier and architect-builder behaviour, where a combined approach to the problem of prevention and control is required.

## Prevention and treatment

*Ref: 11-15*

The experience of the authors has fully vindicated the findings of Bastings and his colleagues, Brien and Denne<sup>11</sup>, who worked on the prevention of mould growth on distempered and wallpapered surfaces at that time. This practical experience will be dealt with in more detail later in this paper, but at this point it is desired to stress the ecological perspective and to direct attention to the practical application of the theoretical aspects under consideration.

A very large number of individual instances of biodeterioration has been investigated, but the greatest increase in knowledge has been obtained where it has been possible to study a large number of similar dwellings at the same time, such as where a city housing authority is concerned. No corporation can entertain any proposed remedial treatment that is uneconomical, and it is when dealing with such instances that an insight can be obtained into how the problem might be solved realistically.

After inspecting many dwellings infected by mould growth, a repeating pattern of common factors was found to occur, immaterial of whether the dwelling was single storey, low-rise or high-rise in construction. The geographical location or the type of construction did not appear to have any great bearing upon the infection. The most significant factors in the infected properties were the conditions created by the occupants, namely lack of ventilation coupled with the amount of heat used and the fluctuations of temperature.

In numerous cases, it was found that comparisons could be made between adjoining identical properties occupied by similar family units; one dwelling might be free from mould, whilst the other had profuse growth. The infected dwellings has the same common factors, or at least the majority of them, prevalent:

- 1a. Windows closed at all times, minimising ventilation.
- 2a. No fireplaces or at the most a gas fire outlet in the room.
- 3a. Too much or too little heating coupled with poor ventilation.



4a. The property was unoccupied for a large part of each day.

5a. Laundry being dried within the dwelling contributed to the development of high humidity.

The uninfected dwellings were found to have the following contrary conditions:

1b. A reasonable degree of ventilation.

2b. A sensible temperature range.

3b. The dwelling was occupied by at least one member of the family for the major part of the day.

In most cases, it was found that even if the dwellings were unoccupied for at least 8 hours per day, compliance with the conditions 1b and 2b was sufficient to avoid mould development. Insufficient ventilation coupled with the normal occupational actions results in the humidity within the dwelling increasing to a level at which mould spores could develop and colonise the surfaces of the rooms.

Common areas of initial mould development are the angle between the ceiling and the wall above a window and down the angle between the window and adjacent walls, these usually being the coldest and darkest areas in a room. It is interesting to note that the room in which the highest humidity is created is not always the one which is most affected by mould. Wall areas, particularly where there is poor ventilation and little light (such as behind cabinets, wardrobes etc), are often the only sites of mould growth.

Before dealing with methods of treatment, it must be recognised that the eradication of biodeterioration is, at best, difficult and inadequate; prevention is the preferred action for anyone with knowledge of the causes and extent of the problem, and it is perhaps only by holding symposia like this one at Manchester that interested parties can be brought to realise this.

In terms of the physical or environmental approach to treatment, one city corporation in the west of England has demonstrated on an experimental basis that this end can indeed be achieved. By removing all infected plaster, studding the walls with Protimised timber, lining with aluminium foil-backed plaster-board, skimming with plaster and redecorating, sufficient insulation was provided to overcome the problem of condensation and the uptake of water by the heavy building fabric. Obviously, such treatment is far too expensive for any local authority to consider for extensive use. Nevertheless, the experiment enabled the corporation concerned to prove that the problem can be solved by such an approach, and, subsequently, to consider a modification of the method with the conventional use of microbiodical coatings. It should be noted, however, that a delicate balance of factors is involved for success. Thus, for example, in the same location, application to the surfaces of a 5mm foam polystyrene failed to prevent mould growth because it provided lodgement for mould spores and did not provide sufficient insulation for the existing conditions.

Suggestions made to this and other local authorities have been restricted to those which could be employed without undue difficulty and at reasonable cost. Thus, in a typical situation where the plaster is basically sound, free from persistent dampness and subject only to the combination of condensation and mould growth, the remedial approach has included:

1. Removal and burning of all infected surface material from rooms, followed by repeated (on three days) and

liberal application of fungicidal wash solution, leaving a minimum period of at least four hours before commencing any scraping or brushing to remove all mould growth, defective paint etc.

2. The provision of background heating to dry out plaster surfaces. The building fabric must again be treated with a liberal wash of fungicide to sterilise the exposed fabric in depth. The surfaces should be left for a period of not less than 72 hours, although any filling of cracks, etc, may be carried out in this period. In cases of serious mould attack, the surface should be kept under observation for a period of weeks, applying further fungicide should mould reappear.

3. In most domestic situations, the above treatment should be sufficient. There are, however, cases where infection is present in depth in porous plasterwork, and where moisture is being absorbed and retained to maintain the development of spores. In these instances, after the treatment described above and drying out of the plaster, a coat of a fungicidal oil primer should be applied to seal off the substrate. Where a wall is wet, due to some structural defect, this should be repaired and sufficient time allowed for thorough drying out.

4. Improvement in the through ventilation of flats and houses by such measures as fitting at least one ventilation brick per room, together with fitting slotted ventilators in the lower part of all doors to facilitate an improvement, no matter how small, in the air movement.

5. In the case of extremely cold walls with a northern aspect subject to more condensation than normal, the application of an expanded polystyrene lined with good quality paper is recommended, followed by papering or painting with a fungicidal treatment system as described below.

6. Following the preparatory treatments described above, redecoration has involved either of the following systems:

- (a) The application of two coats of fungicidal quality emulsion paint or alkyd based satin finish, allowing 24 hours between coats. In each case the coatings have been rendered fungicidal by the addition of a persistent fungicide.
- (b) The hanging of a suitable wallpaper, followed 48 hours after drying by the brush application of a fungicidal quality wallpaper glaze. Papers should be hung with a fungicidal adhesive.

Such an approach has resulted in a very high level of success in the treatment of serious instances of mould growth in the interiors of buildings. The very small number of partial failures has enabled local authorities to deal with them by more radical alteration of the building fabric and by the use of speciality finishes, which are normally reserved for breweries, dairies etc.

With the objective of the prevention of the problem, it is emphasised that the significant aspects of the above approach are (a) attempts at encouraging improvements in building practice and architectural design, and (b) from the viewpoint of surface treatment; the replacement of conventional glue-size treatment of plaster by a fungicidal oil-based primer, the formulation of surface coatings with a persistent microbiodical agent and the provision of this protection in all coats of a paint system, primer, undercoats and finishing coats, and the provision of a similarly protected overglaze for wallpaper.



A survey of the literature reveals that more workers are coming to realise the importance of microbiocide persistence in a paint film as a major contribution to its effectiveness. The work of Nye and Mackie<sup>12</sup>, Rayne<sup>13</sup>, Hoffmann and Bursztyn<sup>14</sup>, and O'Neill<sup>15</sup> can be cited in this connection. The temptation to use a volatile or ephemeral microbiocide for in-can preservation of the liquid product, hoping that the dry film will not be subjected to conditions which result in microbiological attack, must be resisted. Preservation of the liquid product and protection of the dry film should never be considered in isolation from each other.

### Conclusion

This paper has sought to show that the prevention and/or treatment of biodeterioration of surface coatings applied to the interior of buildings is neither simple nor straightforward; rather it is complex and multifactorial, calling for an interdisciplinary approach to its understanding and practical implementation. The concept of the biologist in the coating industry as an "insurance salesman" is inadequate and outdated, and certainly fails to appreciate the contribution that he is capable of making.

By taking an ecological viewpoint, that is by studying the environmental relations of the living world and by studying micro-organisms in relation to their environment, a deeper

understanding can be gained of the inter-dependence of all the relevant factors and of the conditions influencing growth and, therefore, it is to be hoped, of how this growth may best be controlled.

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# Accelerated testing of durable coatings\*

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## Summary

The natural weathering of five acrylic/melamine systems at two exposure sites has been compared with three accelerated test methods, using two levels of pigmentation.

An assessment has been made mainly on the basis of the rate of loss of gloss, which was the most noticeable form of degradation observed during the exposures. Five grades of titanium dioxide

manufactured by the chloride process and four sulfate process grades were used in the evaluations.

It is concluded that the "Emmaqua" apparatus, using natural sunlight as a source of radiant energy, gives a high degree of correlation with conventional weathering tests carried out at a Florida site.

## Keywords

*Processes and methods primarily associated with analysis, measurement and testing*

accelerated testing  
accelerated weathering

*Binders, resins etc*

acrylic resin  
melamine resin

## Les essais accélérés sur quelques revêtements résistant aux intempéries

### Résumé

Dans le cas de cinq systèmes acrylique/mélatamine à deux niveaux de pigmentation, on a fait comparer le vieillissement qui se produisait à deux emplacements à l'extérieure auprès de celui mis en évidence par trois diverses méthodes accélérées. L'appréciation des résultats a été basée largement sur le taux de perte de brillant, qui était, lors de l'exposition, le genre de dégradation le plus saillant. On a utilisé, au cours des évaluations, cinq types de

dioxyde de titane fabriqué par le procédé chlorure, ainsi que quatre types à partir du procédé sulfate. On conclut que l'appareil "Emmaqua", en utilisant la lumière solaire en tant que source de l'énergie radiante, donne un degré important de corrélation avec les essais de vieillissement normaux effectués à un emplacement en Florida.

## Kurzprüfen dauerhafter Beschichtungsmittel

### Zusammenfassung

Die natürliche Bewitterung von fünf Akrylat/Melamin Systemen in zwei Prüfstationen wurde mit drei Kurzprüfungsmethoden verglichen, wobei zwei Pigmentierungshöhen benutzt wurden.

Die Beurteilung beruhte hauptsächlich auf dem Grade des Glanzverlustes, der bemerkenswertesten bei der Bewitterung beobachteten Degradierungsform. Bei der Bewertung wurden fünf Qualitäten

von im Chloridverfahren und vier von im Sulfatverfahren hergestellten Titandioxyden verwandt. Man zog den Schluss, dass der, natürliches Sonnenlicht als Quelle für die Strahlungsenergie, benutzende "Emmaqua" Apparat in hohem Masse mit konventionellen, in einer in Florida befindlichen Station durchgeführten Bewitterungsprüfungen in Korrelation steht.

## Ускоренные испытания прочных покрытий

### Резюме

Натуральное выветривание пяти акрило-меламиновых систем проведенное в двух местах испытаний, сравнивалось с тремя ускоренными испытательными методами, применяя два уровня пигментации.

Делается оценка основанная преимущественно на скорости потери лоска, которая являлась наиболее заметной формой деградации наблюдаемой в течение выветривания. Пять сортов двуокиси титана изготовленных хлоридным процессом и

четыре сорта изготовленных сульфатным процессом применялись для оценки.

Авторы приходят к заключению что аппарат «EMMAQUA», применяющий натуральный солнечный свет в качестве источника чистой энергии, дает высокую степень корреляции с общепринятыми испытаниями на выветривание, проведенными во Флориде.

## Introduction

The correlation between different accelerated test methods and natural weathering has been discussed for many years by many authors, but much more work still remains to be done in this field. This paper presents the results of some work carried out by the laboratories of Tioxide International Ltd. The interest to pigment manufacturers lies largely in the relationships between pigment, resin and test conditions because it is necessary to design grades of pigment to achieve optimum performance in the widest possible field of application.

The objectives were, firstly to establish the relative performance of a series of nine pigments, secondly to explore

any possible interaction between pigments and resin type, which could give rise to differing orders of pigment performance, even with resins of the same basic type that were dissimilar only in their detailed composition.

## Experimental

### Ref: 1

The testing was carried out in five different environments to examine the possible effects that these might have on the relative behaviour of either pigments, polymers, or any interaction that may have been determined.

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Table 1 summarises the factors involved in the experiment. Firstly, there are nine pigments (1-9), five of which are manufactured by the chloride route and four by the sulfate process.

Table 1  
Experimental programme

| Pigments   | Acrylic resin                                                                                          | Colour pigmentation                             | Location                                                                                             |
|------------|--------------------------------------------------------------------------------------------------------|-------------------------------------------------|------------------------------------------------------------------------------------------------------|
| 1 Sulfate  | A } differing in monomer composition combined with a standard melamine resin in the proportion 7/3 w/w | W White—<br>1/1 w/w<br>TiO <sub>2</sub> /binder | C Carlton, UK<br>F Florida, USA<br>T Twin-arc, Marr<br>D Dew-cycle, Atlas<br>E Emmaqua, Phoenix, USA |
| 2 Sulfate  |                                                                                                        |                                                 |                                                                                                      |
| 3 Sulfate  |                                                                                                        |                                                 |                                                                                                      |
| 4 Chloride |                                                                                                        |                                                 |                                                                                                      |
| 5 Chloride |                                                                                                        |                                                 |                                                                                                      |
| 6 Chloride | G Grey—<br>0.5/1 w/w<br>+ 1 per cent carbon black                                                      |                                                 |                                                                                                      |
| 7 Chloride |                                                                                                        |                                                 |                                                                                                      |
| 8 Chloride |                                                                                                        |                                                 |                                                                                                      |
| 9 Sulfate  |                                                                                                        |                                                 |                                                                                                      |

The grades are not all available commercially and not all are manufactured by Tioxide International Ltd, four samples being of North American origin. Secondly, there are five acrylic resins (A-E) each modified with the same melamine/formaldehyde resin. The resins were prepared by BP Chemicals International Ltd and varied in composition with respect to the type and proportion of the monomers used in the manufacture. Thirdly, the finishes were prepared both as white and as pastel grey colour; the former pigmented at a 1/1 ratio of pigment/binder using TiO<sub>2</sub> only, and the latter at 0.5/1 pigment/binder with TiO<sub>2</sub> plus one per cent carbon black, calculated on the TiO<sub>2</sub> content.

All the finishes were applied as two coats "wet-on-wet" by compressed air spray and, after a suitable "flash-off" period were stoved for 30 minutes at 120°C.

Steel panels, bonderised and primed with a grey epoxy based primer were prepared for exposure at Carlton in the UK and at Miami in Florida, and also for the tests with the "Emmaqua" apparatus. Bonderised aluminium panels were used for the artificial weathering tests in a Marr twin carbon arc apparatus and in an Atlas XWR Weatherometer, operated as a "Dew-cycle" apparatus.

The conditions of test and the operation of the various machines have been published previously<sup>1</sup>. They may be summarised as:

1. Exposure at the Tioxide Weathering Test Station at Carlton, Teesside. The panels were exposed at 45° on a south facing rack and removed for examination every four months.
2. The Florida test site of the South Florida Test Service Inc. Although current practice favours 5° exposure angle for testing automobile lacquers, the samples were exposed at 45° as in the UK to avoid introducing an additional variable into the evaluation. At this site the panels were examined monthly.
3. The twin carbon arc apparatus has been modified from the original DEF 1053 specification and now incorporates two 900W enclosed carbon arcs and a water spray. The drum rotation is three revolutions per minute, the radiation is continuous and the water spray wets the sample once during each revolution. Samples were examined every 250 hours.
4. The "Dew-cycle" test was carried out in an Atlas XWR weatherometer, which operates using an unscreened

3000W sunshine carbon arc as the radiation source. The test cycle is alternate periods of one hour of irradiation at 50 per cent relative humidity and one hour at 4°C at 100 per cent relative humidity. During the dark period the backs of the panels are sprayed with cold water which causes condensation to form on the painted surface. The panels were examined every 16 hours.

5. The "Emmaqua" apparatus, developed by the Desert Sunshine Exposure Tests Organisation, utilises natural sunlight as the source of radiant energy and a system of polished aluminium mirrors to concentrate eight times the natural intensity on to the samples. The samples are sprayed with water and cooled by a continuous stream of air to maintain humidity and sample temperature at a reasonable level. The panels were examined at intervals, weekly at first and subsequently every two weeks, but the inspection interval can be described more conveniently in energy terms, that is, by the total Langleys falling per cm<sup>2</sup>.

The gloss values and chalk ratings recorded at each test location were tabulated, but examination of the data indicated that it would be almost impossible to detect any significant trends in performance without recourse to some form of mathematical analysis.

Results

Since the main criterion of performance with this type of finish is usually recognised to be gloss retention, the analysis was restricted to the recorded gloss values. By inspection of the results, the pigments were segregated into three groups, each comprising three pigments; that is, grouping according to best, moderate and worst performance for each resin and for the samples at all locations. Assuming that the Florida result would be regarded as normal, then it could be demonstrated readily that there was a considerable variation in the relative performance of the pigments. For any one set of finishes, tested in five environments, there is a very random distribution of best and worst performance among the nine pigment samples being evaluated (Fig. 1). The scatter is still

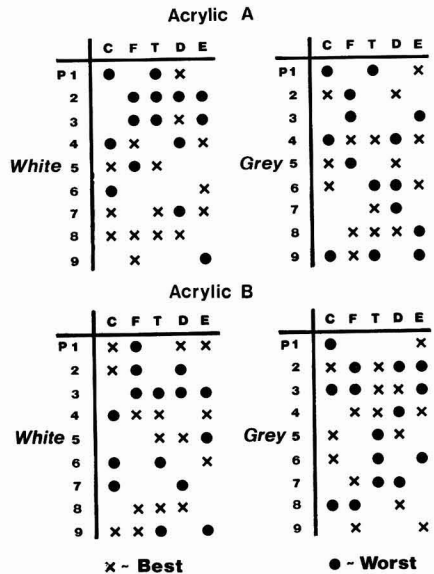


Fig. 1. Ranking by order of merit

more evident if the two levels of pigmentation are considered, and greater still if the results of two acrylics are incorporated. If this lack of any pattern becomes apparent with only two resins of the five used in the evaluation, then the need for a mathematical treatment is clearly indicated.

As a preliminary exercise, some of the data was analysed using the Spearman Rank Correlation method (Fig. 2) and

|       |       | A    | B    | C    | D     | E      |
|-------|-------|------|------|------|-------|--------|
| F v T | P     | 0.63 | 0.50 | 0.33 | 0.50  | 0.43   |
|       | Sign. | >75% | >75% | <75% | <75%  | >75%   |
| F v D | P     | 0.10 | 0.67 | 0.05 | -0.62 | -0.75  |
|       | Sign. | <75% | >95% | <50% | <10%  | <2.5%  |
| F v E | P     | 0.38 | 0.40 | 0.77 | 0.57  | 0.91   |
|       | Sign. | <75% | <75% | >98% | <90%  | >99.5% |

Fig. 2. Spearman Rank correlation analysis

the results proved to be of interest. Calculation of the correlation coefficients for the order of pigment merit, Florida versus each of the other locations, showed that only in four instances can the correlation be considered significant; that is, with acrylic B, between Florida and Dew-cycle rankings and in acrylics C, D and E between Florida and "Emmaqua" rankings, which suggests some merit in "Emmaqua" testing as means of assessing pigment performance. Two of the Florida versus Dew-cycle coefficients imply a negative correlation.

A comparison of the order of pigment performance in the five acrylic resins at Florida showed a highly significant correlation, suggesting that the choice of the acrylic had a negligible effect on the relative performance of the pigments.

These preliminary assessments confirmed the view that a more sophisticated treatment of the results would be necessary if the maximum value was to be extracted from the available data. The IBM 1130 computer available and a programme which has a capacity of 32 x 32 four digit values enabled a representative cross-section of the data to be processed for analysis of variance on a full factorial experiment.

The first analysis involved five factors, namely: eight pigments (P) (No 5 pigment, an experimental product, was omitted to facilitate full utilisation of the programme capacity); five acrylic resins (A); two pigment contents (= colour C); four locations (L) and three gloss levels at regular time intervals (T).

The most significant factor was T (Table 2), which is understandable because all films deteriorate on exposure. This influenced the results to such an extent that a re-analysis was carried out with T incorporated in the residual, resulting in the new table of variance (Table 3), in which the greatest sources of variance were location, the acrylic resin and, to a lesser extent, the pigment (Fig. 3). The interactions acrylic resin/location, pigment content/location and pigment type/location were also highly significant. Examination of the acrylic resin/location interaction (Fig. 4) shows how the Dew cycle and the twin arc exposures exaggerate the differences in the resins, which appear to be relatively insignificant on natural weathering in Florida. The colour (pigment content)/location interaction (Fig. 5) is interesting because two test methods actually reversed the expected effect of a decrease in pigmentation. At Florida and in the twin arc, the gloss

retention of the grey finishes is superior, but with "Emmaqua" and in the Dew cycle test the white, more highly pigmented finishes are superior. Closer examination of the pigment type/location interaction (Fig. 6) shows how inadequate the Dew cycle test is in discriminating between pigments and because of the possible damping effect of these values on other interactions, the results were reprocessed with the Dew cycle results omitted.

