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**JOURNAL OF THE
SOCIETY OF
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A comparison of Continental and British practice in the manufacture and use of emulsion paints on buildings *H. Lehmann*

Some factors affecting the opacity, colour and gloss of thermosetting powder coatings *J. K. Rankin*

Some physical properties of pigments — 1. Compaction under pressure *M. J. Smith*

The ANLAB colour system *A. C. Cooper and K. McLaren*

An investigation into the durability characteristics of masonry finishes *J. Boxall*

Rub-out stability



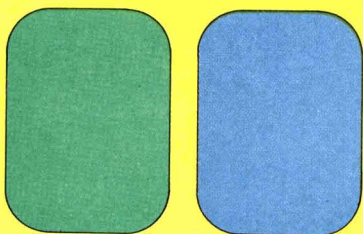
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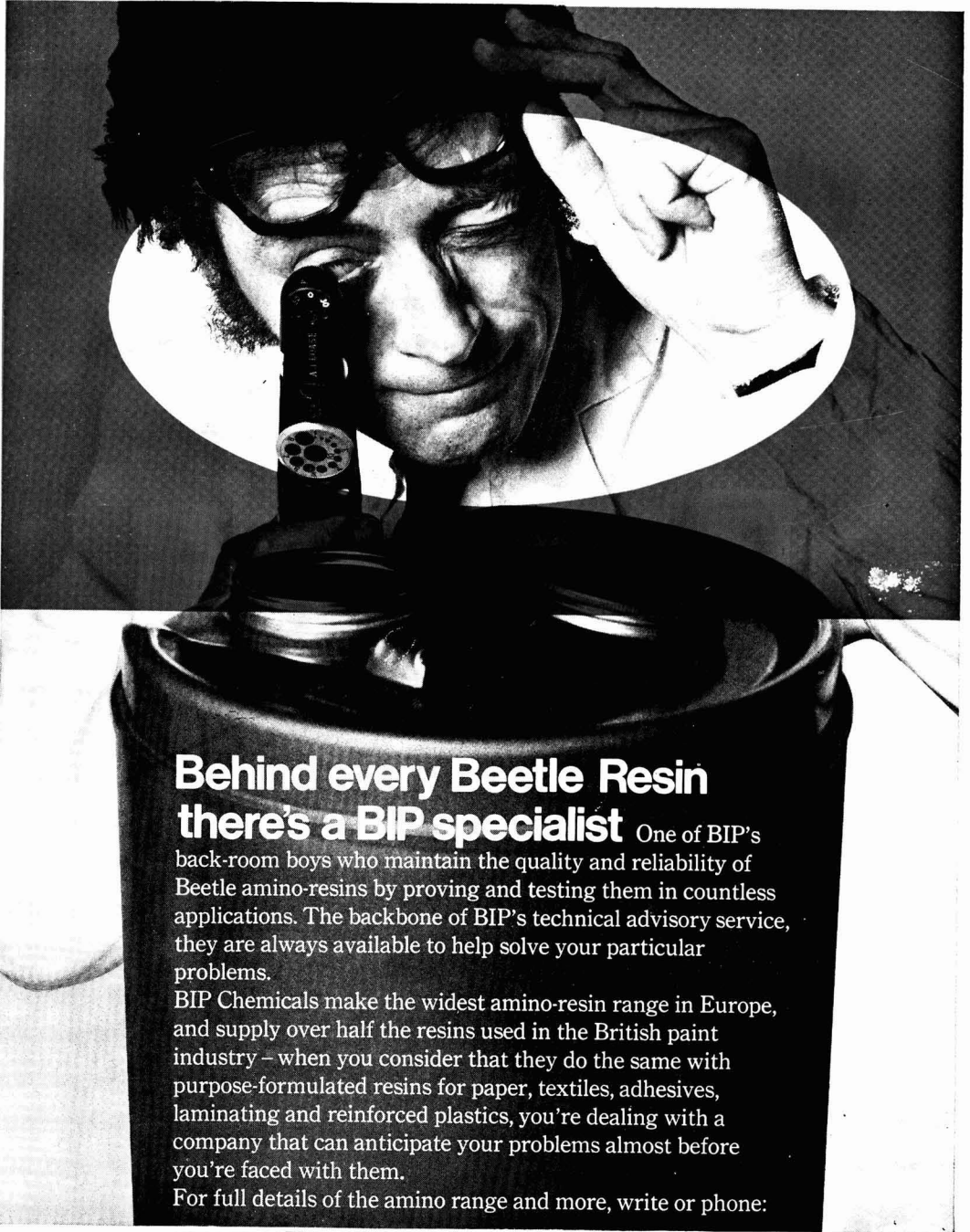
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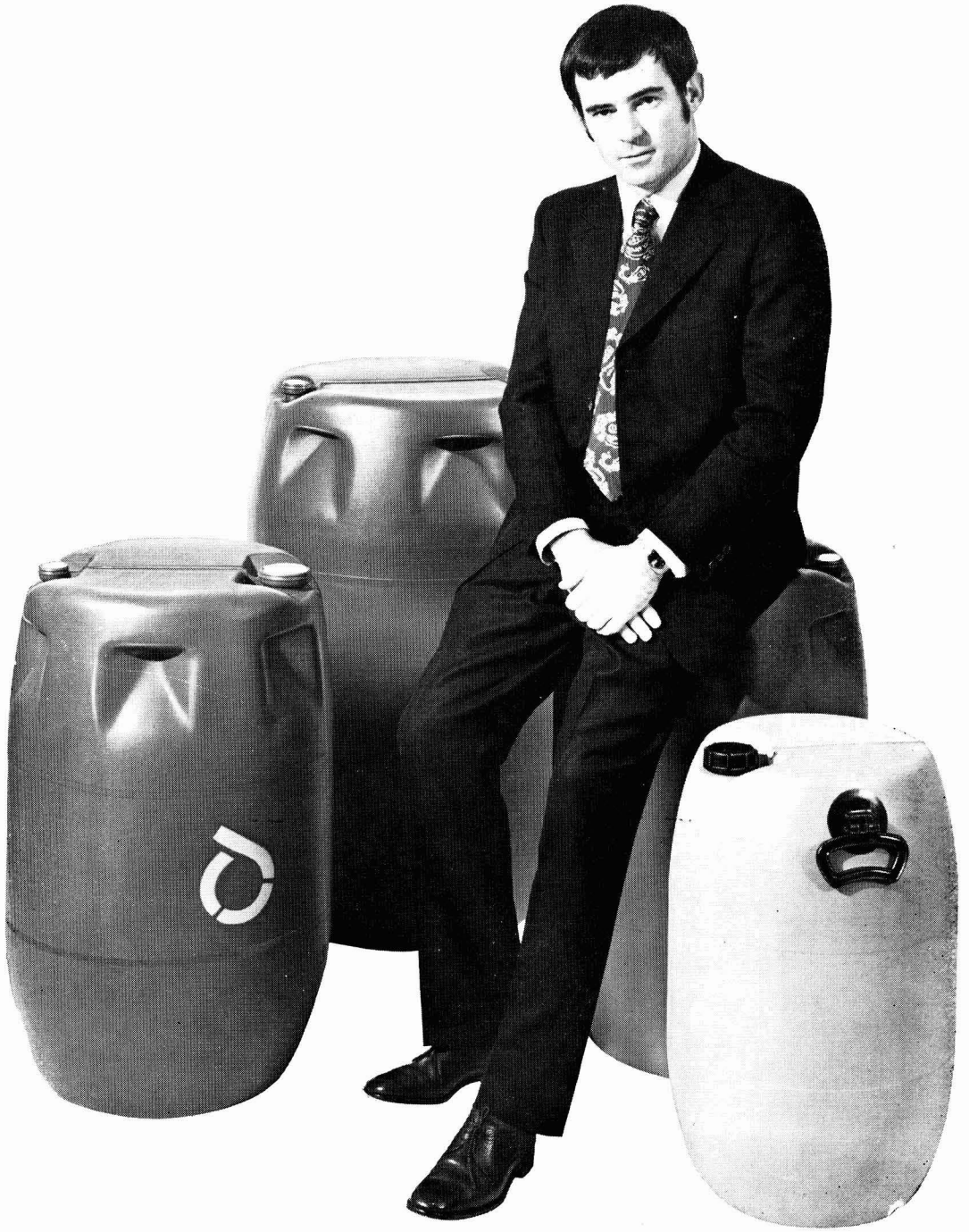
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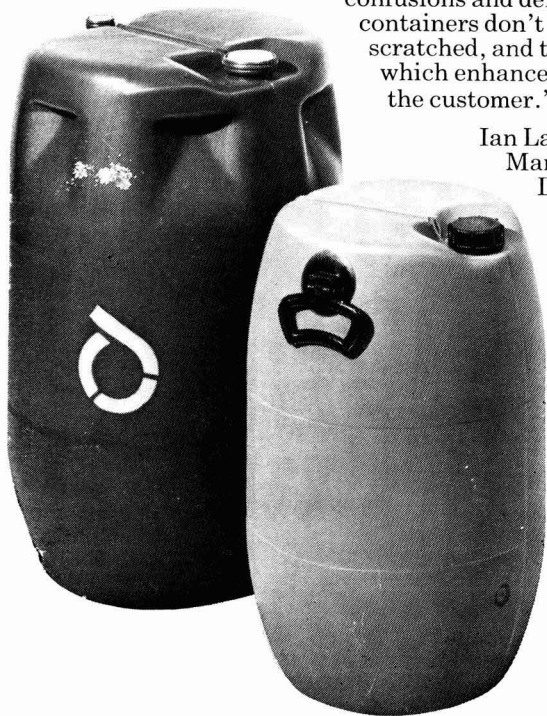
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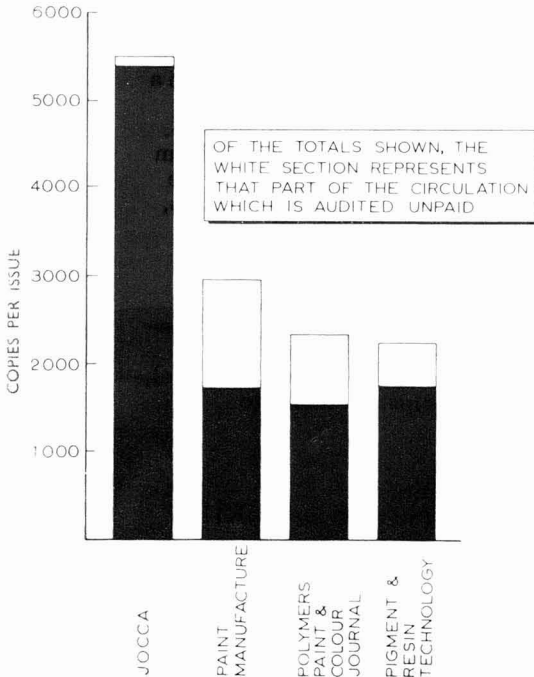
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Comparison of circulations of U.K. publications to the paint, printing ink and allied industries