Table 2  
Table of variance 1

| Factor or interaction | Sum of squares | Degrees of freedom | Mean squares | Variance ratio | Significance (per cent) |
|-----------------------|----------------|--------------------|--------------|----------------|-------------------------|
| P                     | 16,611         | 7                  | 2,373        | 129.45         | 0.1                     |
| L                     | 172,834        | 3                  | 57,611       | 3142.83        | 0.1                     |
| PL                    | 17,362         | 21                 | 826          | 45.10          | 0.1                     |
| T                     | 182,016        | 2                  | 91,008       | 4964.70        | 0.1                     |
| PT                    | 1,088          | 14                 | 78           | 4.24           | 0.1                     |
| LT                    | 24,289         | 6                  | 4,048        | 220.84         | 0.1                     |
| PLT                   | 3,388          | 42                 | 81           | 4.40           | 0.1                     |
| C                     | 3,053          | 1                  | 3,053        | 166.55         | 0.1                     |
| PC                    | 3,077          | 7                  | 440          | 23.97          | 0.1                     |
| LC                    | 23,607         | 3                  | 7,869        | 429.28         | 0.1                     |
| PLC                   | 3,943          | 21                 | 188          | 10.24          | 0.1                     |
| TC                    | 668            | 2                  | 334          | 18.21          | 0.1                     |
| PTC                   | 1,040          | 14                 | 74           | 4.05           | 0.1                     |
| LTC                   | 5,089          | 6                  | 848          | 46.26          | 0.1                     |
| PLTC                  | 1,515          | 42                 | 36           | 1.96           | 0.1                     |
| A                     | 49,809         | 4                  | 12,452       | 679.29         | 0.1                     |
| PA                    | 2,409          | 28                 | 86           | 4.69           | 0.1                     |
| LA                    | 54,263         | 12                 | 4,522        | 246.68         | 0.1                     |
| PLA                   | 7,036          | 84                 | 84           | 4.56           | 0.1                     |
| TA                    | 2,479          | 8                  | 310          | 16.90          | 0.1                     |
| PTA                   | 1,444          | 56                 | 26           | 1.40           | —                       |
| LTA                   | 11,007         | 24                 | 459          | 25.01          | 0.1                     |
| PLTA                  | 5,218          | 168                | 31           | 1.69           | 0.1                     |
| CA                    | 2,065          | 4                  | 516          | 28.16          | 0.1                     |
| PCA                   | 1,115          | 28                 | 40           | 2.17           | 0.5                     |
| LCA                   | 7,771          | 12                 | 648          | 35.32          | 0.1                     |
| PLCA                  | 3,786          | 84                 | 45           | 2.45           | 0.1                     |
| TCA                   | 774            | 8                  | 97           | 5.27           | 0.1                     |
| PTCA                  | 1,124          | 56                 | 20           | 1.09           | —                       |
| LTCA                  | 3,559          | 24                 | 148          | 8.09           | 0.1                     |
| PLTCA                 | 3,080          | 168                | 18           | 1.00           | —                       |
| Total                 | 616,521        | 959                |              |                |                         |

Table 3  
Table of variance 2

| Factor or interaction | Sum of squares | Degrees of freedom | Mean squares | Variance ratio | Significance (per cent) |
|-----------------------|----------------|--------------------|--------------|----------------|-------------------------|
| P                     | 16,611         | 7                  | 2,373        | 6.13           | 0.1                     |
| L                     | 172,834        | 3                  | 57,611       | 148.81         | 0.1                     |
| PL                    | 17,362         | 21                 | 827          | 2.14           | 0.1                     |
| C                     | 3,053          | 1                  | 3,053        | 7.89           | 0.5                     |
| PC                    | 3,077          | 7                  | 440          | 1.14           | —                       |
| LC                    | 23,607         | 3                  | 7,869        | 20.33          | 0.1                     |
| PLC                   | 3,943          | 21                 | 188          | 0.48           | —                       |
| A                     | 49,809         | 4                  | 12,452       | 32.16          | 0.1                     |
| PA                    | 2,409          | 28                 | 86           | 0.22           | —                       |
| LA                    | 54,263         | 12                 | 4,522        | 11.68          | 0.1                     |
| PLA                   | 7,036          | 84                 | 84           | 0.22           | —                       |
| CA                    | 2,065          | 4                  | 516          | 1.33           | —                       |
| PCA                   | 1,115          | 28                 | 40           | 0.10           | —                       |
| LCA                   | 7,771          | 12                 | 648          | 1.67           | —                       |
| PLCA                  | 3,786          | 84                 | 45           | 0.12           | —                       |
| Residual              | 247,778        | 640                | 387          |                |                         |

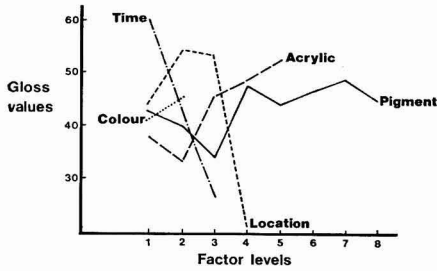


Fig. 3. Main factor fluctuations

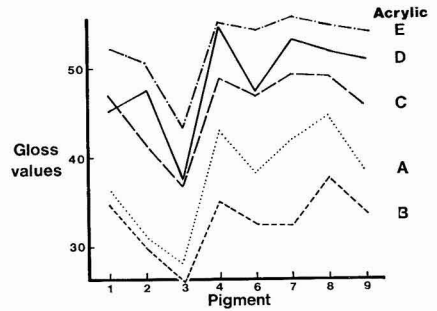


Fig. 7. Pigment/acrylic interaction

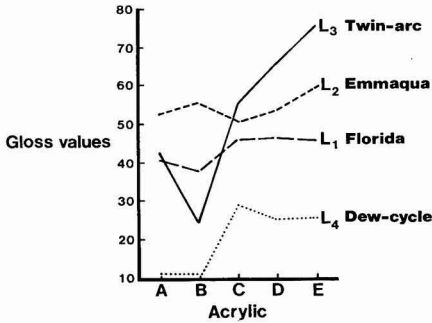


Fig. 4. Location/acrylic interaction

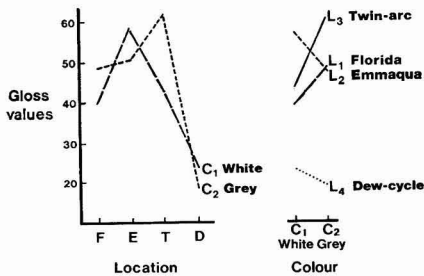


Fig. 5. Location/colour interaction

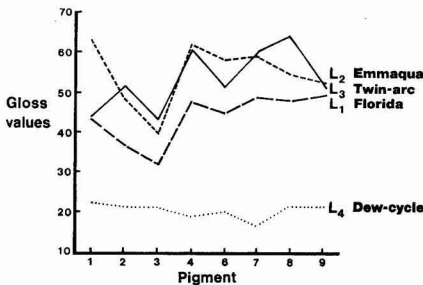


Fig. 6. Pigment/location interaction

Before considering the re-analysis, it is of interest to note the pigment type/acrylic interaction (Fig. 7), which proved insignificant, confirming the earlier Spearman coefficient analysis.

The new table of variance (Table 4) shows how the significance of location has been greatly reduced, indicating much better agreement between Florida, "Emmaqua" and the twin arc than between the Dew cycle and any other location.

Table 4  
Table of variance 3

| Factor or interaction | Sum of squares | Degrees of freedom | Mean squares | Variance ratio | Significance (per cent) |
|-----------------------|----------------|--------------------|--------------|----------------|-------------------------|
| P ..                  | 23,317         | 7                  | 3,331        | 7.94           | 0.1                     |
| L ..                  | 16,248         | 2                  | 8,124        | 19.36          | 0.1                     |
| PL ..                 | 9,626          | 14                 | 688          | 1.64           | —                       |
| C ..                  | 7,019          | 1                  | 7,019        | 16.73          | 0.1                     |
| PC/ ..                | 4,150          | 7                  | 593          | 1.41           | —                       |
| LC ..                 | 18,445         | 2                  | 9,222        | 21.98          | 0.1                     |
| PLC ..                | 2,090          | 14                 | 149          | 0.36           | —                       |
| A ..                  | 42,856         | 4                  | 10,714       | 25.53          | 0.1                     |
| PA ..                 | 3,057          | 28                 | 109          | 0.26           | —                       |
| LA ..                 | 50,104         | 8                  | 6,263        | 14.92          | 0.1                     |
| PLA ..                | 5,440          | 56                 | 97           | 0.23           | —                       |
| CA ..                 | 2,871          | 4                  | 718          | 1.71           | —                       |
| PCA ..                | 1,055          | 28                 | 38           | 0.09           | —                       |
| LCA ..                | 4,063          | 8                  | 508          | 1.21           | —                       |
| PLCA ..               | 2,672          | 56                 | 48           | 0.11           | —                       |
| Residual              | 201,433        | 480                | 420          | —              | —                       |

Fig. 8 shows the values for the main factors, pigment type, location, pigment content and acrylic resin, all of which are highly significant. The Dew cycle results, processed without the values of the other locations (Table 5) show that the only factor of real significance is the choice of the acrylic resin. The analysis confirms that the Atlas weatherometer did not discriminate between pigment types; this is shown in Fig. 9 in comparison with the results for the other three locations and those for all four locations.

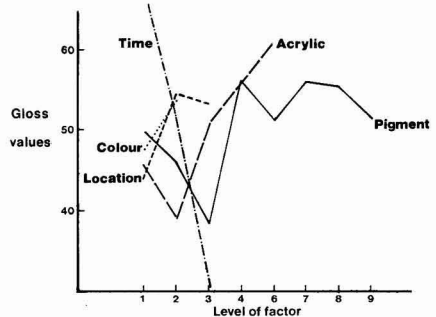


Fig. 8. Main factor fluctuations



Table 5  
Table of variance 4

| Factor or interaction | Sum of squares | Degrees of freedom | Mean squares | Variance ratio | Significance (per cent) |
|-----------------------|----------------|--------------------|--------------|----------------|-------------------------|
| P ..                  | 1,030          | 7                  | 147          | 0.51           | —                       |
| C ..                  | 1,197          | 1                  | 1,197        | 4.13           | 5.0                     |
| PC ..                 | 780            | 7                  | 111          | 0.38           | —                       |
| A ..                  | 11,113         | 4                  | 2,778        | 9.59           | 0.1                     |
| PA ..                 | 947            | 28                 | 34           | 0.12           | —                       |
| CA ..                 | 2,902          | 4                  | 725          | 2.50           | 5.0                     |
| PCA ..                | 1,175          | 28                 | 42           | 0.14           | —                       |
| Residual              | 46,346         | 160                | 290          |                |                         |

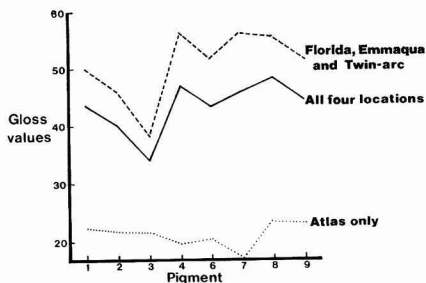


Fig. 9. Pigment/location variations

The disappointing results obtained with the Dew cycle tests were suspected of having an adverse influence on the analysis of the other data, as has been demonstrated in the earlier tables of variance (Tables 4 and 5). These earlier analyses also suggest some anomalies with respect to the pigment content, so a new expanded set of results was programmed for analysis with these factors omitted. The new analysis examined all nine pigments (1-9) in the five acrylic resins (A-E), but now at only three locations and using six results for each sample. As in earlier analyses the time factor is again by far the most significant, and in the final analysis of variance (Table 6) this has been included in the residual. The main factors, pigment type, location and the acrylic resin are all highly significant. (Fig. 10).

The pigment type/acrylic interaction (Fig. 11) was still not significant, suggesting that pigment performance is not,

generally, specific to a particular type of acrylic resin. The pigment type/location interaction (Fig. 12) was also not significant suggesting a wide area of agreement between Florida, "Emmaqua" and the twin arc as methods of assessing relative pigment performance. Fig. 13 shows how the discrimination between pigments develops with further film deterioration.

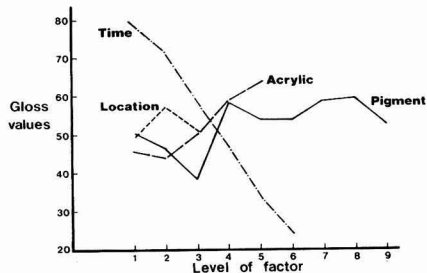


Fig. 10. Main factor variations

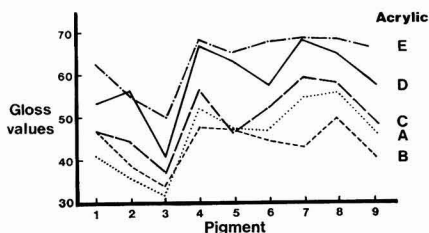


Fig. 11. Pigment/acrylic interaction

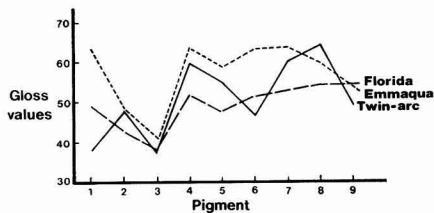


Fig. 12. Pigment/location interaction

Table 6  
Table of variance 5

| Factor or interaction | Sum of squares | Degrees of freedom | Mean squares | Variance ratio | Variance averaged over time | Significance (per cent) |
|-----------------------|----------------|--------------------|--------------|----------------|-----------------------------|-------------------------|
| P ..                  | 32,265         | 8                  | 4,033        | 106.29         | 7.31                        | 0.1                     |
| L ..                  | 10,090         | 2                  | 5,045        | 132.95         | 9.14                        | 0.1                     |
| PL ..                 | 12,097         | 16                 | 756          | 19.92          | 1.37                        | —                       |
| A ..                  | 47,206         | 4                  | 11,802       | 311.03         | 21.38                       | 0.1                     |
| PA ..                 | 5,073          | 32                 | 159          | 4.17           | 0.29                        | —                       |
| LA ..                 | 52,347         | 8                  | 6,543        | 172.45         | 11.86                       | 0.1                     |
| PLA ..                | 9,697          | 64                 | 152          | 3.99           | 0.27                        | —                       |
| T ..                  | 500,940        | 5                  | 60,188       | 1586.26        | Residual sum                | —                       |
| PT ..                 | 7,615          | 40                 | 190          | 5.01           | Residual sum                | —                       |
| LT ..                 | 17,934         | 10                 | 1,793        | 47.26          | Residual sum                | —                       |
| PLT ..                | 7,680          | 80                 | 96           | 2.53           | Residual sum                | —                       |
| AT ..                 | 7,215          | 20                 | 360          | 9.50           | Residual sum                | —                       |
| PAT ..                | 6,693          | 160                | 42           | 1.10           | Residual sum                | —                       |
| LA ..                 | 12,339         | 40                 | 308          | 8.12           | Residual sum                | —                       |
| PLAT ..               | 12,142         | 320                | 38           | 1.00           | Residual sum                | —                       |

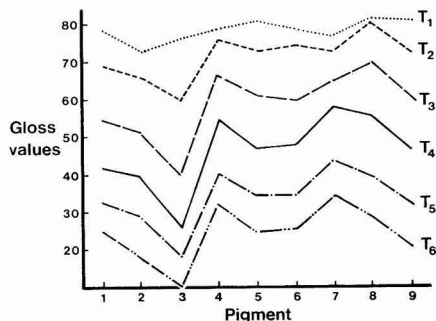


Fig. 13. Pigment/time interaction

The location/acrylic resin interaction was highly significant. Fig. 14 shows that there was considerable discrimination between the acrylic resins during exposure in the twin arc apparatus.

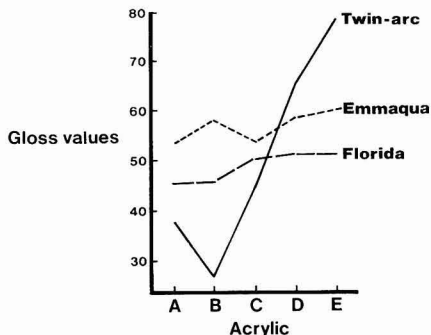


Fig. 14. Location/acrylic interaction

The location/time interaction illustrated in Fig. 15 shows a fairly regular rate of deterioration at all locations beyond a certain point in time, but indicates a possible energy threshold value before decay becomes pronounced on "Emmaqua" and possibly also in Florida.

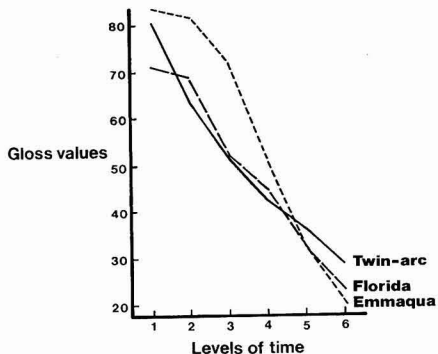


Fig. 15. Location/time interaction

The interaction time/acrylic resin (Fig. 16) illustrates the relative decay rates of the polymers. Reference to Fig. 14

suggests that the difference between the acrylic resins may be due almost entirely to the influence of the results obtained in the twin arc apparatus.

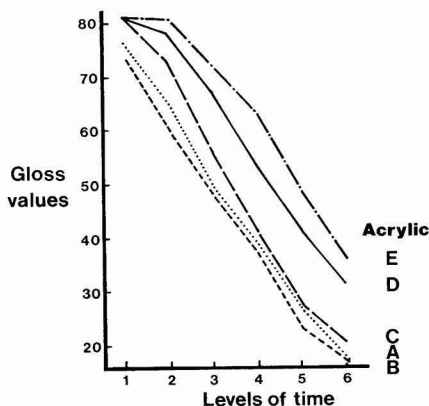


Fig. 16. Time/acrylic interaction

It is obvious from the earlier analyses that the time factor plays a major role and a decision was taken to reprocess the results once again, so that they would be expressed in terms of a decay cycle rather than upon an intuitive time factor. Taking the initial gloss value before exposure of each sample as 100, time intervals were determined for the reduction of the gloss levels to 80 per cent, 60 per cent and then 40 per cent of the initial value. The range of the initial gloss values was relatively small and the differences were assumed to be insignificant in relation to the overall deterioration.

Each location was assessed on a different time scale and, for mathematical convenience, it was desirable that all should be of a similar order of magnitude. The units were converted, therefore, by using suitable factors. The analysed results were expressed in tenths of a month for Florida exposure; units of 5,000 Langley's on "Emmaqua;" tens of hours for the twin arc, and tenths of hours for the Dew cycle.

The four factors involved in the new analysis were the pigments (1-9); the acrylic resins (A-E); the locations (Florida, "Emmaqua," twin arc and the Dew cycle) and the time to a given decay level, of which there were three values.

The greatest sources of variance (Table 7) were time, location, acrylic resin, pigment type, rate of decay at varying locations (the T/L interaction) and the acrylic/location interaction. The pigment type/location reaction is much less significant than in the earlier analyses and reference to the graph of this interaction (Fig. 18) will show that the Atlas is much more discriminating between pigments when the assessment is on the basis of the decay period.

The significant interactions were plotted and the main factors are shown in Fig. 17. The pigment/location interaction shows that "Emmaqua" gives the closest correlation with Florida exposure (Fig. 18), although the twin arc weatherometer is also reasonably close. The obvious exceptions in the "Emmaqua" results are the relative displacement of P7, P8 and P9 compared with the other pigment samples. With the

twin arc pigments P1, P6 and P9 are anomalous, showing markedly inferior performance compared with the Florida results.

Table 7  
Table of variance 6

| Factor or interaction | Sum of squares | Degrees of freedom | Mean squares | Variance ratio |
|-----------------------|----------------|--------------------|--------------|----------------|
| P .. .. .             | 133,236        | 8                  | 16,655       | 69.10          |
| T .. .. .             | 1,127,755      | 2                  | 563,878      | 2339.54        |
| PT .. .. .            | 13,901         | 16                 | 869          | 3.60           |
| L .. .. .             | 557,259        | 3                  | 185,753      | 770.69         |
| PL .. .. .            | 145,241        | 24                 | 6,052        | 25.10          |
| TL .. .. .            | 394,985        | 6                  | 65,831       | 273.13         |
| PTL .. .. .           | 34,518         | 48                 | 719          | 2.98           |
| A .. .. .             | 153,225        | 4                  | 38,306       | 158.93         |
| PA .. .. .            | 50,833         | 32                 | 1,589        | 6.59           |
| TA .. .. .            | 12,653         | 8                  | 1,582        | 6.56           |
| PTA .. .. .           | 14,021         | 64                 | 219          | 0.90           |
| LA .. .. .            | 421,204        | 12                 | 35,100       | 145.63         |
| PLA .. .. .           | 170,911        | 96                 | 1,780        | 7.38           |
| TLA .. .. .           | 59,829         | 24                 | 2,493        | 10.34          |
| PTLA .. .. .          | 46,276         | 192                | 241          | 1.00           |

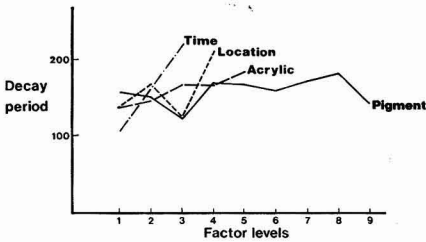


Fig. 17. Correlation via half life

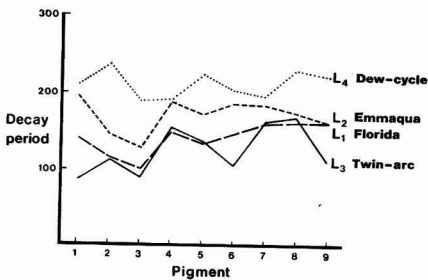


Fig. 18. Pigment/location interaction via half life

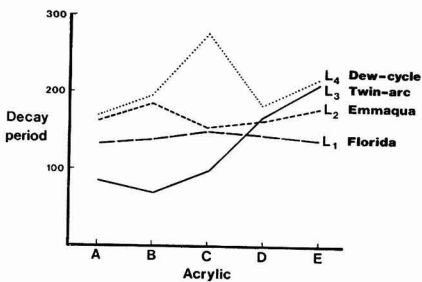


Fig. 19. Location/acrylic interaction via half life

“Emmaqua” gives better correlation with Florida, showing lack of discrimination between acrylic resins (Fig. 19), whilst the Dew cycle and the twin arc suggest a considerable variation in durability.

The location/time interaction shows that correlation is not so good between “Emmaqua” and Florida in terms of the rate of decay (Fig. 20) and if the decay values are converted to show the rate of loss gloss at each location (Fig. 21) there is a close correlation between twin arc testing and Florida. The decay pattern on “Emmaqua” testing, although showing less satisfactory correlation, is better than with the Dew cycle, and the earlier correlations with respect to both relative pigment and acrylic resin performance are factors to be considered when selecting a rapid test method for durable coatings.

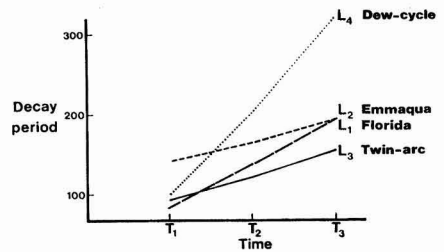


Fig. 20. Time/location interaction via half life

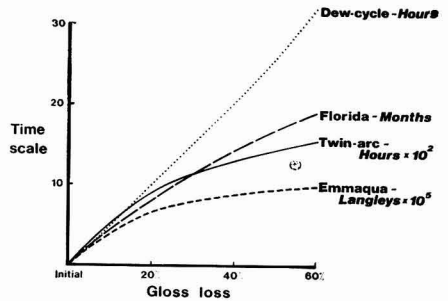


Fig. 21. Loss of gloss versus location

**Conclusion**

It is suggested that the twin arc weatherometer as used in this work gives a high degree of correlation with respect to the relative performance of pigments, but is likely to be much less satisfactory for assessing the durability of polymers, even where these are very similar in composition.

The Dew-cycle method, although the most rapid means of promoting film degradation, has been shown to be extremely unreliable as a means of evaluating polymer performance. Considerable care must be exercised in interpreting the loss of gloss results when assessing pigment performance. It is suggested that the results should be interpreted on a half-life

decay basis to achieve discrimination between pigments.

In conclusion, it is considered that the results of these tests show that the rapid testing of durable coatings on the "Emmaqua" apparatus, using natural sunlight as the source of radiant energy, is most likely to achieve a high level of correlation with conventional weathering tests carried out in the Florida environment.

### **Acknowledgment**

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# A study of microfouling on antifouling coatings using electron microscopy

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## Summary

Scanning electron microscopy has shown that diatoms are the predominant microfouling organisms on a number of antifouling coating specimens from ships and test panels. Surface deposits of amorphous material, attributed to bacteria, have been infrequently observed. Transmission electron microscopy has shown that relatively few species of small, pennate diatoms are present, predominantly of the genus *Amphora*.

Diatoms alter the surface topography and coating structure in

such a way that both the frictional resistance and the service life of the coating could be affected. The adhesion of paint applied over diatom layers is adversely affected. The effects of both diatomaceous and bacterial slimes upon the toxic release rate are discussed.

Coating composition and the amount of light reaching the coating surface are considered to be major factors affecting the growth of microfouling organisms on antifouling coatings.

## Keywords

*Types and classes of coating and allied products*  
antifouling coating

*Raw materials: biologically active agents*  
copper salt

*Processes and methods primarily associated with analysis, measurement or testing*  
electron microscopy

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

## Une étude à l'aide du microscope électronique sur la microsalissure de revêtements "anti fouling"

### Résumé

Au moyens de la microscopie électronique à balayage, on a montré que les diatomées sont les organismes prédominants dans la formation de microsalissures sur une gamme de spécimens de revêtements "anti fouling" à partir de navires ou de panneaux d'essai. On a noté inféquentement les dépôts de matière amorphe, que l'on attribue aux bactéries. La microscopie électronique par transmission a montré la présence de relativement peu d'espèces de petites diatomées pennées, parmi lesquelles prédomine le genre *Amphora*.

Les diatomées provoquent un changement de la topographie superficielle et de la structure du revêtement de sorte que la

résistance frictionnelle et la durée utile puissent être altérées. Les couches de diatomées exercent une influence défavorable sur l'adhérence des peintures appliquées au dessus d'eux. On discute les effets qu'exercent les limons de diatomées ou de bactéries sur le taux de dégagement des agents toxiques.

On considère que la composition du revêtement et la quantité de lumière qui atteint la surface du revêtement ce sont les facteurs les plus importants en ce qui concerne la croissance des organismes qui provoquent les microsalissures sur les revêtements "anti fouling".

## Eine Elektronenmikroskopische Untersuchung von Mikrofouling auf Antifoulinganstrichen

### Zusammenfassung

Mit dem Abtastelektronenmikroskop wurde an zah-reichen von Schiffen und Testtafeln herrührenden Antifouling-Prüfmustern festgestellt, dass Diatomeen die hauptsächlichsten Mikrofouling-organismen sind. Mitunter wurde oberflächlicher Anwuchs deren Ursprung amorpher Stoffe beobachtet, Bakterien zugeschrieben wurden. Transmissionselektronenmikroskopie erwies die Anwesenheit von verhältnismässig wenig Spezies kleiner, geflügelter Diatomeen, vorwiegend vom Genus *Amphora*.

Diatomeen verändern die Oberflächentopographie und Anstrichstruktur derart, dass sowohl Reibungswiderstand als auch

Lebensdauer der Beschichtung betroffen werden können. Die Haftung von über Diatomeenschichten angewandten Anstrichmaterialien wird beeinträchtigt. Die Wirkung sowohl von Diatomeen—als auch Bakterienschleimen auf die Geschwindigkeit des Giftauslaugens wird besprochen.

Als wesentliche Faktoren, welche das Wachstum von Mikrofoilingorganismen auf Antifouling beeinflussten, werden die Zusammensetzung des Anstrichmittels und die seine Oberfläche erreichende Lichtmenge betrachtet.

## Анализ микро-обрастания в необрастающих покрытиях, применяя электронную микроскопию

### Резюме

Развертывающая электронная микроскопия показала что диатомы являются преобладающими микроорганизмами обрастания в ряде образцов необрастающих покрытий на судах и испытательных панелях. Иногда наблюдались поверхностные отложения аморфных материалов приписываемые бактериям. Передаточная электронная микроскопия показала что присутствует сравнительно мало перистых диатомов, преимущественно породы *Амфора*.

Диатомы изменяют поверхностную топографию и структуру

покрытия таким образом, что как сопротивление трения так и долговечность покрытия могут подвергаться воздействию. Адгезия краски нанесенной над слоями диатомов подвергается неблагоприятному влиянию. Обсуждается влияние диатомной и бактериальной слизи на скорость токсического выделения.

Авторы считают что состав покрытия и количество света проникающего до поверхности покрытия, являются главными факторами влияющими на рост обрастающих микроорганизмов в необрастающих покрытиях.



## Introduction

Ref: 1-20

Bacteria, diatoms and other micro-organisms form a slime film on surfaces immersed in seawater<sup>1-3</sup>. Some of these microfouling organisms are very resistant to copper poisoning,<sup>3-7</sup> and settle on cuprous oxide based antifouling coatings within a few hours of immersion.<sup>3, 8, 9</sup>

Although the effect of microfouling upon the performance of a ship is small compared to that of larger organisms,<sup>3, 10, 11</sup> the frictional resistance of the ship is affected appreciably by the presence<sup>11</sup> of the former. The slime film can, therefore, alter the boundary layer thickness and flow pattern of the seawater adjacent to the ship's hull, and thus affect the rate of release of toxic material from the underlying antifouling coating.<sup>12</sup> In addition, studies<sup>13, 14</sup> have shown that large numbers of diatoms alter the coating's microstructure, which is another factor controlling the rate of release of toxic material from antifouling coatings.<sup>12</sup>

Recent reviews<sup>2, 15-18</sup> show that little work has been carried out since the second world war on the toxic-resistant slime film. Studies<sup>5, 18-20</sup> on the effect of slime on the rate of release of toxic material indicated that bacterial action might be significant but, generally, these were inconclusive. In view of this lack of data, microfouling on antifouling coatings was further examined by electron microscopy to assess its effect upon the performance of antifouling paints.

## Experimental

Ref: 14, 21-25

### Antifouling paint specimens

Samples were taken from the underwater hulls of Australian naval vessels at Williamstown Naval Dockyard, Melbourne, during the period 1967-71. The oleoresinous antifouling paint used contained approximately 75 per cent by weight of cuprous oxide. The coatings had been in service for periods ranging from six to eighteen months and little fouling had occurred.

In addition, test panels coated with a laboratory-formulated antifouling paint (90 per cent by weight of cuprous oxide in a polyvinylchloride matrix, VYHH grade) were exposed in running seawater for three months at Williamstown Naval Dockyard. For comparison, panels coated with a non-toxic paint (consisting of polyvinylchloride pigmented with sodium chloride) were examined after exposure under similar conditions.

### Preparation of specimens for scanning electron microscope

Specimens were prepared for scanning electron microscopy as previously described.<sup>14</sup> Cross-sections were obtained by fracturing the flakes of brittle paint, but the more flexible experimental vinylite-matrix coatings were fractured after cooling in liquid nitrogen.<sup>21</sup>

### Specimen preparation for the identification of diatoms by transmission electron microscopy

Flakes of antifouling paint, shown by scanning electron microscopy to be populated with strongly silicified diatoms, were boiled with tetrahydrofurane. The remaining solids,

consisting of diatomaceous material, paint pigment and small amounts of paint matrix material, were washed with distilled water and warmed with 2M hydrochloric acid to dissolve the paint pigment. The remaining undissolved material was boiled with concentrated hydrochloric acid, and then with concentrated sulfuric acid to which sodium nitrate was added cautiously until the black coloration was completely dispelled. After washing, the cleaned diatom frustules were transferred in aqueous suspension to a thin carbon film supported on a copper grid, then dried and examined in the transmission electron microscope. This cleaning procedure was based on that of Hendey.<sup>22, 23</sup>

The diatoms were identified by comparison with previously published transmission electron micrographs of the different species.<sup>24, 25</sup>

## Discussion

### Composition of the slime film

Ref: 1-3, 13, 14, 22, 26-34

Diatoms were invariably the predominant form of microfouling on the specimens of antifouling coating from Australian naval vessels. Large numbers of diatoms impart a greyish-green surface coloration to this particular antifouling coating<sup>13, 14, 26</sup>. Reactions between some metallic ions and porphyrin-based pigments (such as those present in diatoms<sup>27</sup>) yielding coloured compounds are well known, divalent copper being the most rapidly reacting ion<sup>28</sup>. The distribution of this greenish coloration showed that high population densities of diatoms were confined to sunlit areas of the hull and were not present on areas such as the undersides of bilge keels where few diatoms were found.

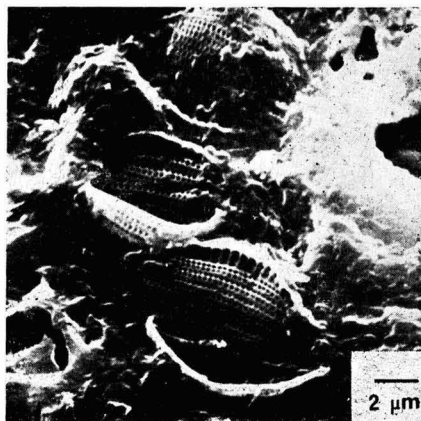


Fig. 1. Scanning electron micrograph showing diatoms in association with amorphous material on an antifouling coating surface

Bacteria are the other predominant form of micro-organisms found in the slime on antifouling coatings<sup>1, 2</sup>. Amorphous surface layers, with the cream colour typical of some bacterial slimes<sup>3</sup>, were observed on several test panels on which no diatoms were present. In a few instances, diatoms on specimens of antifouling coatings from ships were associated with appreciable amounts of similar amorphous material, as shown in Fig. 1. The same species of diatoms were observed on antifouling coatings with the same formulation

in the presence of little or no amorphous material. Hence, these amorphous deposits were considered to be of bacterial rather than diatomaceous origin.

Amorphous surface deposits were not found in the cavities left by dissolution of the cuprous oxide pigment and other soluble coating components. However, both the amorphous material and diatoms penetrated the larger crevices in the coating. Fig. 2 shows one side of an existing crack exposed by fracturing the coating specimen. The edge of the layer of amorphous material can be seen at the bottom of the crack.

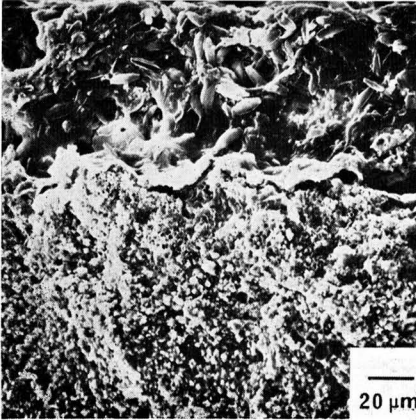


Fig. 2. Scanning electron micrograph showing a cross-section through an antifouling coating along the line of an existing crack

In contrast to this observed predominance of diatoms, Hendey<sup>3</sup> noted a seasonal variation (from bacterial to diatomaceous) in the composition of slime on antifouling coatings exposed in Chichester Harbour. This difference can be explained by relating the presence of diatoms to the amount of light available (one of the main factors, together with temperature and the availability of nutrients<sup>29</sup>, controlling diatom growth).

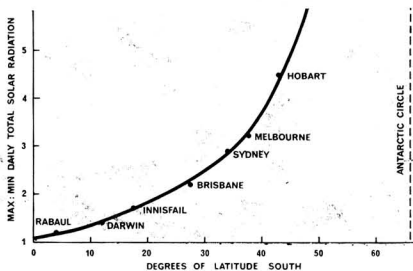


Fig. 3. Ratio between annual maximum and minimum daily total solar radiation falling on horizontal surfaces plotted against latitude

Fig. 3, based upon the figures of Southwell and Ballantyne<sup>30</sup>, shows the variation with latitude of the ratio of the annual maximum and the daily minimum total solar radiation falling on a horizontal surface (such as that of the sea) at a number of Australasian sites. This variation with latitude is more pronounced than the accompanying temperature changes. It would be surprising if the large annual variation in light intensity at Chichester (51°N latitude) did not have

a considerably greater effect than the much smaller variations encountered in Australian waters (mostly north of 38°S).

This hypothesis is supported by direct observations. Greater seasonal fluctuations in diatom population occur at Chichester<sup>3</sup> and Caernarvon, in the UK (53°N)<sup>31</sup>, than at Sydney (34°S)<sup>32</sup> and Mar del Plata (38°S)<sup>33</sup>. Further support is given by the annual changes in weight and composition of slime<sup>1</sup>.

Thus, the available evidence suggests that diatoms will grow throughout the year between approximately latitudes 40°N and 40°S on sunlit areas of antifouling coatings. At latitudes closer to the poles, slime composition should vary with season, and bacteria will increase in relative importance as the amount of available light decreases. This hypothesis would, at least in part, explain why diatom deposits have been observed on tankers travelling between the Persian Gulf and Europe via Capetown, but not on those using other routes<sup>34</sup>. However, more information is needed on the effect of environmental factors on toxic resistant micro-organisms, because many other factors, such as water turbidity and temperature, affect the population dynamics of bacteria and diatoms<sup>3, 22, 29</sup>.

#### Effect of non-toxic coating materials on slime composition

Ref: 3, 7, 14, 15, 20, 21, 35-38

Greenish-coloured surface deposits occur only on insoluble-matrix antifouling coatings<sup>13</sup>; that is, on those coatings containing sufficient insoluble matrix material to form a stable structure after elution of the soluble components. The continuously eroding surface of a soluble-matrix coating would prevent the formation of permanent slime films or other surface deposits. The repeated slime formation and stripping observed on some coatings, particularly those containing a rosin-wax mixture<sup>7</sup>, demonstrates how the dissolution and biodegradation of paint coating constituents can prevent permanent microfouling. Water speeds as high as 40 knots do not appreciably erode insoluble-matrix antifouling coatings<sup>35, 36</sup>.

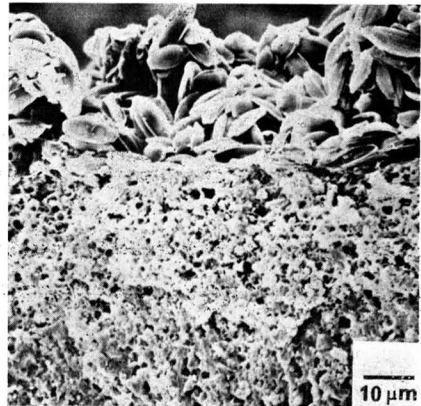


Fig. 4. Scanning electron micrograph of cross-section through an antifouling coating, showing leached layer and heavy settlement of diatoms on surface

In Fig. 4, the sponge-like leached layer typical of insoluble matrix coatings<sup>14, 21</sup> can be seen directly under the surface

layer of diatoms. Thus, this coating is of the insoluble-matrix type although it contains a substantial amount of soluble rosin.

The diatom layers on the specimens examined were firmly bonded to the underlying coating and attempts to remove the slime by scraping resulted in removal of the leached surface layer of the coating too. Examination of specimens from a destroyer, docked after six years at moorings, showed that successive coats of vinylite-based, cuprous oxide antifouling paint had been applied over thick layers of diatoms. The continued presence of these coats of paint showed that the diatoms were firmly bound both to each other and to adjacent paint layers. However, adhesion failure in the dried-out coating system occurred at these interfaces when the ship was finally docked, indicating that this bonding should not be relied upon. Reports that diatom slimes were easily washed off<sup>7</sup> are in apparent conflict with these observations and with reports that the growth of diatomaceous slime occurred on rotor surfaces moving at speeds of up to 11.2 knots<sup>37</sup>. However, this conflict can be resolved by postulating that the permanence of diatomous limes depends upon the diatomaceous species present. Soluble and biodegradable matrix materials in antifouling coatings favour the growth of bacteria and diatoms<sup>3, 20</sup>. The diatoms probably benefit indirectly from the bacteria, although some diatoms are capable of heterotrophic growth<sup>38</sup>. Rosin and other such materials, therefore, stimulate the growth of microfouling.

#### Effect of toxic coating materials on slime composition

Ref: 1-4, 6, 7, 22-24, 26, 29, 32, 38-46

There are more than ten thousand species of diatoms<sup>29</sup>, and even more types of bacteria, adapted to a wide range of environmental conditions. Over seven hundred marine diatomous species are found in British coastal waters<sup>39</sup>. Micro-organisms are vastly more diversified biochemically than are the more complex organisms, and they can mutate more rapidly to cope with new conditions<sup>40</sup>. Thus, it is not surprising that some species of diatoms and bacteria are sufficiently resistant to copper poisoning to be the principal micro-organisms forming slime films on the surfaces of active, cuprous oxide based antifouling coatings<sup>1-3, 7, 41</sup>.

Available information<sup>4, 6, 42</sup> suggests that the optimum rate of release of copper from cuprous oxide based antifouling coatings would have to be increased by an order of magnitude to prevent diatomaceous microfouling and by two orders of magnitude to prevent bacterial microfouling. The growth of diatoms is unlikely to be stimulated by high copper concentrations<sup>38</sup>, but their growth is favoured by the absence of competing organisms<sup>3, 7, 32</sup>.

Toxin-resistant bacteria and diatoms can be expected to colonise insoluble-matrix coatings containing other toxic materials, unless the toxic release rates necessary to prevent macrofouling are high enough to prevent such colonisation. For example, diatoms colonised a number of coatings containing tributyltin compounds<sup>43</sup> and a concentration of pentachlorophenol high enough to prevent macrofouling did not prevent slime formation<sup>44</sup>. The existence of copper-resistant strains of *Ectocarpus siliculosus*<sup>45</sup> demonstrates that strains of micro-organisms resistant to new toxic materials could also develop over a period of time.

Relatively few species of small, pennate diatoms were found on the antifouling coating specimens examined, but these species were often present in large numbers, as shown in

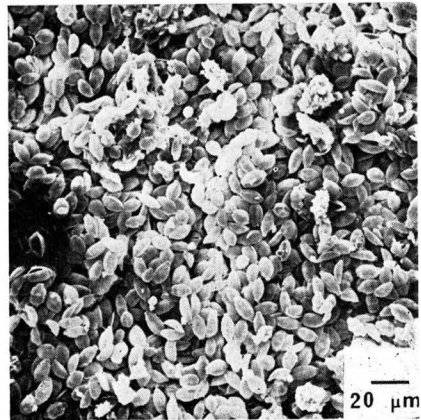


Fig. 5. Scanning electron micrograph of antifouling coating surface, showing presence of large numbers of pennate diatoms

Figs. 4 and 5. The diatom species present were identified using transmission electron microscopy, which has been used widely for diatom classification<sup>22-24</sup>. Transmission electron microscopy also showed, better than scanning electron microscopy, some aspects of frustule structure, such as the holes in the cell wall, but there are difficulties in interpretation<sup>23</sup>. Scanning electron microscopy gave a clearer idea of the general structure of diatoms and their arrangement on the coating surface<sup>46</sup>. The numerical predominance of the genus *Amphora* on antifouling coating specimens from ships<sup>26</sup> is in accordance with the observations of other workers<sup>3, 32</sup>.

A number of *Foraminifera*, such as the specimen shown in Fig. 6, were observed on the surface of the non-toxic underwater coating used on the hull of a ship permanently moored at Melbourne. No traces of *Foraminifera* or other protozoa were detected on antifouling coating specimens. Diatom species other than those found on antifouling coatings were observed on non-toxic control panels exposed in constantly running seawater. Thus, the effect of toxic coatings on slime composition was demonstrated.