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Transactions and Communications

A comparison of Continental and British practice in the manufacture and use of emulsion paints on buildings*

By H. Lehmann

Farbwerke Hoechst A.G., Frankfurt (M) Hoechst

Summary

The proportion of emulsion paints produced in the UK and Europe is very much lower than that in the USA; this is particularly so in the UK where emulsion paints amounted only to 17 per cent of the total production of paint in 1970. The reasons behind this are discussed, and factors affecting the different usages of paint in the UK and Europe outlined.

Even within Europe there is a large variance in paint usage, caused by great difference in climatic and social conditions. Even within Germany there are large differences, and the way in which the prevailing conditions have led to the development of particular formulations is shown.

Keywords

Types and classes of coating
emulsion paint

Une comparaison des pratiques britannique et européenne à l'égard de la fabrication et le mode d'emploi des peintures-émulsion pour bâtiments

Résumé

Au Royaume Uni et en Europe la quantité de peintures émulsion fabriquée en proportion à la production totale de peintures est fortement plus faible que celle aux Etats Unis; surtout au Royaume Uni où les peintures-émulsion ne reviennent qu'à de 17% de la production totale de peintures. On discute les raisons dont il s'agit, et on trace les grandes lignes des facteurs qui influencent les divers modes d'emploi qui se trouvent au Royaume Uni et en Europe.