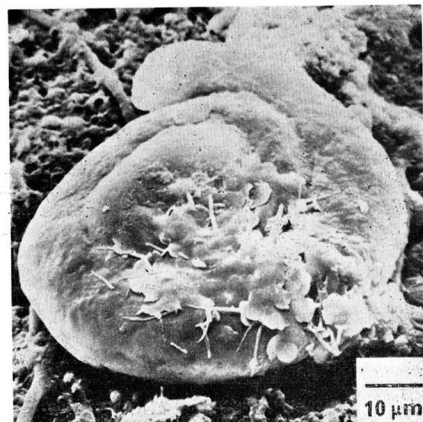


Fig. 6. Foraminifera on the surface of a non-toxic coating removed from an underwater hull

### Chemical and physical effects of microfouling on antifouling coating performance

Ref: 2, 3, 8, 9, 12, 15, 23, 34, 43, 47-52

Bacterial slimes should have a lower pH than the surrounding seawater due to the continuous production of carbon dioxide, one of the main factors controlling pH<sup>47</sup>. A lower pH, together with the bacterial degradation of coating constituents, considerably increases the rate of release of cuprous oxide or organo-tin derivatives from coatings containing these materials, whose solubilities increase markedly with decreasing pH<sup>3, 43</sup>. Marine fungi, known to degrade some coating constituents<sup>2, 43</sup>, would have a similar effect. In contrast, the pH of diatomaceous slime should vary diurnally, the carbon dioxide concentration being reduced by photosynthesis and increased by nocturnal respiration. Therefore, since diatoms are also unlikely to cause substantial biodegradation<sup>3</sup>, diatomaceous slime will have little chemical effect upon the rate of release of toxic material from an antifouling coating.

Continuous surface layers, composed of large numbers of diatoms and varying in thickness between 2 and 30 $\mu$ m, were found on antifouling coatings. These layers formed an appreciable barrier to the diffusion of toxic material from the coating. The rapid growth of such layers can occur within a week of immersion<sup>8, 9</sup> and will reduce the wastefully high initial release rate of toxic material from insoluble-matrix coatings. Diatom deposits up to 500 $\mu$ m in thickness have been observed on several tankers<sup>34</sup>.

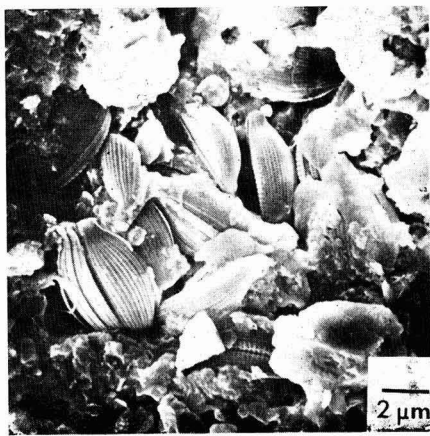


Fig. 7. Scanning electron micrograph showing diatoms (*Amphora* species) colonising surface crevices in an antifouling coating

Figs. 2 and 7 illustrate the colonisation by diatoms of the larger cracks and crevices in the coating surface. Diatoms were found more than 70 $\mu$ m below the coating surface in cracks, such as that shown in Fig. 2. Quite small numbers of diatoms can, therefore, appreciably hinder the diffusion of dissolved toxic material. Fig. 8 shows an example of clusters, with radii of up to 30 $\mu$ m, formed by groups of diatoms. Because these clusters exceed 7.6 $\mu$ m in diameter<sup>49</sup>, the frictional resistance of ships will be affected by their presence. An associated decrease in seawater boundary layer thickness will increase the rate of release of soluble materials from the underlying coating<sup>12</sup>.

The extent to which diffusion is hindered by diatoms, and the rate and mode of growth of diatom colonies, will depend

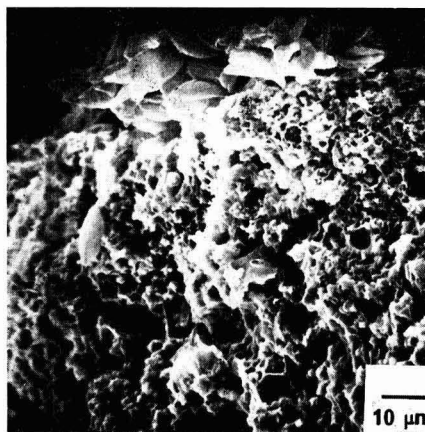


Fig. 8. Scanning electron micrograph of an antifouling coating cross-section, showing a cluster of diatoms projecting from the surface

upon the species involved. The shapes of the diatoms will determine their packing characteristics and affect the toxic diffusion rate through the diatom layer. Their internal structure will determine the diffusion rate through the diatoms themselves. Holes in the diatom frustule can account for 10 to 30 per cent of its total area and the frustule may have either a locular or lamellar structure<sup>23</sup>. As shown in Fig. 2, the amorphous material sometimes forms a continuous layer over the coating surface. Marine bacteria are known to form such films<sup>30</sup>, which will physically impede the diffusion of toxic material from coatings.

Insoluble-matrix coatings must be formulated to function with slime on their surfaces, because microfouling will occur on many such coatings. Coatings may otherwise fail because the toxic material has been either depleted too rapidly by bacterial action or released too slowly because diffusion has been hindered by diatoms. An antifouling coating must release toxic material at a sufficiently high rate to prevent macrofouling when large numbers of diatoms are present. Infestation by diatoms of such a coating would extend that coating's life by conserving its toxic component. Variations in coating performance, resulting from variations in the amount and composition of microfouling, would be minimised by avoiding the use of biodegradable materials in antifouling paint formulations as far as possible.

On very rare occasions, the scanning electron microscope showed the presence of crystalline salt deposits, but the precipitation of insoluble copper salts<sup>51</sup> played little or no part in the formation of the greenish surface layers observed on the antifouling coating used by the Royal Australian Navy. Van Londen's<sup>15, 52</sup> observations support this conclusion. However, a complex series of reversible reactions is involved in the formation of insoluble copper salts<sup>51</sup>, and a number of variables (basically temperature and concentrations of reactants) determine whether these salts are precipitated. Their formation cannot, therefore, be ruled out under other environmental conditions or when the coatings have a composition differing from that used by the Royal Australian Navy.

## Conclusions

The slime film on the antifouling coating used on Australian naval vessels consisted of small, pennate diatoms predominantly of the genus *Amphora*. Diatoms sometimes modified the surface topography sufficiently to affect the frictional resistance of the ship. Diatoms also presented a physical barrier to the diffusion of toxic material from the underlying antifouling coating, but it is considered that they will have little other effect on antifouling coating performance. Failure to remove diatom layers before adding further coats of paint increased the risk of adhesion failure in the paint system. The exact effect of diatoms on antifouling coating performance will depend upon the species present.

Diatoms were sometimes associated with a surface film of amorphous material which was attributed to bacteria, and was sometimes present when diatoms were absent. Bacteria may increase the rate of release of toxic material from the antifouling coating by lowering the pH of seawater at the coating surface and by biodegradation of coating materials. Surface deposits of inorganic salts were very rare.

Permanent microfouling cannot occur on soluble-matrix coatings. The amount and composition of microfouling on insoluble-matrix coatings depends upon coating composition and environmental factors. The amount of biodegradable material in antifouling coatings should be minimised to reduce the amount of surface slime present and its effect on the performance of the coating.

The amount and intensity of light reaching the coating surface is suggested as one of the major factors influencing slime composition. It appears that diatomaceous slimes will normally form on antifouling coatings immersed between approximately 40°N and 40°S latitudes with free access to sunlight. Slime composition should vary with the seasons at latitudes closer to the poles, the importance of bacteria increasing as light intensity decreases. Turbid waters would increase the likelihood of bacterial colonisation.

More information is needed on the interaction between marine micro-organisms and antifouling coatings, since it is clear that this relationship is of greater significance than is generally realised. In particular, there is a need for investigations into the relationships between slime composition and coating composition, between slime composition and the service lives of antifouling coatings, and between slime composition and frictional resistance.

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# Some physical properties of pigments and their importance in relation to dispersion in plastics

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## Summary

In common with all powder systems, pigment powders readily follow processes which allow them to reduce their surface energy. This is achieved by the close approach of the constituent particles resulting in the development of strength and structure within the

powder, and leads inexorably to agglomeration. The presence of agglomerates in pigmented plastics has until now been thought to arise mainly from the difficulty with which agglomerates are broken down. Attention is here centred upon avoiding their formation.

## Keywords

*Raw materials: prime pigments and dyes  
pigment dispersion*

*Process and methods primarily associated with  
manufacturing or synthesis  
agglomeration*

## Quelques caractéristiques de pigments et leur importance à l'égard de leur état de dispersion en matières plastiques

### Résumé

Comme toutes les poudres, celles des pigments s'assujettissent volontiers aux processus qui leur permettent de réduire leur énergie superficielle. Elles l'accomplissent en faisant s'approcher étroitement les particules constituantes qui provoque le développement de résistance et de structure dedans la poudre. Cela aboutit

inexorablement à la formation des agglomérés. Jusqu'à présent on a considéré que la présence des agglomérés en matières plastiques pigmentées se produit largement à cause de la difficulté de leur éliminer lors des procédés de confection. Dans cet exposé on vise l'attention sur la possibilité d'éviter la formation des agglomérés.

## Einige Physikalischen Eigenschaften von Pigmenten und deren Bedeutung für die Dispergierbarkeit in Kunststoffen

### Zusammenfassung

Wie alle Pulversysteme haben Pigmentpulver das Bestreben bereitwillig Vorgängen zu folgen, welche es ihnen ermöglichen, ihre Oberflächenenergie zu verkleinern. Dies wird durch enge Annäherung der konstituierenden Teilchen erreicht, wodurch innerhalb des Pulvers Festigkeit und Struktur entwickelt wird,

und unerbittlich Agglomerate entstehen. Bisher existierte die Ansicht, dass die Anwesenheit von Agglomeraten in pigmentierten Kunststoffmassen hauptsächlich durch die Schwierigkeit Agglomerate abzubauen verursacht wird. In dieser Abhandlung geht es um die Vermeidung solcher Gebilde.

## Introduction

*Ref: 1*

During recent years one common cause of dissension between pigment manufacturers and pigment users in the plastics industry has been the difference of opinion relating to dispersibility of a given pigment. This has been due to more than just a simple difference of viewpoint: it is historical. Pigment manufacturers have naturally attempted to approach the problems of the plastics industry with concepts better suited to paint and ink—earlier established markets which still consume much greater quantities of pigment. Only recently has it been realised that there are some very basic differences between these systems and those generally used for plastics compounding. They arise principally because of the absence of a liquid phase; for colouring plastics materials with pigments is, in the main, a powder mixing process, and remains so right up to the final stages of compounding. Only when the polymer finally softens, does the system even begin to behave like a liquid.

Pigments as normally supplied to the plastics industry are very fine dry powders which, in a variety of ways<sup>1</sup>, will agglomerate in transit and in use. Studies of the problems associated with the dispersion of pigments into plastics have, in the past, been associated with the removal of agglomerates. More recently, work aimed at evaluating some of the physical properties of pigment powders has been reported. The influence

of these powders upon dispersive mixing is discussed here, as is the dependency of their physical properties upon other more fundamental characteristics; and this leads to suggestions for ways in which the problem of agglomerates can be overcome simply by avoiding their formation.

## Pigment behaviour

*Ref: 2*

The behaviour of pigments in the fine powder form in which they are usually supplied to industry is difficult to predict, despite the fact that solid and fluid systems are themselves both comparatively simple. The solid state is open to description and test in reasonably precise and well understood ways; but, having broken up a solid into small particles, no two of which are identical, interposing solid and liquid interfaces, and surrounding the particles with a mobile gas so that pressure and shear within the system must be transmitted by inter-particle contact, it is possible to produce powder systems whose behaviour is of great variation and complexity<sup>2</sup>.

There is now abundant evidence to show that pigment agglomerates are normally produced under circumstances hitherto largely overlooked and unsuspected; and since it is largely true to say that, once formed, an agglomerate stands a reasonable chance of never meeting conditions which can

break it down, the production of good quality dispersions is made difficult or even impossible.

### The production of pigmented plastics

Ref: 3-11

There are three stages in the manufacture of pigmented plastics where it can be expected that agglomerates will be formed in the pigments used for their coloration: prior to use, during the powder mixing stage, and during the early stages of compounding.

#### Prior to use

The plastics manufacturer finds that even at the stage where he is weighing out his pigments, they will already be partially agglomerated. This may have arisen from the powder not being completely agglomerate-free when made, but is more likely to have arisen from a number of causes during handling and storage, either before purchase or after receipt in his works.

Most pigments are produced by precipitation, flocculation and filtration to produce a press paste containing about 30 per cent pigment. This is subjected to further processing to remove the water and obtain a fine powder, during which the pigment is dried, ground, treated with various materials (for example, to reduce dusting or improve dispersibility), and finally reground in a final attempt to produce an agglomerate-free powder.<sup>3-5</sup> This fine powder is the source of many subsequent difficulties, the most important of which is its tendency to agglomerate spontaneously; for in this way both the surface area and surface energy are reduced. It is natural for particles suspended in a mobile medium (air or liquid) to tend to settle under the influence of gravity. Unless stabilised electrostatically (as in a colloidal sol) or mechanically (as in a polymer matrix) the particles will move closer together until finally the forces of repulsion are in equilibrium with those of attraction. Under normal conditions the time taken for this to occur is quite long, but it is possible to detect changes in the tensile strength of a powder over several days. Whilst agglomeration will occur by perfectly natural processes during the storage of fine powders such as pigments, it is accelerated by the application of outside forces such as vibration and compaction.

Another factor which affects the tendency of a powder to agglomerate concerns the inclusion of impurities (whether intentional or otherwise) which lead to an increase in agglomeration. Failure to remove all manufacturing contaminants can lead to the formation of crystal bridges,<sup>6-8</sup> or if these are deliquescent, to the formation of liquid bridges.<sup>7-8</sup> The addition of other materials which are by design beneficial, can also result in increased agglomeration. For example, the addition of silicones<sup>9</sup> to prevent dusting, which is always a problem with fine powders, frequently has the immediate effect of increasing the number of coarse particles present as well as the tensile strength developed upon compaction.<sup>10</sup>

The initial production of pigment in powder form, and its subsequent grinding to a state of extreme fineness, causes it to become very bulky. For example, uncompacted titanium dioxide has been shown to occupy only one-sixth of its apparent volume;<sup>11</sup> and as this high voids content is an embarrassment in transporting the powder, various methods are adopted to reduce it. If the powder is to be "bagged off" it may be compressed before being packed. Alternatively, the powder may be sealed in a bag which is then compressed to

reduce its volume. For movement in bulk, a tanker may be driven round the factory before returning to be topped-up. All these practices have one objective, the reduction of volume. Unfortunately, they also increase the level of agglomeration in the powder which, whilst mild at this stage, can lead to the formation of much harder agglomerates after subsequent mixing operations.

These processes of compaction extend into subsequent stages of transport, storage and handling, which occurs before the powder is used by the plastics compounder. At various stages, the powder may be subjected to high local pressures, for example at the bottom of a pile of bags or at the bottom of a silo or hopper. As a powder of high voidage is incapable of supporting more than a small outside stress without undergoing compaction, this will readily occur. Furthermore, the high voidage allows plenty of scope for particle rearrangement and consequent close packing.

#### During powder mixing

When granules of pigments and plastics are tumble mixed to prepare a uniform premix and to distribute the pigment as evenly as possible, the weak agglomerates and flocculates already present may be broken down through inter-particle shear. Depending upon the texture and compressibility of the pigment, agglomeration may increase through compaction between adjacent granules and between granules and the mixer wall. The final level of dispersion achieved thus depends upon the overall balance between dispersion and agglomeration. As the size of the polymer granules is reduced, so the surface area of the plastic increases, and this reduces the likelihood of forming further agglomerates, but the kinetic energy of the polymer particles may then be reduced beyond the minimum which is required for disrupting the weak agglomerates already present. In this event, flocculates will remain in the mixture and will act as a primary source of agglomerates during the early stages of compounding.

The use of devices, such as impellers, to introduce higher mixing energies into the system when small particle size polymers are used, is not a complete solution since it only substitutes one agglomerating mechanism for another. Segregation of the mixture occurs readily in this type of mixer (particularly at high pigment concentration); and the high level of fluidisation produced allows the large flocculates to concentrate in that part of the mixture where there is the greatest free space. This is in the high velocity stream where high speeds have resulted in a reduced particle density. Unfortunately, this stream passes close to the mixer blades and the flocculates may be expected to impinge upon them. This may cause the particles to be dispersed; or they may be compacted directly into hard agglomerates; or they may contribute to the build-up which often occurs on the blades. Not only is this build-up a cause of pigment loss (it has been known to reach over 20 per cent by weight of the pigment present) but, if it breaks away from the blades during the mixing cycle, it becomes a secondary source of pigment agglomerates.

#### The early stages of compounding

When powder mixtures containing pigment are hot compounded into plastics there is always the chance of further agglomeration. This occurs, generally, before the polymer has softened, when pigment powder is trapped between two relatively hard surfaces, for example polymer/polymer or polymer/mixer, and in some cases even mixer/mixer, and is

then subjected to pressure. The likelihood of this occurring increases with increasing pigment concentration.

### Determination of pigment properties

Ref: 12

An overall assessment of the dispersion behaviour of a pigment may be quickly and easily obtained by tumble mixing it with polymer granules, compounding the mixture, and determining the final level of pigment dispersion by particle size analysis using an automatic particle counter.<sup>12</sup>

An alternative is the "tinting strength" method in which a pigment is compounded into a white pigmented polymer and the development of colour followed using a colorimeter; the more intense the colour, the better the dispersion. Comparisons are made with a well dispersed standard, not only to judge the depth of shade, but also to assess the presence of specks and streaks, which result from incomplete dispersion of the pigment. Such comparisons reveal three possible types of behaviour:

1. the dispersion of the pigment deteriorates with tumbling, or
2. its dispersion improves with tumbling, or
3. its dispersion is unaffected by tumbling.

The apparently poor dispersion behaviour of some pigments during tumble mixing can be improved considerably by the addition of a proportion of powdered polymer. The level of dispersion achieved appears to be related to the total available surface area, and involves a balance between agglomeration and dispersion. If the pigment flocculates are strong, agglomeration will predominate at low powder concentrations; if they are weak, dispersion will predominate at smaller excesses of granules. It is evident from this type of examination that investigations into the dispersive mixing of pigments and plastics must embrace more than just measurements of the final stage of dispersion. They should include an examination of the ability of the pigment to agglomerate, and a determination of the effect of powder mixing processes upon agglomeration levels. They should also include an examination of dispersive mixing in more detail in order to ascertain the strength of pigment agglomerates and their response to varying levels of shear.

### Powder mechanics

Ref: 13-16, 18-20

The elements of powder mechanics have long been known<sup>13</sup> but only recently, with the development of new materials and techniques, has the need to investigate these systems become pressing. These studies have developed considerably, and though they have centred in the main around the determination of flow patterns in bins and hoppers and rates of discharge through apertures, they have provided some information on static systems such as powder beds.

Activity has centred upon the determination of primary powder properties, such as cohesive strength and tensile strength. The fundamental equations from which these are derived are identical to those used in soil mechanics, but important differences exist; for example, some powders known from their behaviour to be cohesive, would, in soil mechanics be regarded as free flowing. This has necessitated the development of new test methods and specialised equipment.

Considerable advances were made by Jenike and Johanson<sup>14, 15</sup> with the development of a shear cell, and this has subsequently been improved by Walker.<sup>16</sup> The apparatus consists of a shallow cylindrical chamber which is split across a horizontal diameter. The bottom half of this cell is fixed, and a controlled stress applied to the top half. Powder is compacted into the cell, and the force necessary to fracture it along the horizontal plane determined by increasing the stress until failure occurs.

A series of increasing loads are applied to the lid of the cell; that is, at right angles to the surface of a number of uniformly packed powder samples. By this means it is possible to alter the failure characteristics of the powder bed. By plotting shear stress against the normal stress a curve is obtained which is known as the "yield locus". From this locus it is possible to determine both the cohesive strength and, with less accuracy, the tensile strength of the powder (Fig. 1). By repeating this procedure using powders with different packing densities, the way in which strength varies with compaction may be determined.

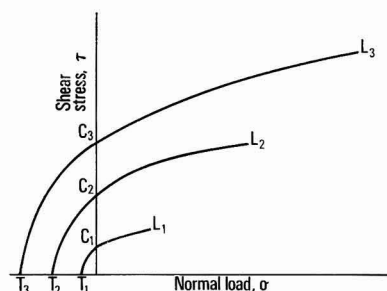


Fig. 1. Yield loci at various packing densities. Intercepts on the abscissa give tensile strength, those on the ordinate cohesive strength (Refs. 14-17)

The loci may also be obtained with the annular shear cell which has the advantage of avoiding the principal difficulty associated with this technique; namely, that of reproducing a given packing density in a number of powder samples. Recent work has established an improved method of extrapolation which involves calculations by computer. Extensions of this technique<sup>18</sup> permit the calculation of a "caking factor".

It is possible to measure the tensile strength of a powder with greater accuracy using a method which avoids extrapolation.<sup>19</sup> The apparatus is similar to the Jenike cell in construction, but the powder bed is split across a vertical diameter instead of in a horizontal plane. Again, the powder is compacted into the cell—an improved technique of compaction has recently been reported<sup>20</sup>—and the tensile strength is measured directly, as the force per unit area of surface which is required to split the sample.

This type of examination can do much towards furthering the understanding of the early stages of agglomeration, such as occur when pigments are transported and stored. Unfortunately, particle interactions do not stop here, and problems of agglomeration are also associated with high compaction. To investigate these, other techniques are required.

## Agglomeration by compression

Ref: 11, 21-24, 24

Since few industrial processes involve low stresses, the vast majority of available literature sources concentrate upon the compaction of powders at high pressures.<sup>11</sup> The high voids content of a loosely packed powder<sup>21</sup>, such as a finely ground pigment, causes it to respond to pressure by rearrangement of particle position. As the pressure is increased the particles move closer together until most of the voids have disappeared. As a result, particle interactions increase, together with the resistance of the powder to further compaction. When this stage is reached, increasing levels of pressure produce diminishing compaction and the pressure response curve may be said to have entered that portion of its cycle which is dominated by high stress.

To study the development of agglomerates under these conditions, it is necessary to use a method in which changes in the powder bed may be monitored during compression. Various methods have been attempted in the past which relied upon an examination of the powder after the compression forces had been removed,<sup>22, 23</sup> and these were predominantly static tests.