Même dedans Europe il y a une grande variation des modes d'emploi des peintures à cause des grandes différences dans les conditions climatiques ou sociales. Même dedans l'Allemagne il y a des différences importantes, et l'on démontre comment les conditions prévalentes ont conduit à la mise au point des formulations spécifiques.

Vergleiche der Herstellungs- und Anwendungsmethoden von Dispersionsfarben für Bautenanstrich in Grossbritannien und auf dem Kontinent

Zusammenfassung

Der Produktionsanteil der im U.K. und in Europa erzeugten Dispersionsfarben ist sehr viel kleiner, als der in den U.S.A. erzeugten; besonders stark ist dies im U.K. der Fall, in dem Dispersionsfarben im Jahre 1970 nur 17% der Gesamtanstrichmittelproduktion ausmachten. Die Gründe hierfür werden besprochen, und die Faktoren, welche die verschiedenartige Verwendung von Anstrich im U.K. und dem Europäischen Festland beeinflussen, skizziert.

Selbst innerhalb des Festlands von Europa bestehen als Folge von klimatischen und sozialen Verhältnissen grosse Verschiedenheiten in der Verwendung von Anstrichfarben. Ja, selbst innerhalb Deutschlands gibt es grosse Unterschiede, und es wird gezeigt, auf welche Weise die herrschenden Umstände zur Entwicklung von besonderen Rezepten führten.

Сравнение континентальной и британской практики в производстве и потреблении эмульсионных красок на зданиях

Резюме

Пропорция эмульсионных красок изготавливаемых в Соединенном Королевстве и в Европе намного ниже чем в Соединенных штатах Америки; это в особенности относится к Соединенному Королевству, где в 1970-ом году производство эмульсионных красок составляло только 17% общего производства красок. Обсуждаются причины этого явления и рассматриваются факторы влияющие на различные применение

ния красок в Соединенном Королевстве и в Европе.

Даже в самой Европе наблюдается большое различие в потреблении красок, по причине весьма различных климатических и социальных условий. Даже в пределах Германии наблюдаются существенные различия и показан путь по которому преобладающие условия привели к развитию специальных формуляций.

Statistical information

The most difficult part in a review of the various markets for emulsion paints is not the conversion of US gallons to Imperial gallons, kilograms to pounds etc, but the lack of a uniform basis for the collection of the statistics.

| | Emulsion paints, tons | Alkyd paints, tons | Total, tons |
|---------|--------------------------|-----------------------|----------------|
| USA | 900,000 (54%) | 390,000 (28%) | 1,660,000 |
| UK | 110,000 (17%) | 350,000 (54%) | 650,000 |
| Germany | 360,000 (36%) | 300,000 (30%) | 1,000,000 |

From these figures it is seen that the consumption of the traditional oil- and alkyd resin-based paints still remains relatively large.

The production figures for 1970 have been quoted as:

* Presented at the London Section Symposium on 26 January 1972.

The figures for consumption of paint per head of population are shown below, and illustrated in Fig. 1.

| | kg |
|------------------------------------|------|
| USA | 19.5 |
| Sweden | 18.1 |
| West Germany | 17.4 |
| France | 13.8 |
| Norway | 13.0 |
| Belgium/Luxembourg/Denmark | 12.3 |
| Austria | 10.7 |
| Switzerland | 10.7 |
| Holland | 9.6 |
| UK | 9.3 |
| Italy | 7.3 |
| Portugal | 2.8 |

(Deutsche Farben Zeitschrift 1971, 25, No. 6, 292)

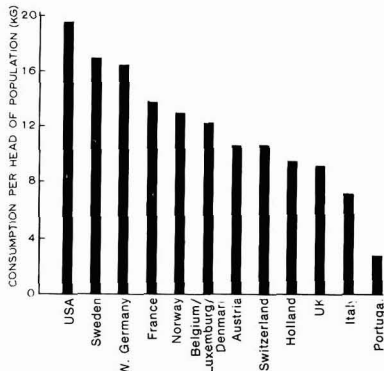


Fig. 1

Differences of European practice

The author's experience suggests that the following picture can be drawn. The Continental market, in general, seems to be more or less static technically; this is to say that the binding medium is seldom changed for an alternative one which may offer only a slight technical advantage. This is the reason why the change from homopolymer to copolymer emulsions was effected only slowly. Even today some countries, such as Belgium and Switzerland, still use homopolymer emulsions and from these produce paints which are even better in their overall performance than some formulations based on more sophisticated copolymer emulsions. Switzerland is of special interest because of its membership of EFTA, whilst being surrounded by Common Market countries, and because of its climatic conditions (high mountain chain and southern warm mediterranean climates). This situation leads to a quality conscious attitude.

Naturally, differences on the Continent can also be seen in other respects. For example, in France a change from one binder medium to another is achieved more easily than in a more conservative country such as the Netherlands. The climatic conditions can have an effective influence in this respect. For example, paints are produced which give a good performance in the climate of Southern France, but these paints may be sold also in Northern France where failures would occur due to the completely different and much more humid climate. This, together with other factors, is the reason why France has reverted to some extent to the use of solvent-based vinyl toluene/styrene media.

In Germany, development work carried out over the last four years has led to a new type of emulsion paint for interior application and, as a consequence of this, to the predominating use of styrene/acrylic emulsions. This, in the author's opinion, is because over-emphasis has been placed on wet rub resistance. Prior to this development, emulsion paints in Germany for interior use were made with an emulsion content of approximately 14 per cent. Today this content has dropped to between 8 and 10 per cent (equivalent to a PVC of up to 85 per cent). This specification was intended to set a lower level of performance for interior paints, but it must be stated that, as a consequence, not only have bad emulsion paints disappeared from the market, but the usage of better quality paints has decreased. Such specifications not only lead to a generally lower standard of quality in paints but also restrict the development of better quality materials.

Another factor that has a considerable influence on the development of paints is the method of selling by volume in the UK and by weight in most Continental countries. This difference influences the overall composition of the paint: for instance, in Germany the majority of emulsion paints are produced with a solids content of 60 to 70 per cent, and 10 to 20 per cent water is added by the user before application. In the UK the majority of paints have a solids content of about 50 per cent. This is the reason why there is usually a higher percentage of titanium dioxide in British paints, as they must have adequate opacity at the 50 per cent solids level. This also determines the viscosity of the emulsions used. In the UK, emulsions with a low viscosity and newtonian flow characteristics are the most suitable, whilst on the Continent high viscosity emulsions with some structure are preferred because of the practice of diluting the paints; consequently, this leads to the observation that British paints tend to exhibit better flow. Whether future developments will lead to the sale of emulsion paints on the basis of their spreading rates combined with good hiding power, brushability etc., cannot yet be foreseen.

These differences in the flow characteristics of the paints lead to differences in the methods of application. Paints with structural viscosity are predominantly applied by roller, whereas in the UK emulsion paints with good flow are mostly applied by brushing. Paints with structural viscosity have a better performance when applied by airless spray and owing to this the development of paints with structural viscosity has been encouraged.

The rather rapid development of "walling compounds" (textured finishes) on an emulsion basis having a high specific gravity of around 2 is certainly connected with the practice of selling by weight. As it is not necessary to take selling by volume into account, these compounds are applied at a rate of up to 3 to 4 kg m⁻², i.e. about 11b ft², and have rapidly become popular. The real reason for this development, particularly in Germany, is that the final thickness of high viscosity paints applied had become too thin because of over dilution and, consequently, the durability, weather resistance, resistance to cracking etc. were adversely affected. For this reason the change to thicker coatings was encouraged.

Naturally, there are also other differences that play some role. Amongst these is the type of substrate, particularly with exterior coatings. In Germany a plaster of lime, cement and sand is very frequently used. Only in Northern Germany are buildings with unplastered brick found, and these are not usually painted. In the Netherlands and Belgium, on the other hand, there are many brick buildings whose painting presents problems because of the jointings and differences in

the quality of bricks. Most wooden houses exist only in Scandinavia. This means that the Continent cannot be regarded as uniform in this field; even in Germany there are large differences—in Bavaria a pure lime/sand plaster is used, which is unknown in other parts of the country. To overcome unforeseen difficulties on account of the variation of substrates, primer-sealers based on polymer solutions have achieved a considerable share of the market. Most painting work, especially repair work, in Germany and the surrounding countries such as Belgium, the Netherlands and Switzerland, is carried out with a primer-sealer and two subsequent coats of emulsion paint.

The introduction of a new coating system is determined not only by its technical advantages; the appropriate craftsmen must be available in the countries concerned. Due to historical influences the trade often grew in various ways in different countries. There are marked differences even between Northern and Southern Germany. In Southern Germany, when a building is constructed, one man is needed to apply the plaster and another to perform the painting afterwards. Plastering techniques are known in Northern Germany only to a limited extent and any plastering has to be done either by a bricklayer or by a painter. New wall painting or plastering techniques based upon synthetic resins can be introduced easily in Southern Germany, but only with difficulty in the northern parts. The ever increasing

number of prefabricated houses being erected may change this situation.

Last, but not least, economic influences play an important part in the development of a market and the binding media chosen for a paint. Thus in the UK the acrylic-ester copolymer emulsions based on vinyl acetate/ethyl hexyl acrylate play a significant part. On the Continent, this particular combination is less common since, from the start of development, butyl acrylate has been used due to the fact that it was cheaper than ethyl hexyl acrylate. In the UK the reverse was true and the technically different properties of these monomers were of minor consideration.

It is hoped that the author's views will give some food for thought and that it will be realised that the Continent is far less uniform than might be thought from the outside. Certainly, due to the further reduction of Customs barriers, Europe and the UK will come closer together in many ways and paint production and consumption will move in the same direction. The mergers of many firms on an international basis will play an important part. It is to be hoped that the individuality of various regions will remain, so that uniformity in an unwelcome direction is not created, and that we shall still retain the colourful world of Europe as it exists today.

[Received 22 June 1972

Some factors affecting the opacity, colour and gloss of thermosetting powder coatings*

By J. K. Rankin

Tioxide International Limited, Billingham, Co. Durham

(J. K. Rankin is now with Vanguard Powder Coatings Limited, No. 4 Terrace Unit, Perry Avenue, Teesside Industrial Estate, Thornaby-on-Tees, Teesside.)

Summary

The surface appearance of any solid material can usually be described in terms of a number of visual characteristics such as colour, gloss, texture and uniformity. In organic coatings, these properties are influenced by the various components of the formula-

tion, the major ones being resin(s), pigment(s) and additive(s).

An attempt is made to consider some of these components in relation to their influence on opacity, colour and gloss of epoxy and acrylic powder coatings.

Keywords

Types and classes of coating

powder coating

Prime pigments and dyes

titanium dioxide

Binders, resins etc.

thermosetting resin

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

colour

gloss

opacity

Quelques facteurs qui exercent une influence sur l'opacité, la couleur et le brillant de revêtements en poudre thermo-durcissables

Résumé

En général, on peut décrire l'apparence de la surface de chaque solide sous termes d'une série de caractéristiques visuelles telles que couleur, brillant, texture et uniformité. Dans le domaine de revêtements organiques, ces propriétés sont influencées par les divers constituants, dont les plus importants ce sont résine(s), pigment(s), et adjuvant(s).

On fait une tentative pour considérer certains de ces constituants au point de vue de l'influence qu'ils exercent sur l'opacité, la couleur, et le brillant des revêtements en poudre, soit époxyde, soit acrylique.

Einige Opazität, Farbe und Glanz wärmehärtbarer Pulverlacke beeinflussende Faktoren

Zusammenfassung

Das Aussehen irgendeines Feststoffes kann gewöhnlich durch eine Anzahl von mit dem Auge wahrnehmbarer Charakteristika, wie z.B. Farbe, Glanz, Textur und Gleichmässigkeit beschrieben werden. Bei organischen Beschichtungen werden diese Eigenschaften durch die verschiedenen Komponenten der Rezeptur

beeinflusst, deren wichtigsten Harz(e), Pigment(e) und Zusatzmittel sind.