The study of compression under dynamic conditions has been possible using techniques developed in the pharmaceutical industry. Powder is compressed in a tableting machine which is equipped with strain gauges on the die wall and the top punch. It is contained and compressed within the die by two punches, the upper one of which introduces pressure to the system. The maximum pressure achieved is controlled by the amount of material present and the size of the space into which the material is being compressed. This pressure is transferred from the upper punch on to the die wall via the powder. By instruments attached to the punch and the die wall, it is possible to measure the pressures developed and follow the behaviour of the powder whilst it is being compressed.<sup>24</sup>

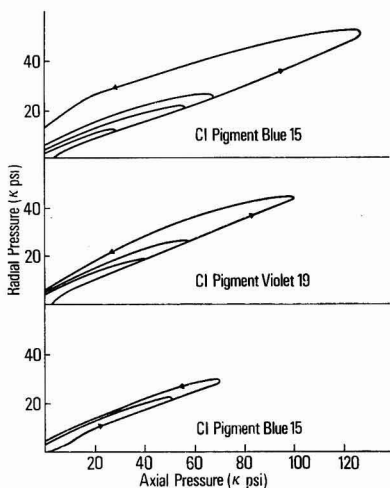


Fig. 2. The development of radial pressure in the die wall with increasing compaction ( $K = 1,000$ ) (Ref. 24)

A set of typical curves is shown in Fig. 2 for three pigments: CI pigments, Brown 6, Blue 15 and Violet 19, illustrating the wide variation in behaviour which may be expected from a

range of pigment types. During the early stages of compaction, some powders flow much more easily than others. This is due not so much to chemical properties as to differences in particle size and surface coating. For example, "beaded" versions of carbon black flow much more easily than their finely powdered equivalents.<sup>24</sup>

The properties of the agglomerate produced also depend upon physical characteristics of the pigment and are sufficiently varied to allow the pigments to be divided into groups. For example, of all the pigments examined, the phthalocyanines as a class were the most elastic and for this reason formed very strong agglomerates. In general, the inorganic pigments were found not only to be much less elastic than the organic pigments, but also gave agglomerates which were much weaker.

## Tensile strength of large agglomerates

Ref: 7, 10

It is unfortunate that the agglomerate strength of many pigments cannot be measured with the instrumentation of the tableting machine. The high level of elasticity shown by the phthalocyanines for example, allows the tablets to deform in the die when the compressive stresses are removed, and the residual stress in the die wall induced by the tablet is not, therefore, a true measure of its strength; however, the tableting machine may be used to produce large, regularly shaped agglomerates (tablets) which have a wide range of density, and it is possible to measure the strength of these by direct methods.

Tablets may be ruptured by the application of a tensile strength along the axis of compression. This method has been used with considerable success<sup>7</sup> but is rather slow and tedious. Each tablet must be sandwiched between, and stuck to two metal surfaces. Care must be exercised to avoid a direct metal to metal bond with the adhesive, either over the edges of the tablet or, if it is porous, by diffusion through the tablet. When the adhesive has set, tensile strength may be measured directly with a tensometer.

A much quicker and more convenient method involves fracturing the tablet across a diameter by compressing it between two metal surfaces.<sup>10</sup> Under these conditions failure occurs across a diameter of the tablet whilst the material is in tension, and knowing the crushing strength it is a simple matter to calculate the tensile strength. A large number of tests are necessary because of the variability of the results obtained due to imperfections in the tablets causing low apparent strength. Even the removal of the tablet from the die can introduce fractures in it which are not readily apparent. The high values obtained will usually be the most valid since impurities and imperfections can only weaken the agglomerate. Emphasis must, therefore, be placed upon those results which show the greatest tensile strength for any given density.

All pigments exhibit a range of tensile strength. The lower end of the range is limited by the strength of the loosely packed powder and can be very low indeed; the upper limit of strength is provided by the solid material free from voids, cracks or flaws. Clearly, the observed strength of the compacted pigment will fall somewhere between these two limits depending upon the level of consolidation. At high void levels it will approach the lower limit of the loose powder; at low void levels it will tend to approach the strength of the solid material; and as both of these limits will be approached

asymptotically, there will be a point of inflection somewhere between the two limits.

In general, this point of inflection occurs at a lower voids content for organic than for inorganic pigments. This reflects the greater ease with which organic pigment particles pack together. Fig. 3 shows some typical results for CI pigments, Blue 15, Brown 6, Green 7 and Violet 19.

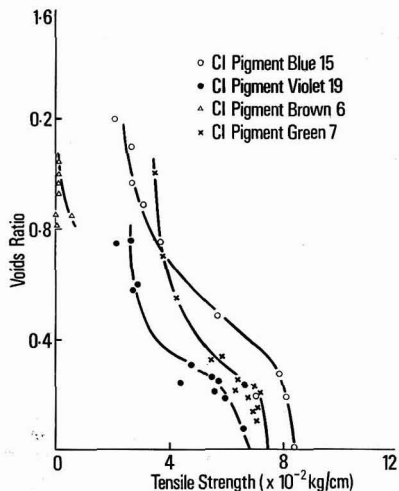


Fig. 3. Development of strength with compaction for various pigment powders (Ref. 10)

It is, of course, difficult to relate the levels of compression achieved in these experiments to those to which pigment powders are subjected during actual use. The properties of a tablet which has been produced in a die must be different from those of an agglomerate produced during tumble mixing. However, it is possible to produce material in, for example, a Henschel mixer which appears to be every bit as hard and strong as that obtained by tableting. Indeed, it is easy to accept that tablets will be weak in comparison with agglomerates produced during mixing because of the strains and flaws which are within them; but a direct comparison between tablets and the large agglomerates which frequently appear as build-up on mixer blades, is difficult, if not impossible, because of the irregular shape and mixed composition of the latter.

The mechanisms by which agglomerates are produced in mixers undoubtedly include compaction, and this will be accompanied by a more ordered type of arrangement than that obtained by tableting. A particle is most likely to contribute to the build-up if it strikes another at a suitable orientation and position. This will contribute to a more highly ordered form of close packing in the agglomerate. The contribution to the final strength of the agglomerate made by the fusion of point contacts<sup>10</sup> will be considerable in both cases, although it could be greater in the case of intensive mixing, since higher temperatures are generated. It would, therefore, be surprising if the agglomerates produced during intensive mixing of powders were not excessively strong.

## Pigment dispersion

### Measurement of particle size

Ref: 25, 26, 10

The examination of pigment particles in a plastic medium is

made difficult by the presence of the medium. This has inevitably led to the development of methods involving visual examination either with the unaided eye, or with some form of microscope. These are typified by the extrusion of the coloured polymer in the form of a thin film, which is then examined at a number of different magnifications.<sup>25</sup> Attempts to use a microscope to extend this technique to the examination of particles only a few microns in size cut in thin sections with a microtome have exposed the subjectivity of the method; but this can be alleviated if the decisions made by the observer during the sizing operation are replaced either in part by using a semi-automatic counter, such as the Zeiss-Endtner microscope, or completely by using a fully automatic image analysing computer such as the "Quantimet".<sup>26</sup>

When the latter is used for this type of study, the image input to the central processing unit is provided by an optical microscope, although other microscopes such as the "Stereoscan" can also be used. This procedure obviously limits the resolution to that of the optical microscope, which is effectively of the order of a few microns; but this is not the disadvantage which it at first would seem to be, for it is the large particles which, if present, cause electrical and mechanical faults in plastic mouldings and extrusions. There is much evidence<sup>10</sup> to suggest that it is the large particle end of the size distribution which has the greatest influence upon pigment dispersion. At the other end of the scale, there appears to be some limiting size below which pigment particles are unaffected by the shear stresses developed within the polymer, though this size limit will vary with pigment type.

## Pigment dispersion

Ref: 27

Most of the mixers used to achieve high levels of pigment dispersion in plastics are batch mixers, in which it may be expected that the rate determining step is the rupture of the pigment agglomerate. The distribution of the particles produced by this rupturing will depend upon the conditions operating within the mixer, and provided these are not altered, distribution will continue at a constant rate. It will, therefore, take the same length of time to distribute a dispersed particle, be it early or late in the mixing cycle. The time taken will vary considerably from one type of mixer to another, and it will be very much longer on a two roll mill (because of the rolling bank) than in a Banbury mixer.

During mixing, greater overall changes can be expected during the early part of the cycle than later on, and this is borne out in practice; so that if the total change in dispersion is plotted against the mixing time required to produce it, a typical exponential decay curve is obtained.<sup>27</sup> The total change in pigment dispersion which results is dependent upon how many potentially dispersible particles are present, which in turn depends upon the level of shear in the mixer. However, the rate at which this change in dispersion is achieved is independent of the number of particles present: it is determined only by how quickly these particles can reach that part of the mixer which contains levels of shear sufficiently high to disrupt them. In batch mixers, this is accomplished more quickly as mixing speeds increase, so that under normal conditions it can be expected that faster mixing would increase both the total change in dispersion and the rate at which dispersion proceeds.

The disruption of an agglomerate occurs only when some threshold force has been exceeded. This does not directly



involve time. Time is only required to pass the particles through the zone of highest energy density. For any given mixing intensity some optimum dispersion level will be attained beyond which nothing will be accomplished by extending the process. Time does not compensate for lack of intensity.

### Evaluation of dispersibility

Ref: 12, 28

In a previous article<sup>12</sup>, a method of evaluating dispersibility was described in which a pigment was compounded into polyethylene, compression moulded to avoid any further mixing, sectioned, and then examined with the "Quantimet" image analyser after various mixing times. Changes in area under the size distribution curve, and from this the changes in the pigment particle population, were then related to these mixing times to give the rate of pigment dispersion under the conditions of the experiment. This experimental method has been designed to reduce the level of many errors, particularly those which arise from sampling.

A variation of this method using the "Quantimet" has been reported for the evaluation of carbon black dispersions in paint and ink.<sup>28</sup>

### Rate at which single pigments disperse in polyethylene

Ref: 12, 29, 30, 12

The technique described above has also been used to determine the rate at which a large number of pigments disperse into polyethylene, and the way in which dispersibility changes with rate of shear.<sup>12</sup> Examples of the results of this study are shown in Fig. 4 for CI Pigments Blue 15, Brown 6, Green 7 (both conventional and predispersed versions), and Violet 19. In general, the inorganic pigments were much more easily dispersed than the organic ones. The most difficult of all of those examined were the phthalocyanines, which is in line with their high elasticity and tensile strength.

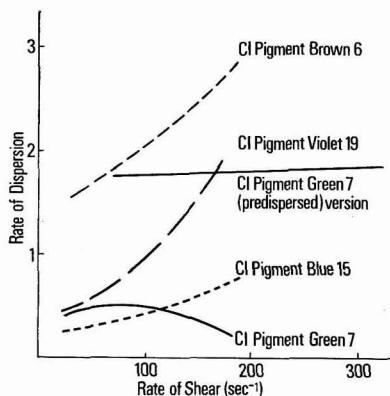


Fig. 4. The variation of dispersion with rate of shear (Ref. 12)

As might be expected, the rate of dispersion of each pigment changed with different levels of shear; but the way in which this occurred, varied from one to another:

- (i) only a few pigments exhibited a linear relationship between rate of shear and dispersion;
- (ii) in some cases dispersion improved considerably for only small changes in shear;

(iii) in other cases dispersion did not improve very much, even after large changes in shear;

(iv) most extraordinary of all, a few pigments appeared to disperse less well at very high than at low shear.

This last and strange behaviour has also been noted elsewhere,<sup>29</sup> where the rate of dispersion of CI Pigment 7 (phthalocyanine green) in polyethylene was studied using the "tintorial strength" method; and where, at high shear rates, the level of dispersion fell away rapidly (Fig. 5).

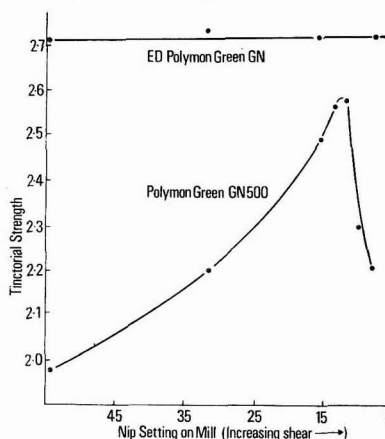


Fig. 5. The development of colour strength with shear for 'Polymon Green GN5000' (Ref. 29)

Work with a similar aim has recently been conducted in the United States,<sup>30</sup> where several pigments were compared for dispersibility using paint as the dispersing medium. In this study, the amount of dispersive energy put into the paint system, using a bead mill, was related to the resulting changes in colour strength; it is interesting to note that the six pigments examined were placed in the same order of dispersibility as was found by the method described above.<sup>12</sup> The results are shown in Fig. 6.

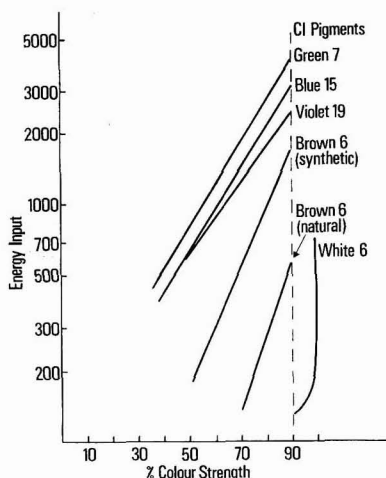


Fig. 6. The dependency of dispersion upon total energy input (Ref. 30)

### The avoidance of agglomerate formation

Ref: 31-35, 12, 36-38, 7, 8, 39, 40

It has been shown that in the three principal stages of colouring plastics materials (the preparation and supply of pigment, the powder mixing process, the compounding or paste mixing process), the nature of these different operations encourages the likely formation of agglomerates in the pigment powder and their perpetuation through powder mixing into the polymer melt. Once individual pigment particles have been surrounded with polymer, all possibility of agglomeration is then removed, but those agglomerates which already exist can only be dispersed slowly and with difficulty.

The evidence accumulated over the years shows that the improvements which result from attempting to disperse agglomerates with high shear are limited. For an agglomerate to be dispersed, it must encounter a stress sufficiently large to break it. However, the dispersing efficiency of compounding equipment is generally low, since most of the energy which is consumed is devoted solely to moving the polymer about. Within a given shear gradient in the polymer, large particles will be more likely to be ruptured than small ones since they will encounter greater stresses; and furthermore, as the strength of agglomerates varies inversely with their size, there is a minimum size of particle below which the mixing process is incapable of affecting further dispersion. This leads to the existence of a steady state, beyond which the level of dispersion can only be improved by increasing the rate of shear. In practice, a mixture made under commercial conditions will not usually have been processed sufficiently long for the steady state to have been reached, because not all of the dispersible agglomerates will have encountered sufficiently high shear. Accordingly, a lengthening of the processing time will permit more agglomerates to come within the reach of the dispersive process. Likewise, improving the strength of the bond between the pigment surface and the polymer (i.e. better "wetting"), or weakening the internal forces of the agglomerate will both lead to improved dispersion.

Many of these improvements can be introduced only by changing the plastics' formulation or the processing conditions; but such changes may not be compatible with the formulation or its final desired properties, and this presents severe restrictions. Obviously, it is better if agglomerates are not formed in the first place.

The problems associated with powdered pigment systems have already been discussed in some detail. It is the physical properties of these powders arising from their particulate structure which is the cause of handling problems. The majority of pigments are produced by processes which at some stage introduce contaminants and develop interparticle structure. Other materials are added subsequently which can also cause agglomeration.

Small particles can carry electrostatic charges or may have electrostatic charges induced in them.<sup>31</sup> Once they have begun to approach one another closely, other attractions such as Van der Waals forces will bring about even more interaction.<sup>32, 33</sup> All powders have measurable tensile and cohesive strengths, and these increase with both the applied load and the length of time for which it is applied. This can occur during storage and transit and can result in the formation of floculates and agglomerates. Preventive methods are not easy to devise as both theoretical considerations and experi-

mental observations have indicated that many variables influence the structure and final strength of a powder bed.<sup>34, 35</sup>

During the powder mixing stage, these loose agglomerates are often retained, or even compressed further. This can lead to the development of particles with high strength through various types of bonding including friction welding.<sup>12, 36</sup> When a powder is sheared, new surfaces are exposed which enhance the formation of direct strong bonds, and by means of this continuing process of compaction, it is possible to produce agglomerates which are just too hard to be dispersed.

Scarcely anything is known about the nature of the interparticle contact within an agglomerate. It is commonly assumed that the equilibrium distance between particles is a few Ångstrom units, but lack of information on the sub-micron structure and the irregularity of particle surfaces prevents a determination of the actual distance. Some attempts at determining these characteristics for clay beds have recently been described;<sup>37, 38</sup> and this type of information has also been used to calculate a theoretical value for the tensile strength of agglomerates and compressed powders.<sup>7, 8, 39, 40</sup>

### Conclusion

A pigment is most suitable for mixing with plastics either if it is only agglomerated with great difficulty or if having agglomerated, the agglomerates are weak and easily dispersed. A pigment will be difficult to use if it compacts easily into hard strong agglomerates or if it builds up easily on a metal surface. Whilst some alleviation is possible here, major improvements will only be possible through greatly increased knowledge of interparticle behaviour. The quickest immediate solution to the problems that arise with these pigments is to develop an alternative method of incorporation into plastics materials.

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## Next month's issue

The Honorary Editor has accepted the following papers for publication, and these are expected to appear in the February issue:

**Some aspects of film formation in emulsion paints. Parts I and II** by *P. D. Aronson*

**Wood varnishes from wattle bark extract** by *H. M. Saayman*

**The determination of copper, mercury and arsenic in antifouling paints by means of X-ray fluorescence** by *C. L. Miniussi and R. H. Perez*

## Short Communications

# The infra-red identification of asphaltic and coal-tar pitches

By A. B. Moustafa

Polymer and Pigments Laboratory, National Research Centre, Dokki, Cairo, Egypt

### Summary

The infra-red spectra of both asphaltic and coal-tar pitches were determined, superimposed and found to be somewhat different.

The ratio of aromatic to aliphatic compounds in coal-tar and asphaltic pitch was found to be 2.11 and 0.116 respectively.

### Keywords

Raw materials: binders, resins etc.

asphalt  
coal tar  
pitch

Processes and methods primarily associated  
with analysis measurement or testing  
infra-red spectroscopy

### Experimental

Pitches are quite often encountered as components of anti-corrosive paints and in many other applications. The main types of pitch are obtained either from coal tar or from asphalt, and it is often difficult to distinguish them by simple means.

Infra-red absorption spectra of samples were determined in the range from  $3,600\text{cm}^{-1}$  to  $700\text{cm}^{-1}$  using a Zeiss Jena UR 10 spectrometer. The infra-red spectra of the two types are superimposed in Fig. 1, and shown to be somewhat different.

linkage in dibenzofurane ( $Ar.C-O-C$ ) and is believed to represent a similar structural grouping. This band is not well characterised, as it occurs in the region where the stretching vibrations of  $C-O$ ,  $S=O$  and  $S-C$  groups absorb<sup>1</sup>, because the intensity of the absorption by these groups is fairly large. The bands which appeared at  $1,380\text{cm}^{-1}$  as a medium band in asphaltic pitch is due to  $C-H$  asymmetrical bonding vibrations of a methyl group;<sup>3</sup> it represents  $C-CH_3$  bonding vibrations. The band which appeared at  $1,460\text{cm}^{-1}$  as a medium band in asphaltic pitch can be assigned to asymmetric bonding vibration of  $C-CH_3$  groups or as asymmetrical deformation vibration of a methylene group of an alkane<sup>2</sup>.

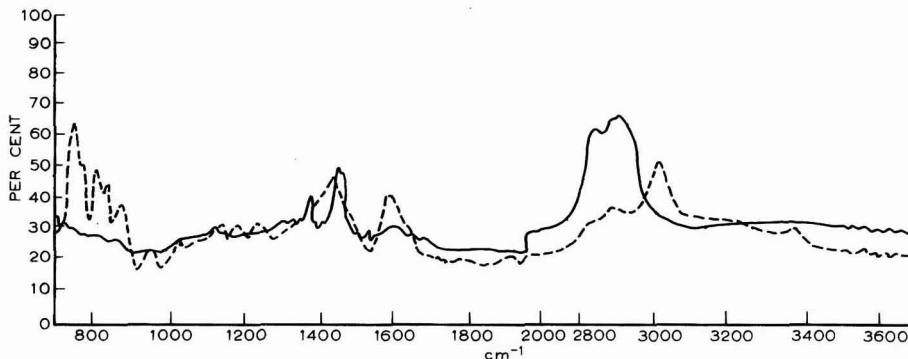


Fig. 1. Infra-red absorption spectra for asphaltic pitch (continuous line) and coal-tar pitch (broken line)

The absorption bands which appeared in the region  $700$  to  $900\text{cm}^{-1}$  can, generally, be assigned to  $C-H$  out-of-plane bonding vibration in aromatic nuclei. The band found at  $815 \pm 5\text{cm}^{-1}$  represents two adjacent free hydrogen atoms, and the band found at  $870 \pm 20\text{cm}^{-1}$  is due to isolated free  $H$  atoms.<sup>1</sup> These bands are found in both asphaltic and coal-tar pitch, with only slight differences in their position of about  $5\text{cm}^{-1}$ . The most significant difference is that the bands are found to be stronger in the case of coal-tar pitch than in that of asphaltic origin. The band at  $960 \pm 5\text{cm}^{-1}$  is due to  $C-H$  out-of-plane deformation vibration. The band at  $1,035\text{cm}^{-1}$  corresponds rather closely to the ether

Aromaticity may be indicated by high relative absorptions at  $1,615$ ,  $880$ ,  $825$  and  $755\text{cm}^{-1}$ . The aromatic  $C=C$  stretching vibration absorbs at  $1,605\text{cm}^{-1}$  in the case of asphaltic and coal-tar pitches, the only difference being that it appeared to be weak in the case of asphaltic pitch, and as a strong band in coal-tar pitch. The bands which appeared at  $2,910$  and  $2,850\text{cm}^{-1}$  are assigned to paraffin side chains on cyclic nuclei<sup>3,4</sup> and may be due to  $-CH_2-$  stretching vibrations of alkanes.<sup>1</sup> Pisental<sup>5</sup> showed that the strong absorption bands in the region  $2,850-2,960\text{cm}^{-1}$  is due to paraffins. The band at  $2,855\text{cm}^{-1}$  represents  $CH-$  stretching vibrations of  $CH_2$  or  $CH_3$  groups. This band appeared stronger in the

case of asphaltic than in coal-tar pitch. The aromatic  $C-H$  stretching vibrations at  $3,030\text{cm}^{-1}$  are present in both types of pitch, but is very weak in asphaltic and strong in coal-tar pitch.

In the case of coal-tar pitch, a weak band appeared at  $3,400\text{cm}^{-1}$ . This is probably due to free  $NH$  (trans) stretching vibration of secondary amides<sup>1</sup>.

### Conclusions

It is concluded from the above facts that the coal-tar pitch infra-red spectrum indicates mainly aromatic compounds, in addition to a number of aliphatic compounds, whilst that of asphaltic pitch indicates mainly aliphatic compounds and a small amount of aromatic ones. The ratio of aromatic to aliphatic compounds was studied by calculating the ratio of the  $C-H$  stretching vibrations in the aromatic nucleus at  $3,030\text{cm}^{-1}$  to that appearing at  $2,910\text{cm}^{-1}$  due to asymmetric  $C-H$  stretching vibrations of  $CH_2$  or  $CH_3$  groups in alkanes.

$(A_{3030\text{cm}^{-1}})/(A_{2910\text{cm}^{-1}})$  is equal to 2.11 in the case of coal-tar pitch, but it is only 0.116 in the case of asphaltic pitch. The base line technique was used for these calculations.

### Acknowledgment

The author thanks Prof. N. A. Ghanem and Dr F. M. Abd-El Karin for valuable discussions.

[Received 9 January 1973]

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# Gravure ink composition changes during transfer

By P. Birkett and S. Elsayad

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In a recent investigation of gravure ink transfer under an applied electric field, changes in the pigment/resin ratio were determined using an X-ray emission technique<sup>1</sup>. Changes found between the unassisted and the electrostatically assisted situations were shown to be dependent on the field strength and direction, and were caused by electrokinetic effects<sup>2</sup>.

However, it was also found that deviations of pigment/resin ratio of the transferred ink from that of the original ink occurred even with no field applied. Moreover, these changes varied with cell depth in a similar manner to those which took place with the field applied in either direction (Fig. 1). The deviation was greatest in the highlight zone. Work with more conductive *n*-propanol inks also showed similar deviations (Fig. 2).

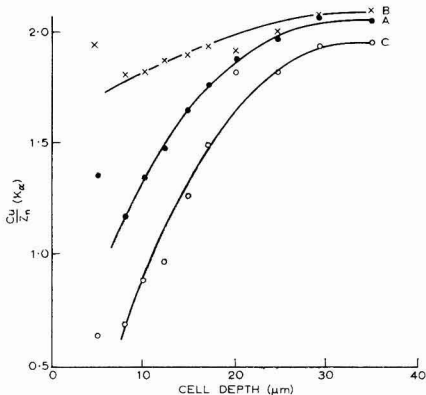


Fig. 1. (Cu/Zn) ( $K_z$ ) in the assisted and unassisted modes. Ink: phthalocyanine blue, ZnCa resinates, toluene

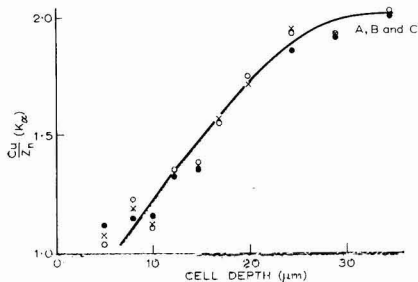


Fig. 2. (Cu/Zn) ( $K_z$ ) in the three printing modes. Ink: phthalocyanine blue, ZnCa resinates, *n*-propanol

The analytical technique was carefully checked to eliminate any possible dependency on ink film thickness before the phenomenon was further investigated.

There are a number of possible causes of selective transfer, involving either the machine, the substrate or the ink. Sub-

strates of polypropylene and coated paper were printed in turn at a speed of  $0.3 \text{ ms}^{-1}$ . The plastic substrate showed no ratio changes for two different viscosity inks (Fig. 3), whilst the paper prints indicated a cell depth dependency (Fig. 4). Similar results were obtained on an Odhams Experimental Press at 1 and  $2 \text{ ms}^{-1}$ .

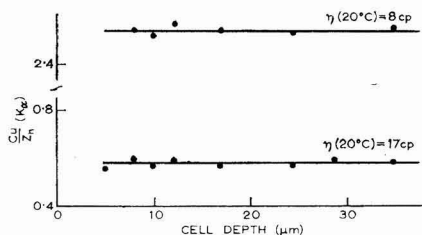


Fig. 3. (Cu/Zn) ( $K_z$ ) in the tonal range on polypropylene substrate

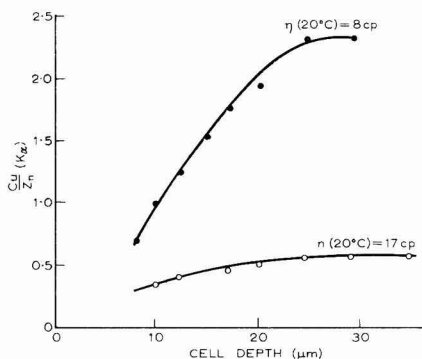


Fig. 4. (Cu/Zn) ( $K_z$ ) along the tonal range on blade-coated paper

Thus, it appeared that an absorbency effect was responsible for the selective transfer; tests with two inks of different viscosity confirm this suggestion, since the higher viscosity ink is only slightly affected, but still shows the expected trend (Fig. 4).

The work done so far shows the effect of selective transfer to be a function of cell depth. It was also shown to be based on penetration of the substrate, where initial penetration through the paper surface layers has an important role. Clearly, the key to the effect which causes some reduction in pigment transfer lies at the ink/paper interface.

Filtration phenomena through the paper body after printing is over, have been previously established<sup>3</sup>. No detailed study exists on what happens in regard to this phenomenon during impression, but preferential absorption in letterpress transfer during the impression phase has been observed by some earlier workers<sup>4</sup>.

From knowledge of the colloid chemistry of ink systems, it can be expected that charges will reside on the surface of dispersed pigment particles. These charges normally assist in preventing flocculation by a simple electrostatic repulsion effect. At the paper surface, as the filtration process proceeds leaving some pigment at the interface, some time is required for these charges to leak away. In the meantime, work must be done by any mobile particle which approaches the surface in overcoming the repulsion. Since the vehicle micelles are not restricted in the same way, a preferential transfer will ensue.

The two observed pigment/resin effects can then be explained as follows: non-porous substrates do not allow filtration to occur, whilst viscous inks will be filtered only slightly.

It is possible that this work will now encourage inkmakers to improve the print quality of gravure highlights.

[Received 14 September 1973]

### Acknowledgment

X-ray measurements were made on Philips PW 1450 and PW 1220 spectrometers by courtesy of Pye-Unicam Ltd., Cambridge.

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## Correspondence

### Chromate and phosphate pigments in anti-corrosive primers

SIR—Thank you for the opportunity to comment on the remarks of Dr Schiefer in his letter to you of 24 August (*JOCCA*, 1973, **56**, 584).

The statement in the paper that zinc phosphate is a non-toxic pigment was based on generally accepted views and particularly those of F. Timmins<sup>1</sup> and British Railways. The writer finds it hard to accept that lead silicochromate, which is believed to be a basic lead chromate coating on a silica core, is innocuous. The fact that it has a low solubility in water is not as important as its solubility in body fluids.

It has been agreed, generally, in the UK that the soluble lead test, first put forward in the Lead Paints Regulations 1927 and still in force, provides a useful guide to the toxicity of lead pigments. Using this test, the soluble lead content of *M50* is 33.6 per cent PbO, and since *M50* contains 47 per cent PbO it appears that 70 per cent of the lead content is dissolved out in the test, which is what one would expect of a basic lead compound. Since the purpose of the soluble lead test is to give indications of the relative toxicity of different lead compounds, it follows that lead silicochromate is much more toxic than normal lead chromate which has a soluble lead content of less than 5 per cent PbO. Under the regulations, a paint based on *M50* would have to be

labelled "containing lead" but a paint made from normal lead chromate would not. Neither paint, of course, could be used on toys.

It is extremely doubtful whether the acute oral toxicity LD<sub>50</sub> value is of any real significance, since lead is a cumulative poison and industrial lead poisoning, when it occurs, is usually the result of absorption of small quantities of lead over a considerable time. Published information indicates that all types of lead chrome pigments<sup>2</sup> together with red lead, litharage and white lead<sup>3</sup>, the last three being well known to be extremely toxic, give LD<sub>50</sub> values of greater than 10g kg<sup>-1</sup>.

Yours faithfully,

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Matlock,  
Derbyshire DE4 3RR.*

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# A scheme for the identification of some polymers and resins

By A. B. Moustafa

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## Summary

A scheme for the identification of some polymers and resins is given. It is based on the determination of the density of the resin, and followed by the heating a small amount of the resin in a gentle flame and an examination of the vapours evolved by

universal pH indicator paper. The solubility of some types of resin in a range of organic solvents affords further information in cases where the initial tests do not give an unambiguous result.

## Keywords

*Processes and methods primarily associated with analysis, measurement or testing*

testing  
thermal degradation

*Properties, characteristics and conditions primarily associated with materials in general*

density  
solubility

## Introduction

The usual analytical methods of polymer analysis may require much time and effort. They differ from those applicable to low molecular weight compounds<sup>1</sup>. Thermal decomposition of polymers is, perhaps, the oldest method which is currently used for the characterisation of polymers; thermal decomposition can occur either at random points in the molecule, resulting in compounds having a wide range of molecular weight but usually greater than that of the original monomer unit, or by a chain depolymerisation process, which results in monomer units in accordance with a mechanism which is temperature dependent<sup>2</sup>.

the material is heated in a test tube (5.0 × 0.5cm) over a small Bunsen burner flame. Heating should be carried out gently to avoid secondary reactions, which might result in increased decomposition and the evolution of vapours which would mask the monomer or primary degradation products having characteristic properties.

The low solubility, together with the chemical inertness, of cross linked or thermosetting polymers favours the use of thermal methods for identification. The vapours evolved are tested with universal pH indicator paper previously moistened with water, and their acidity noted. Observation of the effect of heat on the polymer must be made, including whether it melts, decomposes or sublimes and also whether or not it leaves a residue after heating. The odour and characteristics of the polymer pyrolysates are summarised in Table 2.

It is possible that the two tests described above will not be sufficient to distinguish between two or more polymers, particularly if, for example, the presence of pigment invalidates the result of density determination. In such a case, the solubility of the polymer provides a useful additional guide. The solubility of some polymers in a range of organic solvents is given in Table 3.

The tests do not provide a means of distinguishing between pitches of asphaltic and coal-tar origin. Infra-red spectroscopy provides a method by which this distinction can be made and this has been described by the author in another paper<sup>3</sup>.

Other methods of polymer analysis are described in the literature<sup>4, 5</sup>.

## Experimental

The density of the polymeric material is conveniently determined by the normal density bottle method. The ranges of densities likely to be encountered are given in Table 1.

Table 1  
Classification of some polymers and resins according to density

| Density range<br>g cm <sup>-3</sup> | Polymers and resins                                                                                         |
|-------------------------------------|-------------------------------------------------------------------------------------------------------------|
| 1.0 to 1.2                          | Polystyrene, poly-methylmethacrylate, poly-carbonate.                                                       |
| 1.2 to 1.3                          | Cellulose esters, poly-esters, epoxy and phenolic resins.                                                   |
| 1.3 to 1.4                          | poly-vinylchloride, phenolic resins with organic fillers.                                                   |
| 1.5 to 1.8                          | poly-vinylchloride (filled), poly-vinylidenechloride, chlorinated rubber compounded with inorganic fillers. |
| 1.8 to 2.5                          | poly-trifluorochloroethylene, poly-tetrafluoroethylene, silicones, plastics reinforced with glass fibres.   |

Once the polymer under test has been classified according to its density, it is further examined by the effect of heat upon it and observation of the vapours evolved. A small amount of

[Received 9 January 1973]

Table 2  
The effect of heat on polymeric materials

| Material                | Behaviour on pyrolysis                                                                                                                                                                          | Vapour                                      | Material                    | Behaviour on pyrolysis                                                                                                                                             | Vapour                                                       |
|-------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------|-----------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------|
| Polystyrene             | Melts, decomposes into colourless, yellow or brown neutral vapours. Burns further on ignition with a smoky flame                                                                                | Light gas (benzene)                         | Phenol-formaldehyde         | Decomposes without any flame and the sample retains its shape                                                                                                      | Smell of phenol and formaldehyde                             |
| Poly-methylmethacrylate | Decomposes and gives neutral monomer vapours, which burn when ignited                                                                                                                           | Very strong sweet fruity odour              | Poly-vinylidenechloride     | Decomposes and darkens, producing very acidic vapours, which scarcely ignite; it only occasionally burns in the flame                                              | Smell of hydrochloric acid                                   |
| Coumarone-Indene resins | Melt and then decompose giving neutral or acidic vapours, which burn when ignited                                                                                                               | Odour of coal tar                           | Poly-vinylchloride          | Decomposes and changes to dark brown giving very acidic vapours; it burns with a green border only in the flame, it may burn further if the polymer is plasticised | Smell of hydrochloric acid                                   |
| Poly-carbonate          | Melts to a clear viscous liquid which gives neutral vapours on heating and on further heating gives weak acidic vapours which burn with a luminous flame when ignited                           | —                                           | Urea-formaldehyde resin     | Decomposes giving neutral vapours. No flame can be obtained and the sample retains its shape                                                                       | Smell of formaldehyde                                        |
| Nitrocellulose          | Decomposes violently at high temperature giving strongly acidic vapours, which are a faint brown due to nitrogen oxides. Polymer burns rapidly with an intense white flame further when ignited | Smell of camphor in the case of celluloid   | Melamine-formaldehyde resin | Decomposes, no flame can be obtained and the sample retains its shape                                                                                              | Strong fish-like smell, together with formaldehyde           |
| Cellulose acetobutyrate | Decomposes and blackens, giving yellow acidic vapours; burns with a predominantly blue flame, which may have a white tip                                                                        | Small of butyric acid and burnt paper       | Chlorinated rubber-filled   | Decomposes with evolution of very acidic vapours. Burns in the flame                                                                                               | Smell of scorching rubber, hydrochloric acid and burnt paper |
| Cellulose acetate       | Melts, decomposes giving yellowish-green vapours, carbonises on strong heating and sparks when ignited                                                                                          | Smell of acetic acid and burnt paper        | Poly-trifluoroethylene      | Melts and decomposes on strong heating, giving strongly acidic vapours which do not burn                                                                           | Smell of hydrochloric acid                                   |
| Epoxy resins—unhardened | Melts and then decomposes to give acidic vapours, burns on ignition with a luminous flame                                                                                                       | Smell of phenol                             | Poly-tetrafluoroethylene    | Does not melt, vapourises slowly at dull red heat with strongly acidic vapours which cannot be ignited                                                             | Wax-like evolution of hydrofluoric acid                      |
| Epoxy resins—hardened   | Decomposes giving neutral or alkaline vapours and sublimate, which burns with a luminous flame when ignited                                                                                     | Smell of amine or ester, followed by phenol | Silicone polymer            | No change in a small flame. A white residue of silica is finally obtained                                                                                          | —                                                            |

Table 3  
Identification according to solubility

| Material                       | Water | Ethanol | Ethyl acetate | Ethylene chloride | Benzene | Diethyl ether | Acetone |
|--------------------------------|-------|---------|---------------|-------------------|---------|---------------|---------|
| Polystyrene                    | I     | I       | S             | S                 | S       | G             | G       |
| Poly-methylmethacrylate        | I     | S       | S             | S                 | S       | I             | S       |
| Coumarone-Indene               | I     | I       | S             | S                 | S       | S             | S       |
| Poly-carbonate                 | I     | I       | G             | S                 | G       | G             | G       |
| Nitrocellulose                 | I     | I       | I             | I                 | I       | G             | S       |
| Cellulose acetobutyrate        | I     | G       | S             | S                 | G       | I             | S       |
| Cellulose acetate              | I     | I       | S             | G                 | I       | I             | S       |
| Epoxy resin (unhardened)       | I     | G       | S             | S                 | I       | G             | S       |
| Epoxy resin (hardened)         | I     | I       | G             | G                 | S       | I             | G       |
| Unhardened phenolformaldehyde  | I     | S       | I             | I                 | I       | I             | I       |
| Poly-vinylidenechloride        | I     | I       | I             | I                 | I       | I             | I       |
| Poly-vinylchloride (pvc)       | I     | I       | G             | G                 | G       | I             | G       |
| Pvc plasticised                | I     | G       | G             | G                 | G       | G             | G       |
| Hardened urea-formaldehyde     | I     | I       | I             | I                 | I       | I             | I       |
| Hardened melamine-formaldehyde | I     | I       | I             | I                 | I       | I             | I       |
| Chlorinated rubber             | I     | I       | S             | S                 | S       | G             | G       |
| Poly-trifluoroethylene         | I     | I       | I             | I                 | I       | I             | I       |
| Poly-tetrafluoroethylene       | I     | I       | I             | I                 | I       | I             | I       |

S = soluble. I = insoluble. G = gelation.

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# Information Received

## Albright/Harrison negotiations completed

Harrisons & Crosfield Ltd. and Albright & Wilson Ltd. have announced a successful conclusion to the negotiations for sale of the chrome chemicals business of Albright & Wilson to Harrisons & Crosfield.

## Penair markets Dunlop filter foam

The newly-formed Penair Co. is now in a position to supply design engineers with a highly specialised advice service wherever air is used for processing, and so forth. The company has also taken over the marketing of Dunlop PRB Filter Foam.



A selection of shapes made from the filter foam now available from Penair

## Zirconal markets Anderson binders

Zirconal Processes Ltd. is now marketing in Europe the "Andercase" 12-16-22 grades of prehydrolysed ethyl silicate binders at present manufactured by Anderson Development Co. Ltd. in the USA. Anderson HFP binder is also available for single-pack manufacture of zinc rich paints.

## New products

### Automatic Pearl Mill system for Carbon bead dispersion

Draiswerke GmbH has developed a special feeding screw disperser (patents applied for) which can be integrated with a normal Pearl Mill and allows the automatic feeding of beaded carbon and other components without prior premixing. Any number of liquid and dry products can be automatically metered into the Pearl Mill for dispersion. The machine could also be used, of course, for continuous paint production without the need for premixing.

### Dynamic WVTR Meter

A new instrument is now available from EPS (Research & Development) Ltd. which can rapidly measure the transmission rate of water vapour through permeable materials. The quick and accurate method is particularly suited to "on-line" quality control. Operation is by the sweep gas method, using an electrolytic hygrometer as the measuring element.

## Laser-Doppler velocimeter

A velocimeter which allows the measurement to within 0.1 per cent accuracy of speed without mechanical contact with the flow stream is being marketed by Metrawatt (UK) Limited. The instrument, designated the LSO1 Laser-Doppler, was the result of research by Brown Boveri Company.

## Liquid masterbatch

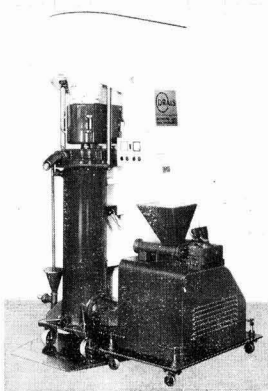
Foscolor Limited, the Wigan-based manufacturer of masterbatches, has announced a new range of liquid masterbatches. Called "Liquicolor Concentrates", these masterbatches are suitable for use with automatic metering units. They are claimed to have excellent flow and, with high colour strength can be used at 0.3 to 0.6 per cent let down in almost any polymer.

## Micro-metering pumps

A new series of glandless diaphragm metering pumps actuated by means of a solenoid driver, is now being marketed in the UK by Apex Fluid Equipment Ltd., Wembley, Middlesex. These "Burdosa WT" series pumps are available with maximum capacities of 52 litres per hour.

## Neumo semi-automatic filling machine

Neumo Limited, New Haven, Sussex has announced its most recent development, an air-operated and controlled semi-automatic filling machine for the handling of liquid and semi-liquid products. The machine features an optional inter-changeable automatic rise and fall filling head for handling foaming products. A 200 litre drum can be filled every three minutes, and accuracy is guaranteed to plus or minus 0.25 per cent by volume.



The special feeding screw disperser from Draiswerke

## Pilot coater from Pagendarm

Erich Pagendarm KG of Germany, a manufacturer of coating and laminating machinery has introduced to the UK a "pilot" laboratory coating machine. This new machine is intended to allow research, product development and production assessment to take place under controlled conditions, but it has roller dimensions large enough to meet actual production requirements. Whilst it is a laboratory machine, tests are carried out under simulated production conditions, and so provide an accurate guide to production costing and material evaluation.

## Release agents from Compounding Ingredients

Compounding Ingredients Limited has introduced a new range of release agents throughout the Common Market countries. The range consists of "Cilase 1812" aerosol and ready-to-use material supplied in bulk, Cilase 1813A in aliphatic solvents, Cilase 1814MC in chlorinated solvents and Cilase 1815F in fluorinated solvents.