Es wird versucht, einige dieser Komponenten in Verbindung mit ihrem Einfluss auf Deckkraft, Farbe und Glanz in Epoxy- und Akrylharz-Pulverlacken zu betrachten.

Некоторые факторы влияющие на непрозрачность, цвет и лоск термореактивных порошковых покрытий

Резюме

Поверхностный вид любого твердого материала может быть обычно описан при помощи ряда визуальных характеристик как например цвет, лоск, текстура и однородность. В органических покрытиях, на эти свойства влияют различные компоненты формуляции из которых главными являются смолы, пигменты и примеси.

Делается попытка рассмотреть некоторые из этих компонентов, в зависимости от их влияния на непрозрачность, цвет и лоск эпоксидных и акриловых порошковых покрытий.

Pigmentation of epoxy and acrylic powder coatings with titanium dioxide

Properties and types of pigment

Pigments currently used in solvent-borne conventional coatings may be used also for thermosetting powder coatings,

provided they are chemically inert, fast to light, and heat resistant.

In white powder coatings, titanium dioxide is universally used for pigmentation of the polymer and for reducing colours when pastel tints are required, because it gives the brightest and cleanest tints obtainable. The titanium dioxide manu-

* Based on a paper given at the 3rd International Conference on Powder Coatings, London 1972, sponsored by Industrial Finishing & Surface Coatings, to whom due acknowledgement is made.

facturer has developed rutile and anatase grades of the pigment, both of which can now be more easily incorporated into polymers than was possible a decade ago. These names denote the fundamental crystal structures of the two types. The rutile pigments have higher specific gravity, refractive index, and reflectivity, and are more weather resistant than the anatase grades, which, although lower in tint reducing power, have a "bluer" tone. Some typical properties of rutile and anatase titanium dioxide are given in Table 1.

Table 1
Some properties of titanium dioxide

| Property | Rutile | Anatase |
|-------------------------------|-----------|-----------|
| Density (g ml ⁻¹) | 4.0 | 3.8 |
| Refractive index | 2.71 | 2.55 |
| Particle size (μm) | 0.17-0.23 | 0.14-0.18 |
| Hardness (Mohs Scale) | 6-7 | 5-6 |
| Reflectance (MgO = 100%) | 98.5 | 98.0 |
| Tint-reducing power | 1550-1850 | 1300-1350 |
| Chemical reactivity | Inert | Inert |

A brief note on the manufacturing processes may be helpful in placing the various grades in the proper perspective. Titanium dioxide pigments are made by either the sulphate process or the more recently developed chloride process. In the former, the ilmenite ore is extracted with sulphuric acid, and titanium hydroxide is precipitated from the extract, filtered, washed repeatedly, and finally calcined to the dioxide at a temperature between 800° and 1000°C. In the chloride process the ore (mineral rutile) is reacted with gaseous chlorine to produce the liquid titanium tetra-chloride which, after purifying by distillation, is burnt in oxygen to obtain titanium dioxide directly.

Irrespective of the method of manufacture, the crude titanium dioxide thus produced requires further refinement and after-treatment to develop pigmentary properties to suit it for various industrial uses. According to the after-treatment the pigments can be classified into four types.

Type I—Dry-milled pigments

The simplest form of processing consists of dry-grinding the crude pigment and selecting the desired range of particle size. The products are known as "dry-milled" or "untreated" grades.

Type II—Coated and refined pigments

These pigments are manufactured by dispersing those of Type I in water and, after wet-milling to remove oversize particles, precipitating between 1 per cent and 3 per cent of other oxides such as silica and alumina on the pigment surface. The product is then filtered, dried, and micronised.

Sometimes an organic compound is then applied to the pigment. A wide variety of such compounds can be used, their function being to promote easier dispersion of the pigment in the polymer.

Some grades have no inorganic coating or only a very small amount, and these show extremely low moisture pick-up. This is particularly useful when the moisture content of the final pigmented polymer is to be kept at a minimum.

Type III—Coated and super-refined pigments

The "super-refined" pigments are of fairly recent introduction, and differ from those of Type II only in their more extended wet-milling and closer particle size segregation, improvements ensuring a minimum of coarse aggregates. These grades are more expensive than Type II pigments, but generally are somewhat superior in dispersion, opacity, gloss, and tint-reducing power. Organic coatings may be applied to pigments of this type for the same reasons as before.

Type IV—Heavily coated and refined pigments

Pigments of this type have also been introduced fairly recently. They are manufactured from those of Type I, but a more voluminous coating (exceeding 10 per cent of the pigment by weight) is applied. These grades, specially developed for latex paints, give superior opacity, and a much more matt film than those of Types II and III. Normally the pigments are not organically treated.

Differences between the types, and the influence of crystal structure, are brought out in Table 2, from which it will be seen that the uncoated grades have the lowest surface area, and, for a given crystal type, the lowest tint-reducing power. Increasing the amount of the coating on the titanium dioxide crystal increases the surface area and the oil absorption. Electron micrographs of the uncoated, lightly coated, and heavily coated rutile grades (Types I, II and III, and IV respectively) are shown in Fig. 1.

Table 2
Pigment properties according to type and crystal structure

| Property | Anatase | | Rutile | | |
|--|---------|-----------|-----------|------------------|---------|
| | Type I | Type II | Type I | Types II and III | Type IV |
| Density, g ml ⁻¹ * | 3.85 | 3.75 | 4.20 | 4.05 | 3.70 |
| Surface area, m ² g ⁻¹ † | 8 | 12-15 | 8 | 12-15 | 20-30 |
| Oil absorption‡ | 19-20 | 24-25 | 15-16 | 18-20 | 25-30 |
| Tint-reducing power | 1300 | 1300-1350 | 1500-1600 | 1700-1850 | 1500 |
| TiO ₂ content, % | 98 | 95-96 | 94-96 | 90-94 | 82-91 |

* Measured by Method 1, BS 3483 : 1962—results quoted to nearest 0.05.

† Measured by the Strohlein area-meter.

‡ Palette-knife method based on BS 3483 : 1962. Expressed in grams oil per 100 grams pigment.