The Company has also announced that "Monothane" can now be produced at 100 Shore A hardness.

## Russell Finex extruders

Russell Finex Limited has added to its range of processing machinery a series of units capable of producing small cylindrical extrusions from suitably moistened materials. The units give outputs of 100 to 300kg per hour of wet charge. The company is equipped to carry out tests with manufacturers materials.

## Steeley biocides

Two new broad spectrum biocides for surface coatings are now available in the UK from the Chemicals Manufacturing Division of the Steeley Co. Ltd. The preservatives, called "Amical 48" and "Amical 77", have been developed and patented by Abbott Laboratories of Chicago and shown to be effective in latex paints with no adverse effects in terms of colour retention, flaking, chalking or cracking. The "Amicals" are substituted phenyl diiodomethyl sulfones supplied as micronised powders.

## Literature

### Science Reference Library

The Science Reference Library has announced that it is to produce a new series of publications called "Guidelines" listing selected publications dealing with different aspects of a general topic. The publication was conceived as an introductory guide to assist those who in the course of research and development work, were approaching the literature of the subject for the first time.



### Bristol

#### Colour in the home

The 196 meeting was held at the Royal Hotel, Bristol, on Friday 28 September, when Mr R. G. Stay and Mr R. Brain of Berger Paints, presented a programme on "Colour in the home."

Mr Stay began with a talk dealing with some of the basic theory of colour and the Munsell system, demonstrating his points with charts. He went on to show how colours may be used together in decorative schemes and how the choice must take into account such matters as lighting, location and relative areas within the home. Mr Stay made copious use of colour illustrations to show the factors that should be considered in making a choice.

There followed the showing of an excellent film relating the colours of nature to the range of colours open to the home decorator and showing their use in colour schemes. The subject aroused considerable interest and a lively discussion followed on which Mr Stay and Mr Brain enlarged on many points. A vote of thanks was proposed by Mrs E. N. Harper and was warmly supported by the 36 Members and guests present.

R.F.N.

#### Testing and performance of flame retardant paints

Mr F. C. Adams, from the Fire Research Station, presented a paper entitled "The testing and performance of flame retardant paints" on Friday 26 October 1973.

Mr Adams explained the work of the Fire Research Station, and its philosophy of testing. He outlined the general action of a confined fire, and the effects of time upon it. Spread of the flame was considered and the lecture was confined purposely to those effects which happened before the "flash-over" period.

The 1972 Building Regulations were considered, particularly their effects upon flame spread and available fuels. The effects of toxic gases, smokes, and internal furnishings were also considered.

The Surface Spread of Flame test (BS476 Part 7) and the Fire Propagation test (BS476 Part 6) were considered in detail. The importance of the correct interpretation of results was explained, and the performance of a wide range of systems was considered.

Video tape was used to illustrate typical results.

The discussion was opened by Mr J. Cox and the 18 guests and Members present participated in the lively discussion which followed. A vote of thanks was proposed by Mr N. Morgan, and was warmly supported in the usual manner.

R.F.N.

### Hull

#### Acrylic polymers in emulsion paints

The first meeting of the new session was held at the Dorchester Hotel, Hull, on 1 October. The Section chairman, Mr F. D. Robinson, introduced Mr K. Horsfield of Allied Colloids Limited, who gave a lecture with the above title.

Mr Horsfield briefly reviewed the role of low molecular weight, water-soluble acrylic polymers and other dispersants in the pigment dispersion process for the manufacture of

emulsion paints. Proof of the adsorption of the acrylic polymer on the pigment particle had been obtained by radio chemical techniques, and stability was conferred on the dispersion by double layer electric repulsion between particles.

The dispersion efficiency of candidate materials may be conveniently obtained by a method attributed by Mr Horsfield to English China Clays Limited, in which the viscosity of a number of pigment dispersions of equal solids' content is plotted against the concentration (on the pigment) of the dispersant. The theoretical concentration of dispersant required for stabilisation was that which produced the lowest viscosity dispersion in the test, but in the final formulation it was necessary to consider the storage stability of the paint, including the influence of heat and alkali, and it was usual to increase the theoretical dose rate of the dispersant.

The lecturer then discussed high molecular weight, water- and alkali-soluble acrylic polymers, and suggested that their thickening action stemmed mainly from the adsorption of each polymer molecule at the surface of two or more pigment particles to produce a three dimensional network. Thickening of the aqueous phase was less important, and the full action of the thickener could take a day or two to develop. Mr Horsfield considered that acrylic thickeners were more resistant to biodegradation than their cellulosic counterparts, and suggested that acidic polymers in emulsion form were particularly convenient in paint manufacture.

Finally, the lecturer pointed to the relationship between the gloss of the paint film and the compatibility of the acrylic polymer with the glycol constituent of the paint.

The lecture was followed by a short discussion period and then a buffet during which Mr Horsfield answered further questions put to him by the audience. The vote of thanks was proposed by Mr J. A. Hasnip and the meeting was attended by 19 Members and 11 visitors.

J.A.H.

### London

#### Some aspects of technical training

A meeting was held on 16 October 1973 at the Great Northern Hotel with Mr R. H. E. Munn in the chair. Mr G. P. Birtles of Selection and Industrial Training Administration Ltd., gave a lecture with the above title. Mr Birtles commenced by describing the limitations of his own introduction to the paint industry, which he considered to be fairly typical. He then went on to give an account of the training scheme for paint technologists which was now presented by his organisation.

The basis of the scheme was that the training requirements of each individual were different, because of their differing backgrounds. So in the training scheme each subject was treated at low and at higher levels. A series of loose-leaf text books was provided, enabling easy up-dating of any topic by insertion of fresh pages. Each student was given a comprehensive index, which was used by the training officer to plan his course of training. The books included a large number of short practical exercises.

In operation, it would be decided that a student would have a definite period or periods each week for training. When the time for his training came, he would consult his individual index to find what work should be done and then study the relevant section of the training manuals, and carry out the experimental work which was described in detail. This training could be done in the laboratory, and where several students were concerned it was found best to schedule each student for a separate period of, for example two hours.

By this means, there was the minimum disturbance to the normal work of the laboratory.

The student's progress was checked every two or three weeks by a senior member of the laboratory, or the training officer if there was one. If the student had not absorbed the information he would be directed to repeat the work. If his progress was satisfactory, he would pass on to the next item indicated in his personal index.

The talk was followed by a fairly extensive discussion, and a vote of thanks to the speaker was proposed by Mr H. C. Worsdall.

V.T.C.

## Manchester

### Student Group

#### Instrumental colour measurement

A meeting was held on Wednesday 17 October 1973, when Mr J. Bravey, of Berger Chemicals Ltd., delivered an extremely well illustrated lecture with the title "Instrumental colour measurement." The content could well have been extended to a short course of lectures but Mr Bravey skilfully covered the salient points in about 70 minutes. Question time gave some opportunity to expand points of specific interest. The appreciation of an audience of ten Members and 11 visitors was ably expressed in a vote of thanks by Mr M. Povah.

A.McW.

## Midlands

### Recent developments in radiation curing

The first meeting of the 1973-74 Session was held at the Birmingham Chamber of Commerce, on Friday 28 September 1973.

A paper entitled "Recent developments in radiation curing" was given by Mr D. R. Bailey of Donald McPherson Ltd.

The lecturer commenced by defining radiation curing processes and the various methods of producing and controlling radiation. Systems utilising electron beams were available both for curing in air and under vacuum, and the relative merits and costs of both systems were compared.

The utilisation of various types of radiation for curing also embraced ultra-violet, infra-red, radio frequency, and plasma torch sources. Again the relevant factors of these various methods were highlighted.

The mechanism of radiation curing was discussed in some detail, as well as the necessary properties of the various types of coating in order for radiation curing to be effective. As with conventional systems, the final properties of the coatings were usually some form of compromise.

A series of slides was shown, illustrating the various items of plant used by the lecturer in the course of his development work.

An interesting discussion followed, and the vote of thanks was proposed by Mr R. McD. Barrett.

F.W.C.

### The current status and role of amino resins in surface coatings

The October meeting was held at Birmingham Chamber of Commerce and Industry on Friday 19 October 1973. The lecture was given by Mr R. McD. Barrett of BIP Chemicals Ltd., Hon. Secretary of the Section.

The speaker commenced with a brief history of amino resins in surface coatings, building this round part of the suppliers' sales literature for 1936. Widespread acceptance of the early urea formaldehyde (u/f) resins had been slow, in spite of their possessing unique properties in pale coloured stoving enamels, mainly because of cost considerations. Also, the first u/f resins had been supplied in powder form, and a considerable amount of preparation by the paint manufacturer was necessary before they could be used; however, the familiar liquid products were soon forthcoming.

A series of slides illustrated relative costs, together with details of other industrial uses and a breakdown of amino resins usage in various surface coatings. Whilst the cost of synthetic resins, generally, had increased, the cost of amino resins had dropped. Only about 8 per cent of the 158,000 tons of amino resins produced in the UK this year would be used in surface coatings. Major outlets were found in moulding materials, chipboard and adhesives.

Next followed an outline of the constitution of amino resins. The melamine/formaldehyde (m/f) resins, with a higher functionality, were more reactive than u/f resins, and also more stable. The mechanisms of etherification and of reaction with other film formers were illustrated, as were the range of resin properties available as a result of various degrees of etherification of m/f resins. The newer film formers had special requirements, and suitable amino resins had been—and were being—developed as necessary. In this context, mention was made of acrylic resin containing hydroxyl groups, where the twin requirements for the cross-linking amino resins of high compatibility and high reactivity had been successfully met.

In summarising, the speaker emphasised the steady progress achieved in amino resins' technology, the key words being adaptability and versatility.

A most interesting discussion followed, during which several points raised during the lecture were expanded upon. The vote of thanks was proposed by Mr T. D. Nation.

F.W.C.

## South African

### Transvaal Branch

#### Sealants for the building industry

A meeting was held on 18 April 1973 at the Sunnyside Park Hotel, Johannesburg, when Mr D. J. Pienaar, of the South African Bureau of Standards, gave a lecture entitled "Sealants for the building industry." The paper was confined to post-formed sealing compounds—those applied by knife, trowel or caulking gun as a viscous paste or mastic, and which underwent a physical and/or chemical change after application and exposure.

The results of a previous survey had shown that the local market for constructional sealants and caulks excluding putty was of the order of R1 to 1.25 million. This could be divided approximately into: polysulfides 60 per cent, silicones 10, polyurethanes and acrylics 10, oleo-resinous and butyl 20 per cent. Indications were that the use of sealants was growing at a much faster rate than the construction industry itself.

The list of applications of sealants was almost inexhaustible but it was possible to categorise them into:

- Exterior sight, exposed (approximately 75 per cent of the total usage).
- Interior sight, exposed.
- Interior and exterior, concealed.

Those under (b) and (c) accounted equally for the remaining 25 per cent (approximately) usage. Specific applications in each class were enumerated in the paper, and the method of specifying the sealants was discussed.

The sealant industry in South Africa had developed largely in the last six or seven years. The advent of curtain wall structures, the general increase in the height of buildings and the method of system buildings or prefabrication had intensified the problem of obtaining a waterproof joint. Although the need for expansion joints in structures was accepted, the major sources of movement, which might affect all joints whether designed specifically as movement joints or not, were detailed.

The properties and major applications of glazing putty, bituminous compounds, oleo-resinous mastics, butyl sealants, and acrylic, polyurethane, silicone and polysulfide sealants were described in detail. The lecturer concluded with a discussion of primers and back-up materials, in which it was emphasised that as adhesion was a prerequisite for a sealant to perform the function for which it was designed, it was essential that this first step be carried out conscientiously and in accordance with recommendations.

A vote of thanks proposed by Mr A. N. Astfalck was warmly responded to by an appreciative audience.

P.A.J.G.

#### Surface-active substances and their importance in the paint industry

A lecture was given on 31 July 1973 at the premises of T & C Chemicals Pty Limited, Isando, at 8 pm when Mr L. A. van Dam of Byk-Mellinckrodt Chemische Produkte GmbH Wesel, West Germany, spoke on surface-active substances and their importance in the paint industry.

Whilst there was a wealth of information on resins, pigments, solvents and to some extent driers in the text books, generally, very little could be found about additives.

Problems where wetting agents could be advantageous were enumerated:

1. Viscosity. It was advantageous on economic grounds to work as much of the pigment into the mill base as possible. A reduction of viscosity was desirable, therefore, and this could be achieved by the addition of wetting agents.
2. Long dispersion times. This problem, which stemmed from the presence of hydrophilic pigments in hydrophobic vehicles, could be alleviated by the addition of wetting agents.
3. Flocculation. This complex subject was discussed in some detail by the lecturer, and the fact that wetting agents could prevent flocculation and also cause it under certain circumstances was explained.
4. Floating and flooding. These phenomena were discussed in considerable detail and their causes analysed. The controlled use of specific wetting agents was mentioned in this connection.

A description of how wetting agents functioned was given, and some of the problems which could be caused by the indiscriminate use of these agents were enumerated. Four types of wetting agents were listed and explained by the lecturer. He then concluded by describing what could be achieved by the correct use of wetting agents.

Following the lecture a film was shown in which controlled co-flocculation was followed under a microscope. The effect of addition of wetting agents was illustrated.

After several questions a vote of thanks to the speaker was proposed by the Chairman Mr P. Gledmacher, who also thanked the host for the evening.

P.A.J.G.

## Thames Valley

### Recent developments in Europe on wood finishing

The first meeting held at the Beech Tree Hotel, Beaconsfield, on 27 September 1973, started at 7.00 p.m. The Chairman Mr Tatton, opened the meeting by welcoming all Members and visitors, and appealed to Members to notify the Committee if they were interested in a social evening to the theatre during the winter. He then welcomed Dr Schwartz, of Glasurit Ltd., and his wife. Dr Schwartz introduced his subject as "Recent developments in Europe on wood finishing." He outlined the whole range of wood finishing materials as applied to flat surfaces, mainly of composition boards and flat hardboard, also chip boarding, explaining the difficulties with each type of board.

The main theme of the lecture dealt with ultra-violet curing, the most widely used and the most economical for fast and suitable curing. Materials mentioned were acrylics, polyesters, polyurethane and two-pack systems.

The lecture was illustrated with slides and diagrams of the machinery used; unfortunately, there was some difficulty and the projector broke down before all the slides were shown. Dr Schwartz handled the situation very well and was to be applauded for his efforts under extreme difficulties.

He explained that the opaque or white finish on the sample panels was created by the use of an extender, and the effect of the ultra-violet light on the coating was to give suitable finishes for shelving and so forth. Curing times of 25 seconds and less were normal with high pressure lamps and 90 seconds with low pressure ones.

Question time drew many questions from an appreciative audience; Dr Schwartz answered to everyone's satisfaction, but would not divulge the extender used.

Mr Prigmore proposed a vote of thanks, to which the audience responded in a suitable manner.

D.F.

### Printing inks in the seventies

The meeting held at the Beech Tree Hotel, Beaconsfield, on Thursday 25 October 1973, started at 7.00 p.m. In the absence of Mr Tatton, Mr Inshaw, Chairman Elect, opened the meeting, introducing Mr G. H. Hutchinson of Croda Polymers Ltd.

After a review of the advances in printing inks that had taken place over the past twenty five years or so, some recent developments and ideas for the future were discussed, illustrating film-forming properties of various types of inks by the use of slides, diagrams and molecular models. Two factors had influenced trends in ink manufacture—the demand for inks to print on faster printing machines at higher production rates, and the need for inks to print on an ever increasing variety of new papers, boards, plastic films and foils.

Some emphasis was placed on a review of ink drying mechanisms—penetration, oxidation, precipitation, evaporation and new systems involving chemical curing (catalysed inks) and radiation techniques including ultra-violet curing, microwave and dielectric drying.

Advances in ink technology had been greatly aided by developments in new pigments, synthetic resins, polymers, solvents and chemical additives, although currently problems were being encountered because of shortages of raw materials. Relatively new products in the ink industry included vapour phase transfer of the dye image via its vapour phase to a dye receptive synthetic fabric under controlled conditions of time, temperature and pressure.

Stimulation for the development of solventless inks had been provided by anti-pollution regulations, for example, in the USA, leading thermo-curing web-offset and web letterpress inks and ultra-violet curing inks, in which latter field the UK printing industry was expressing growing interest.

The speaker gave a few statistics on ink production in countries within the EEC, emphasising the competitive element and the trend towards more automation in printing ink manufacture, and the adoption of improved pigment dispersion techniques.

With the current shortage of raw materials in mind, the speaker touched on alternative sources of raw materials with some ideas on the use of unsaturated vegetable oils as raw materials for chemical intermediates, and polymers to supplement petrochemical sources. Water-based inks for flexographic printing were expanding, and there would be a greater interest in the future in water-based gravure inks. The problems in printability and drying of these products were being actively studied.

After numerous questions from the Members and visitors, the meeting concluded with a vote of thanks to Mr Hutchinson for his efforts and enthusiasm for the work and preparation which had gone in to the lecture. The audience gave a warm response in the usual manner for a lecture which was well presented and easily followed.

D.F.

## Student Group

### Aerosols

The first meeting of the 1973-4 session was held on 4 October 1973, at Slough College of Technology. Mr A. Barnes took the chair, introducing Mr Southby, of Aerosols International Ltd., who gave the lecture.

Mr Southby outlined the development of aerosols from the original patents in 1823, to the first practical aerosols in 1942 by Goodhew and Sullivan, followed by a review of the tremendous increase in the usage of aerosols from 44 million in 1960, to over 400 million in 1973, and to an estimated increase in 1975 of 550 million.

This was followed by a detailed description of the principles of two- and three-phase systems of propellants and products and the use of compressed gases. The pack working pressure is usually 35 to 40 psi limited by the pressure performance of the container and its closure, plus appropriate safety margins.

The various components were then reviewed, including the containers, various types of valves and available propellants. The problems of formulations and action of propellants and products on the aerosol was widely discussed, and followed by a description of filling methods. The lecture was rounded off with a discussion on the future development of aerosols, and possible impact on the environment.

A lively discussion followed, ending with a vote of thanks by Mr Kemp, who congratulated the speaker on his presentation of a lucid and interesting lecture.

A.B.

## West Riding

### The performance of wood primers

A meeting was held on Tuesday 9 October 1973 at the Griffin Hotel, Leeds. Mr P. Whiteley, of the Building Research Station, presented a paper dealing with the above mentioned subject.

Mr Whiteley's paper was mainly concerned with the background and results of work carried out over the past six years aimed at producing performance standards for oil-based leadless primers.

The speaker discussed the problems encountered with the decay of joinery and the possible causes of failure of primers. A large programme of work had been undertaken to investigate the effect of preservative treatment on primer performance and it was established that certain types of preservative were able to enhance the performance. Another area of interest which had been examined was that of paint film permeability.

A paint film extensibility test had been developed which it was hoped could be used to assess performance without the necessity for long exposure tests. The change in extensibility after one, two and three weeks' weathering was measured, and there appeared to be a relationship between extensibility after two weeks' weathering and the degree of cracking of a primer on wood after ten weeks' weathering.

A considerable number of commercial primers had been examined. They were exposed on tongue and grooved board having 8 to 9 per cent moisture when coated and painted on the front face only. A set of panels was used for each primer and consisted of:

- (a) untreated wood plus primer,
- (b) untreated wood (different batch) plus primer,
- (c) untreated wood plus primer plus gloss finish, and
- (d) preservative treated wood plus primer.

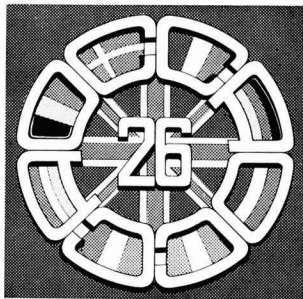
The wood used in set (b) was the same species as for (a), but contained a different number of rings per inch.

Undercoat was omitted in order to accelerate the weathering process. Various properties of the paint film, notably cracking and extensibility, were noted over a period of time and it was found that the results on each set of panels were similar, but with different rates of failure.

It was found that some primers failed in one day from cracking, and over 50 per cent failed in three months. A few failed through chalking. Bad primers were found to be equally bad whether one or two coats were affected, whereas a good primer was slightly better with two coats. It was confirmed that certain preservative treatments gave improved primer performance. A gloss coat was found to have little effect on the performance.

A lively discussion followed, which indicated the high degree of interest the paper held for all present. A vote of thanks was proposed by Mr T. Apperley.

R.A.C.



## East and West join in OCCA 26— the European Economic Community's forum for technical display and discussion in the surface coatings industries 1974

The Exhibition Committee is pleased to report that the first allocation of space at OCCA-26 includes companies from 13 overseas countries. Not only is the EEC well represented but on this occasion participants have been drawn from a wider field, including many exhibitors who had not shown on previous occasions. The full list of countries at present represented is:

Australia, East Germany, Finland, France, Holland, Hungary, Italy, Poland, Rumania, Spain, Switzerland, USA and West Germany. Of the exhibitors, 12 have never shown at previous OCCA Technical Exhibitions, whilst 15 others did not show at the 1973 Exhibition.

The preliminary list of Exhibitors is as follows:

- \* Abbey Chemicals Ltd.
- † Allied Colloids Ltd.
- Anchor Chemical Co. Ltd.
- Bachofen, Willy A.
- BIP Chemicals Ltd.
- Boulton, William Ltd.
- Buhler-Miag (England) Ltd.
- \* Cardew, Alexander, Ltd.
- CdF Chimie
- \* Chemolimpex (Hungary)
- \* Ciech (Poland)
- † Cole, R. H. Ltd.
- † Cray Valley Products Ltd.
- † Crosfield, Joseph & Sons Ltd.
- D. H. Industries Ltd.
- Diffusion Systems Ltd.
- † Draiswerke GmbH
- Eastman Chemical International AG
- Elcometer Instruments Ltd.
- Erlich, G. J., Ltd.
- Ferranti Ltd.
- Fischer Instrumentation (GB) Ltd.
- \* GAF (GB) Ltd.
- † Godrich Chemcol
- † Godrich, John Ltd.
- ICI Limited
- \* Imeco (Rumania)
- Industrial Colours Ltd. (Sericol)
- Instrumental Colour Systems Ltd.
- Jacobson Van den Berg & Co. (UK) Ltd.
- † Jenag Equipment Ltd.
- Kingsley & Keith (Chemicals) Ltd.
- \* Knauer, Dr Ing. Herbert & Co. GmbH
- Kollmorgen (UK) Ltd.
- † Langer, Georg M. (with Cray Valley)
- Laporte Industries Ltd.
- Marchant Bros. Ltd.
- Mastermix Engineering Co. Ltd.
- \* Metal Box Co. Ltd.
- Microscal Ltd.
- \* Mikropul Ltd.
- Montedison
- National Westminster Bank Ltd.

- Polymers, Paint & Colour Journal
- \* Raschig, Dr G., GmbH
- Research Equipment (London) Ltd.
- Rehm & Haas (UK) Ltd.
- † SCC Colours Ltd.
- Schering AG
- † Scott Bader & Co. Ltd.
- Sheen Instruments (Sales) Ltd.
- † Stanhope-Seta Ltd.
- Strazdins, A., Pty. Ltd.
- \* Sun Chemical Corporation (Pigments Division)
- † Swada (London) Ltd.
- † Tin Research Institute
- Toioxide International Ltd.
- Torrance & Sons Ltd.
- Torsion Balance Co. (GB) Ltd., The
- Transportmaschinen
- † Urachem International
- \* Wayfarers Travel Agency Ltd.
- Winter Osakeyhtio
- Worsdall Chemical Co. Ltd.
- Zeiss, Carl (Oberkochen) Ltd.

\* Denotes companies who have not previously shown at an OCCA Exhibition

† Denotes companies who were not at the 1973 Exhibition

### Hours of Opening

There is no charge for admission to the Exhibition which will once again take place in the Empire Hall, Olympia, London, W14 and will be open on the following dates and times:

|                    |    |               |
|--------------------|----|---------------|
| Tuesday 23 April   | .. | 09.30-18.00hr |
| Wednesday 24 April | .. | 09.30-18.00hr |
| Thursday 25 April  | .. | 09.30-18.00hr |
| Friday 26 April    | .. | 09.30-16.00hr |

### 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. An application form for tickets will be enclosed in each copy of the Official Guide.

### Official Guide

The Official Guide will be prepared well in advance of the dates of the Exhibition so that copies can be circulated widely, 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 Com-

mittee 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.

### Aims of the Exhibition

The Exhibition Committee wishes the aims of the Exhibition as stated in the Invitation to Exhibit, to be as well known as possible and accordingly these are reproduced below.

The aim of the Exhibition is the presentation of technical advances in those industries supplying the paint, varnish, printing ink, colour, linoleum and other allied industries. The technical advances may relate to: new products, new knowledge relating to existing products and their uses, or in suitable cases, existing knowledge which is not generally available in the consuming industries.

The Committee stipulates that exhibitors present a technical theme—i.e. to display in a technical manner the technical developments in raw materials, plant or apparatus illustrated by experimental evidence. It is essential that a technically or scientifically trained person who has full knowledge of the products displayed, be available on the stand throughout the official hours of opening.

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

### Travel Agents

The Wayfarers Travel Agency Ltd., Cranfield House, 97-107 Southampton Row, London WC1B 4BQ, will be allocated a stand facing the OCCA Information Centre at the Exhibition and will be prepared to advise on, and arrange, hotel accommodation and travel facilities to the Exhibition. They will also be able to make theatre ticket reservations for the evenings of the Exhibition.

### International character

The Exhibition, which has long been known as the forum for technical display and discussion for the surface coatings



## OCCA 26—continued

industries, in 1973 attracted visitors from more than 50 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 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.

### Other Services

Special folders for technical literature will be once again distributed free of charge at the entrance to the Empire Hall. Facilities available at the Empire Hall include a licensed restaurant and two licensed buffets, one on each of the two floors on which the Exhibition will be located. Escalators to and from the first floor will be in operation throughout the hours of opening and attractive seating areas—which have long been a feature of OCCA Exhibitions—will be provided on both floors.

## President's visit to SLF Conference

The President, Mr L. H. Silver, attended a two day conference of the Scandinavian Federation of Paint & Varnish Technicians (SLF) held in Sandefjord, Norway, from 1 to 3 October 1973. There were 310 delegates and 50 companions present, and 11 papers were presented, one of which was presented on behalf of the Association by Dr G. de Winter Anderson and this was well received.

Mr Silver replied on behalf of the guests at the Dinner Dance and through the aid of a Norwegian friend, had been able to begin his address in Norwegian.



From left to right, Mrs Rook, A. G. Rook (FSPT), A. O. Brantsaeter (SLF), Mrs Silver, L. H. Silver (OCCA), Mrs Rechmann, H. Rechmann (FATIPEC).

## 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.

## Obituary

### Mr J. T. Peacop

With regret, we record that whilst on an American tour with his wife, Mr J. T. Peacop of Penquit, 18 Langley Road, Slough, was taken ill and died in hospital in Hollywood, on Monday 29 October 1973.

Mr Peacop who was 65, was a native of Blackpool, and went to live in Slough about 21 years ago.

He was Principal of the Uxbridge firm of Technical Paints Services, a business he founded in Hillingdon in 1946, then the need for expansion took him first to Yiewsley, then to Uxbridge.

He was a Fellow of the Royal Institute of Chemistry, and an Associate of the Manchester College of Technology. As Chief Chemist with various large paint concerns, he was responsible for the introduction of many new brands of paint which are now household names.

He was the founder Honorary Secretary of the Bristol Section and a founder member of the Thames Valley section. He also acted as a consultant, and an Air Ministry Inspector was a member of the Uxbridge Industrial Estate Association, and of the Uxbridge Chamber of Trade.

He, and Mrs Peacop had visited New York, San Francisco, and Los Angeles before visiting Hollywood, and they were due to go to Washington when Mr Peacop was taken ill.

He leaves his widow, three daughters, and a son who is a member of the Uxbridge business, and our condolences are passed to them.

## Admissions to the Professional Grade

The following admissions were authorised by the Professional Grade Committee at its meeting in December 1973. The Section to which the Member is attached is given in italics.

### Fellow in the Technology of Surface Coatings (FTSC)

Hill, Lawrence Albert (*General Overseas—Australia*)

Talbot, Ernest Alexander (*Thames Valley*)

### Associate in the Technology of Surface Coatings (ATSC)

Caffery, George Francis (*London*)

de Waal, Tielman Johannes (*South African*)

Patrick, Alan Clive (*Irish*)

### News of Members

Following his lecture to the Fachgruppe Anstrichstoffe und Pigmente of the Gesellschaft Deutscher Chemiker, Mr A. R. H. Tawn (Honorary Research and Development Officer) has been elected to honorary membership by the Council of the GDCh-Fachgruppe "Anstrichstoffe und Pigmente". This will entitle him to receive all circulars and reports on the Section's activities automatically, and congratulations are offered to Mr Tawn on the honour which has been conferred upon him.



## 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

- DAVIES, THOMAS ALAN, The United Paint Co., Victoria Street, E15 4PW. (London)
- DRUMMOND, COLIN STEPHEN, 2 Freeland Grove, High Acres Estate, Kingswinford, Staffs. (Midlands)
- HILL, LAWRENCE ALBERT, BSc, 13 Melby Avenue, Balaclava, Victoria 3183, Australia (General Overseas)
- JARVIE, ROGER WALTER, 105 Castle Road West, Oldbury, Warley, Wores. (Midlands)

- MACPHERSON, RODERICK SOMERLED, BSc, PhD, 3B Poplar Road, Leatherhead, Surrey. (Thames Valley)
- OWEN, DAVID JOHN, 50 Sussex Court, Abel Road, Berea JHB, South Africa. (South Africa)
- SAUNDERS, HARRY DOUGLAS, BSc, 5 Private Road, Somerset West, CP, R. S. Africa. (South Africa)
- SHAH, P. N., 119 Lyon Park Avenue, Wembley, Middlesex. (London)
- TAYLOR, GEORGE/MILNE, BSc, 69 Beech Lane, Earley, Reading, Berks. RG6 2QA. (Thames Valley)
- WILSON, DEREK MONTGOMERY, "Charnwood," Chapel Lane, South Cockerington, Louth, Lincolnshire. (Hull)

### Registered Student

- JACKSON, GRAHAM PHILLIP, 65 Scholes Lane, Scholes, Cleckheaton, Yorkshire. (West Riding)

## 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.

### January 1974

#### Thursday 3 January

*Newcastle Section:* "Masonry coatings" by G. W. Rothwell, Building Research Association.

#### Monday 7 January

*Hull Section:* "New developments in colour instrumentation" by R. P. Best of Instrumental Colour Systems Ltd., at the Dorchester Hotel, Beverley Road, Hull at 7.00 p.m.

#### Tuesday 8 January

*West Riding Section:* "Epoxy resin curing agents" by Dr C. G. Tilley of Anchor Chemical Co. Ltd., to be held at Griffin Hotel, Leeds at 7.30 p.m.

#### Thursday 10 January

*Midlands—Trent Valley Branch:* "Pollution" by Mr J. B. Davies, Goodlass Wall & Co. Ltd., Speke, Liverpool to be held at British Rail School of Transport, London Road, Derby at 6.30 p.m.

#### Friday 11 January

*Manchester Section:* "Colour difference, the measure of the future" by Mr K. McLaren ICI Ltd, Organics Division, at the Manchester Literary and Philosophical Society, 36 George Street, Manchester 1, at 6.30 p.m.

*Scottish Section:* Annual Dinner Dance in the Albany Hotel, Glasgow.

#### Wednesday 16 January

*Scottish—Eastern Branch:* "Marketing in Europe" speaker to be announced. To be held at Carlton Hotel, North Bridge, Edinburgh at 7.30 p.m.

#### Thursday 17 January

*Scottish Section:* "A trade journalist speaks his mind" by D. E. Eddowes (Polymers Paint and Colour Journal) to be held at St. Enoch's Hotel, Glasgow at 6.00 p.m.

#### Friday 18 January

*Irish Section*—to be announced.

*Midlands Section:* Annual Dinner Lecture "Who needs paint anyway?" by L. H. Silver (SPL Group) in the Birmingham Chamber of Commerce and Industry, PO Box 30, 75 Harborne Road, Birmingham B15 3DH at 6.30 p.m.

#### Saturday 19 January

*Scottish—Student Group and Eastern Branch:* Works visit to Continex International-carbon black manufacturers.

#### Monday 21 January

*London Section:* "Modern marine coatings and their application" by Mr J. C. Smith of the Central Dockyard Laboratory, at East Ham College of Technology, High Street South, London E6.

#### Thursday 24 January

*Thames Valley Section:* Paint Research Association's "Inventions" by Mr G. L. Holbrow at Beech Tree Hotel, Beaconsfield. Bucks at 7.30 p.m.

#### Friday 25 January

*Bristol Section:* "Printing ink developments" by Mr G. H. Hutchinson of Croda Polymers Ltd., Ink Division, to be held at the Royal Hotel, Bristol at 7.15 p.m.

#### Wednesday 30 January

*Manchester—Student Group:* "Organic pigments in paints—automotive finishes" by a lecturer from CTBA-GEIGY (UK) Ltd., Pigments Division, at the Manchester Literary and Philosophical Society at 4.30 p.m.

## February

#### Monday 4 February

*Hull Section:* Ladies Evening. "Wallpaper design and manufacture" by Mr B. C. Callaghan of ICI Wallpapers Ltd., to

be held at the Dorchester Hotel, Beverley Road, Hull at 7.00 p.m.

#### Thursday 7 February

*Newcastle Section:* "Profitability in the paint industry" by Mr L. H. Silver (SPL Group) to be held at the Royal Turks Head Hotel, Grey Street, Newcastle-upon-Tyne.

*Thames Valley—Student Group:* "The basic principles of Dispersymers" by Dr D. Pont of ICI Paints, to be held at the main Lecture Theatre, Slough College at 4 p.m.

#### Friday 8 February

*Manchester Section:* Instrument Evening, to be held at the Manchester Literary and Philosophical Society at 6.30 p.m.

(1) "The Gel Permeation Chromatograph" by Dr R. Ellis of The Walpamur Co. Ltd.

(2) "The Zeiss Goniophotometer" by Dr W. Carr, CIBA-GEIGY (UK) Ltd., Pigments Division.

*Scottish—Eastern Branch:* Burns Supper in the Lady Nairn Hotel, Edinburgh.

*Thames Valley Section:* Buffet Dance at Great Fosters, Egham.

#### Tuesday 12 February

*London Section:* "Recent developments in metal decorating" by Mr J. Holt and Mr M. Reid of the Research Division, Metal Box Company, to be held at Great Northern Hotel, King's Cross, London N1 9AN at 7.00 p.m.

*West Riding Section:* "Fast air drying vegetable oils" by Dr Gorko of Chemische Werke Huls AG, to be held at the Griffin Hotel, Leeds at 7.30 p.m.

#### Thursday 14 February

*Midlands Section—Trent Valley Branch:* "Developments at British Rail" by Mr D. M. Bishop to be held at British Rail School of Transport, London Road, Derby at 6.30 p.m.

*Scottish Section:* Talk on matters of topical interest by R. W. Kay of HM Factory Inspectorate, to be held in the St Enoch Hotel, Glasgow at 6.00 p.m.

## Forthcoming Events—continued

### Friday 15 February

*Irish Section:* "Works visit" to the Irish Glass Bottle Company.

*Midlands Section:* "We need paint" by R. J. King of Blundell Permoglaze Ltd., to be held at the Birmingham Chamber of Commerce and Industry PO Box 30, 75 Harborne Road, Birmingham B15 3DH at 6.30 p.m.

### Saturday 16 February

*Scottish Section—Student Group:* "Communications" by Mr D. Glenn of CIBA-GEIGY (UK) Ltd., to be held at St. Enoch's Hotel, Glasgow at 10.15 a.m.

### Tuesday 19 February

*London Section—Southern Branch:* "Wall

and ceiling decoration" by J. T. Icey, Iotex Products (Decorations) Ltd. to be held at the Pendragon Hotel, Southsea at 7.00 p.m.

### Wednesday 20 February

*Scottish Section—Eastern Branch:* "Computers in general" by Mr F. Lawry, Croda Polymers, to be held in the Carlton Hotel, North Bridge, Edinburgh at 7.30 p.m.

### Thursday 21 February

*Thames Valley Section:* "Corrosion: the culture of metallurgy" by Dr M. Clarke of Sir John Cass School of Science and Technology, to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks at 7.00 p.m.

### Friday 22 February

*Bristol Section:* Details to be announced.

*Newcastle Section:* Ladies Night. Five Bridges Hotel, Gateshead.

### Saturday 23 to Sunday 24 February

*London Section:* Weekend conference on "Colour" with participation from the French Association AFTPV at the Queen's Hotel, Hastings, Kent.

### Wednesday 27 February

*Manchester Section—Student Group:* "Paint additives" by a lecturer from Byk-Mallinckrodt to be held at the Manchester Literary & Philosophical Society at 4.30 p.m.

## Preliminary notice of the Annual General Meeting 1974 and one-day symposium

"OPTIMUM USE OF RESOURCES IN THE SURFACE COATINGS INDUSTRIES"

The Council wishes to announce as early as possible the preliminary arrangements which have been made for the holding of a one-day symposium in conjunction with the Paintmakers Association of Great Britain on the occasion of the Association's Annual General Meeting, 1974.

The Association's Annual General Meeting will take place at 5.30 pm on 26 June 1974 at University College London; a notice allowing for the nomination of the three Elective Members of Council will be sent to Members attached to the United Kingdom, Irish and General Overseas Sections early in the New Year and completed forms must be returned to the Association's offices not later than 1 May 1974. The notice will also give the first information concerning the one-day symposium which is being arranged at the same time on a techno-commercial subject, the general heading of which is "The Optimum Use of Resources in the Surface Coatings Industries". It is envisaged that the

first session will take place from 10.00 am to 12.30 pm. Following a Reception, Luncheon will be taken at 1.00 pm and the second session will be arranged from 2.00 pm to 5.00 pm. Dinner will be taken at 7.00 pm following a Reception at 6.30 pm. It is stressed that it is not necessary for those who wish to attend only the Annual General Meeting to register for the symposium. The Agenda, Voting Paper and Annual Report will be circulated to Members in the usual way approximately six weeks before the date of the Annual General Meeting. Council has arranged the inclusive charge for the symposium, which will include both Luncheon and Dinner together with preprints, Conference badges etc., to be £20 plus £2 VAT and forms will be circulated in due course in a forthcoming issue of the *Journal*. Further information, including details of papers to be given will be published in the *Journal*. The Paintmakers Association will be sending application forms to their members. Non-members, who are welcome to attend the symposium, can obtain application forms from the Association's offices in the New Year.

## Association Dinner Dance 1974

Early in the New Year members attached to the United Kingdom, Irish and General Overseas Sections will be 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 pm for Dinner at 7.30 pm and, on this occasion, there will be two short speeches only—a Welcome by the President and a Reply on behalf of the guests—so that dancing, to the Jerome Orchestra, can commence as soon as possible and continue until 1 am.

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.



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## Oil & Colour Chemists' Association

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Applications with details of age, qualifications and experience and the names of two referees, should be sent to the Deputy Secretary's Section (Ext. 2400), The University, Southampton SO9 5NH, not later than 17 January 1974. Please quote reference No. 136/R.

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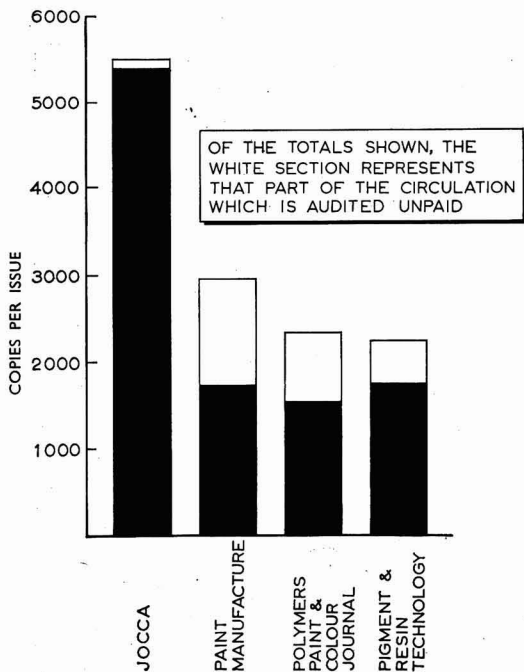
Japanese visitors to OCCA-26 wish to meet manufacturers of heat resistant paints at the Exhibition to discuss the possibility of production under license of the paint in Japan.

Manufacturers wishing to meet the Japanese visitors concerned should write at once to The Director & Secretary of the Oil and Colour Chemists' Association enclosing a sealed envelope marked "to be forwarded to the Japanese manufacturer" and listing on the outside of the envelope the names of any Companies to which the envelope should not be forwarded. All such envelopes will be forwarded to the Agent arranging the visit who will make the necessary arrangements for a meeting.

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



(Reference Audit Bureau of Circulations Reviews, Jan-Dec 1971)

For full details of advertising in this, and other Association publications, contact **C. A. Tayler**, Assistant Editor

### JOCCA Journal of the Oil and Colour Chemists' Association

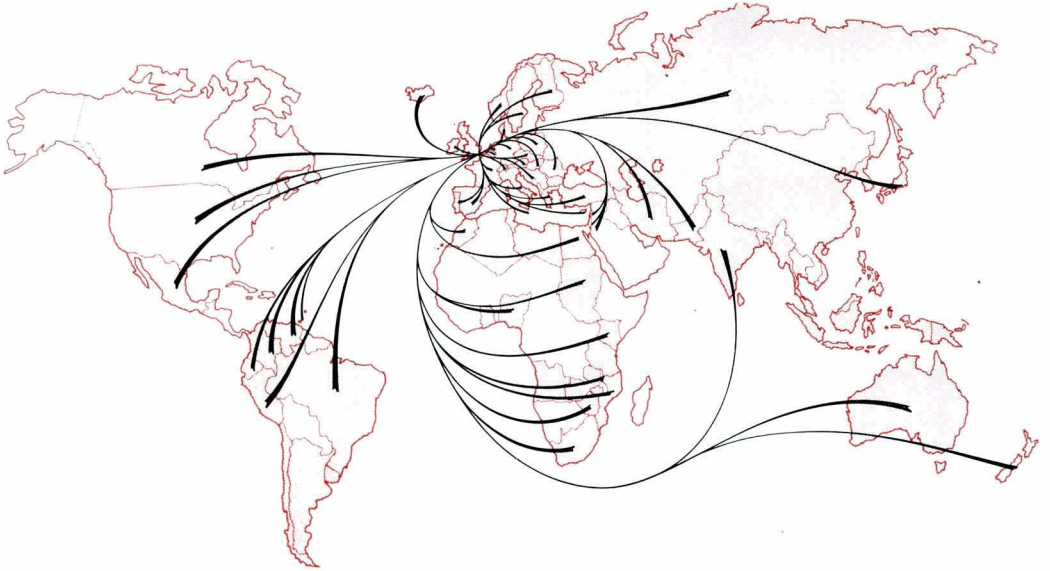
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For further information concerning advertisements in these or other OCCA publications, contact C. A. Tayler, JOCCA, Journal of the Oil and Colour Chemists' Association, Priory House, 967 Harrow Road, Wembley, Middlesex, England, HA0 2SF. Tel: 01-908 1086. Telex 922670 (OCCA Wembley).

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