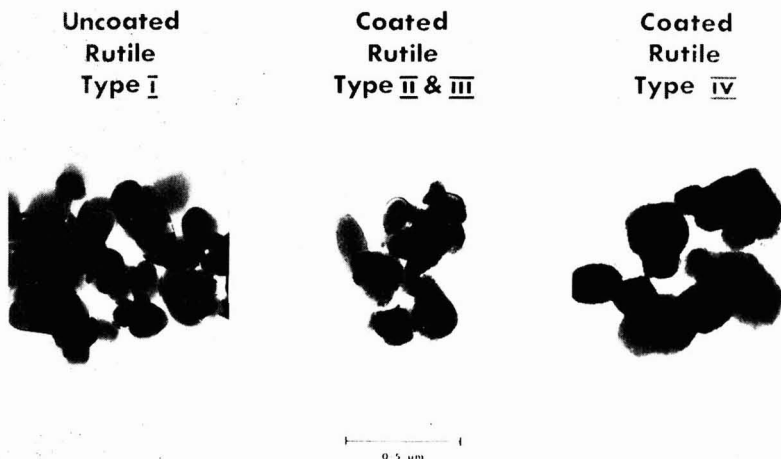


Fig. 1. Electron micrographs of titanium dioxide

Opacity

Because rather thicker films can be applied with powder coatings (40-100 μ m compared with 25-35 μ m dry film thickness for solvent-thinned finishes) they give satisfactory opacity with a smaller proportion of pigment. For example, the pigmentation range of white industrial solvent-borne coatings is from 16-22 per cent PVC (d)*, which is given by a pigment:binder ratio (P:B) of 0.8:1-1.1:1. For thermosetting powder coatings the pigmentation may be between 8 per cent and 36 per cent PVC (d), (P:B 0.3:1-2.1:1).

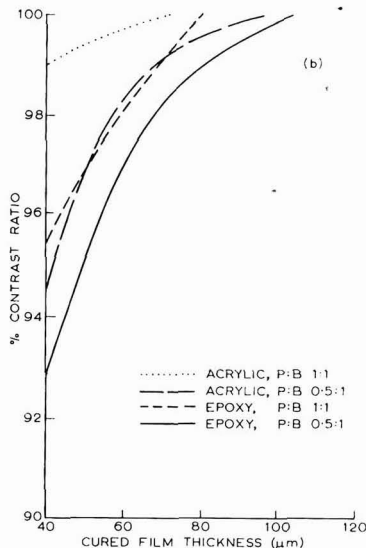
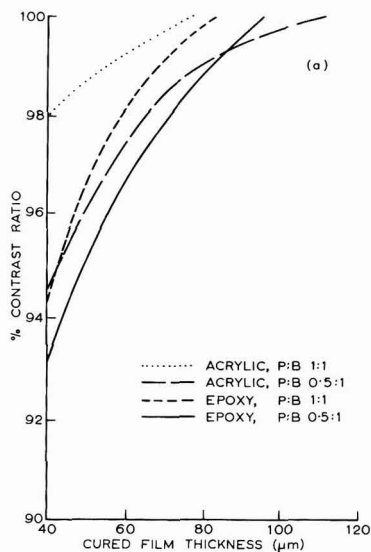


Fig. 2. Variation in contrast ratio with film thickness of acrylic and epoxy powder coatings (a) pigmented with rutile Type II, (b) pigmented with rutile Type IV

However, owing to the high cost of the epoxy resin it is economically advantageous to use the highest pigment or pigment/extender content consistent with the other film properties required, such as gloss, colour, flexibility and adhesion.

Fig. 2 shows the opacities at various dry film thicknesses of simple epoxy and acrylic powder coatings pigmented with Type II and Type IV titanium dioxide at various pigment:binder ratios.

* Pigment volume concentration in a dry film:

$$\text{PVC (d) \%} = \frac{\text{volume of pigment}}{\text{volume of (pigment + non-volatile binder)}} \times 100$$

Fig. 3 shows the opacity at various dry film thicknesses of an epoxy and an acrylic powder coating pigmented with Type IV titanium dioxide at various pigment: binder ratios.

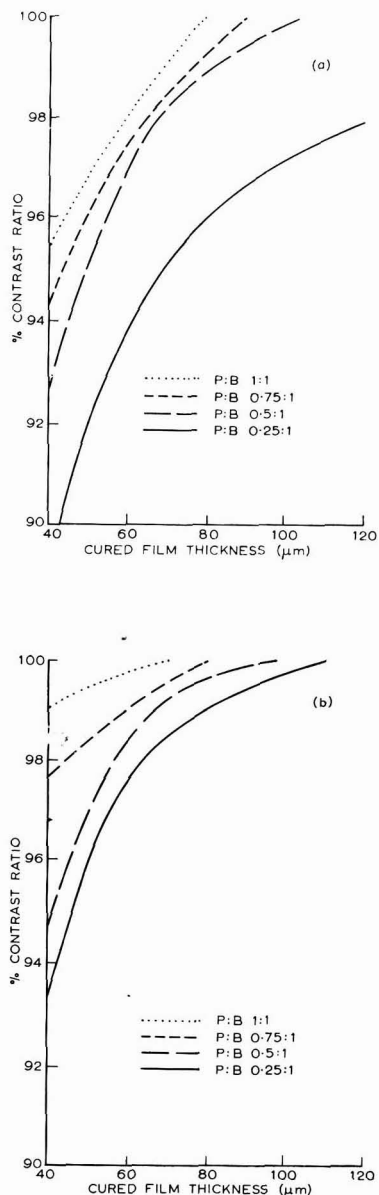


Fig. 3. Variation in contrast ratio with film thickness of a powder coating at various pigment:binder ratios: (a) epoxy, (b) acrylic

For these determinations the following simple formulations and procedures were used.

Standard test formulation for an epoxy powder coating

| | Pt by wt |
|--|----------|
| Epoxy resin (equivalent epoxy weight 900-1000, mp 95°-105°C) | 100.0 |
| Titanium dioxide | 75.0 |
| Flow-control agent, "Modaflow" (Monsanto Ltd.) | 0.4 |
| Substituted dicyandiamide hardener (No. 4, Table No. 3) | 4.0 |

The epoxy resin was first reduced to size, dry-blended in a high-speed mixer with pigment, hardener and flow-control agent and then extruded. The ratio of epoxy resin to curing agent and to flow-control agent was kept constant, only the pigment content was varied.

Standard test formulation for an acrylic powder coating

| | Pt by wt |
|--|----------|
| Acrylic resin | 100.0 |
| Titanium dioxide | 75.0 |
| Flow-control agent, "Modaflow" (Monsanto Ltd.) | 0.5 |

The acrylic resin was dry-blended with pigment and flow-control agent in a high-speed mixer, and then extruded. The ratio of acrylic resin to flow-control agent was kept constant, only the pigment content being varied.

After cooling, each mass was kibbled, pin-disk-milled to <math>< 75\mu\text{m}</math>, and electrostatically sprayed on to solvent-degreased phosphated* 20-gauge steel panels† to give a range of film thicknesses and finally, the panels were stoved for 15 minutes at 180°C and 20 minutes at 220°C for the epoxy and the acrylic systems respectively.

Because it was impossible to obtain a completely smooth coating of uniform thickness every time, each panel was scanned with an electromagnetic thickness gauge in order to find uniform areas of film. The reflectance of each uniform area was measured on a Harrison Colorimeter equipped with a green filter, and the contrast ratio calculated as follows:

$$\text{Contrast ratio} = \frac{\text{Reflectance over grey substrate}}{\text{Reflectance at infinite film thickness}} \times 100$$

Although contrast ratio is normally expressed as the quotient of reflectance over black divided by reflectance over white, for convenience it was assumed that a grey phosphated substrate could be used instead of the black substrate for comparative purposes. It was also demonstrated that, at high film thicknesses, reflectance became independent of thickness, and so reflectivity at infinite film thickness was used instead of the reflectance over white. Contrast ratio was then plotted against film thickness.

Two points are immediately apparent from a study of Figs. 2 and 3. The heavily coated grade of titanium dioxide, Type IV, gives higher opacity than the Type II pigment both in the acrylic and in the epoxy system. This is the opposite of what occurs in industrial solvent-based stoving finishes.

The second point is that the acrylic film, at all film thicknesses, gives higher opacity than the epoxy film.

* Grey phosphate coating to BS 3189: Class C 1959.

† Steel panels to BS 1449: Part 1B 1962.

Fig. 4 shows microtomed sections of the acrylic and epoxy films viewed through an electron microscope, and shows that differences in pigment distribution may be responsible for the superior opacity of the acrylic compared with the epoxy coating.

The acrylic polymer has been pigmented with Type II titanium dioxide at three pigment : binder ratios (0.25, 0.5, and 0.75:1) and the epoxy polymer at 0.75:1. It can be seen that there is a much more even distribution of the

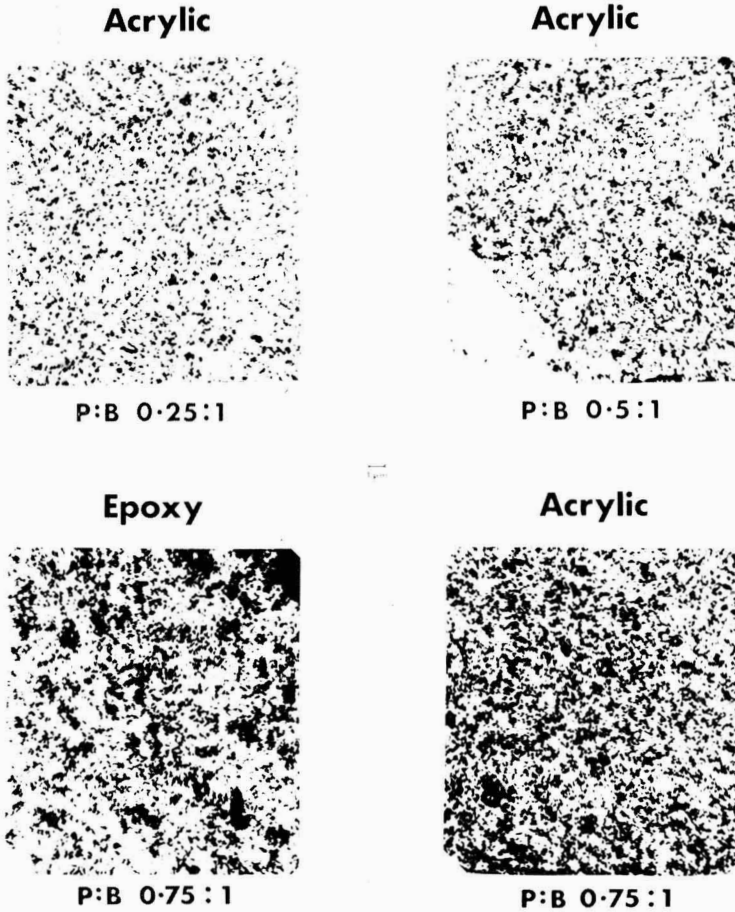


Fig. 4. Electron micrograph sections of TiO_2 pigmented coatings



Fig. 5. Montage of electron micrographs of TiO_2 pigmented acrylic powder coating

titanium dioxide particles through the acrylic film, although there are some pigment aggregates, but these are few compared with those in the epoxy film. The greater degree of pigment aggregation in the epoxy film may account for its opacity being lower than that of the acrylic film.

Fig. 5 shows a montage of the pigmented acrylic film (0.75:1 pigment:binder) at 100 μ m film thickness. It can be seen that there is a fairly even distribution of the pigment throughout the whole section of the film.

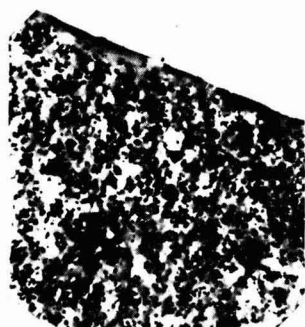
Another interesting aspect of the pigmentation of powder coatings is the difference in the distribution of pigment at the surface of the cured film compared with that of solvent-based industrial finishes.

In conventional pigmented solvent-based gloss systems (either air-drying or stoving), the amount of titanium dioxide pigment distributed at the paint film surface is very much less than in the bulk of the film. The only method of showing directly the existence of a surface "clear layer" is to cut a section of the film and examine it microscopically.

From the micrographs obtained, measurement establishes the shortest distance between the film surface and a given pigment particle surface. The diameter of each crystal can be measured with a transparent sizing graticule. Details of this technique have been published^{1, 2}.

Fig. 6 shows surface cross-sections of three pigmented solvent-based industrial finishes—a thermosetting acrylic, a stoving alkyd, and a polyurethane, and compares them with an acrylic powder coating. As may be seen, the top 0.1-0.15 μ m of the stoving alkyd and of the thermosetting acrylic film is deficient in pigment, i.e. has a "clear resin layer," and in the polyurethane the thickness of this clear layer is 0.3 to 0.5 μ m.

These are not true "clear layers" entirely devoid of titanium dioxide, but show a statistical distribution of pigment with a decreased probability of pigment particles occurring closer to the surface. Below this layer of reduced concentration lies another, roughly 0.15-0.2 μ m from the film surface in the thermosetting acrylic and in the stoving alkyd, containing a higher concentration of pigment particles than the bulk of the film.



Stoving Alkyd



Thermosetting Acrylic



Polyurethane



Acrylic Powder Coating

1 μ m

1 μ m

Fig. 6. Electron micrograph section of TiO₂ pigmented films

It is significant that the excess of particles in this second layer roughly equals the deficiency in the surface layer when the numbers in each layer are compared with that in the remainder of the film.

This suggests that during application of the solvent-based systems the pigment is uniformly distributed throughout the paint and during film formation, i.e. solvent evaporation and polymer cross-linking, the titanium dioxide particles close to the surface experience a force driving or pulling them away from the surface. It is also interesting that the titanium dioxide particles close to the film surface have a smaller size than average; in other words, the larger particles show a greater tendency to move away from the film surface in these systems.

However, the acrylic powder coating film, as can be seen from Fig. 6, has no "clear layer." This is understandable because the mobility of the components in a powder coating during the fusion and cross-linking stage is much smaller than in a solvent-based coating, so that the pigmented polymer particles in a powder coating probably remain very close to where they were deposited.

Colour

Whether the powder coating is white or a pastel colour, if the cleanest or bluest tone is required an anatase pigment would normally be selected. However, several grades of rutile titanium dioxide have undertones approaching the bluish white given by the anatase grades, and they also give a high gloss, which the anatase grades cannot do. These are the fine-crystal rutiles. Where a cream undertone is required, a rutile pigment of normal crystal size would be selected.

These undertone differences are illustrated in Figs. 7 and 8. The curves were produced on a Beckman DK2A recording spectrophotometer, and show the percentage of light of different wavelengths reflected by epoxy and acrylic powder coatings pigmented with four different titanium dioxide

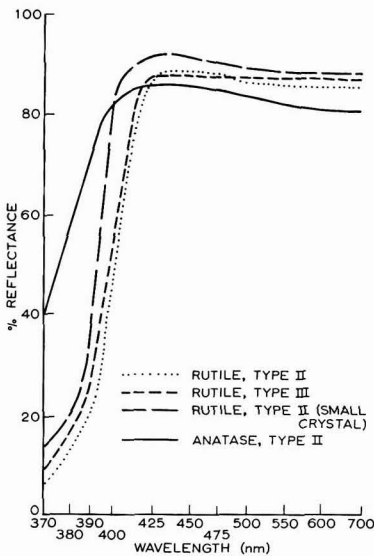


Fig. 7. Spectral reflectance of TiO_2 pigments in an epoxy powder coating

types, the reflectance from a smoked-magnesium-oxide standard being taken as 100 per cent. The film thickness used in these determinations was $100\mu\text{m}$, and the stoving conditions were appropriate to the polymer species.

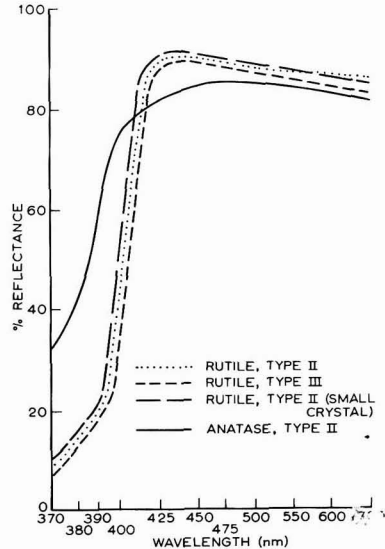
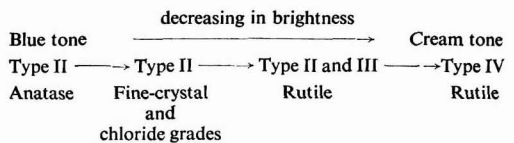


Fig. 8. Spectral reflectance of TiO_2 pigments in an acrylic powder coating

The higher reflectance of the fine-crystal rutile at the blue end of the spectrum, between 410nm and 460nm, indicates that this grade gives a bluer, cleaner undertone than the normal-crystal rutile.

The scheme below indicates the general change in tone to be expected of the various types of pigment in thermosetting powder coatings.



Gloss

Surface conditions

An ideal industrial coating would be one giving a smooth, glossy, bright, uniform surface of good durability.

Gloss is given by mirror-like reflection of light from surfaces. A glossy pigmented organic coating may be taken as consisting of a layer of pigment particles surrounded and covered by a smooth film of vehicle or polymer. Incident light is partially reflected by the polymer surface, and the light penetrating the film surface is scattered or absorbed by the pigment particles. Thus light is reflected in all directions at varying intensities, which can be physically measured.

The light reflected by the air/polymer interface will, if the surface is smooth, travel in one direction only, and give rise to a high gloss. If the surface is not optically smooth, a

portion of the surface-reflected light will be scattered, producing a less glossy appearance.

With powder coatings it is much more difficult to achieve a smooth, glossy finish than it is with a properly formulated, carefully applied, and correctly baked solvent-based coating. One of the biggest difficulties with a powder coating is to arrive at a suitable balance giving satisfactory flow and "sharp-edge cover" during the fusion and cross-linking process. With most currently available commercial powder coatings, this compromise invariably gives a degree of "orange peel," which detracts from the high gloss that would otherwise be expected.

For the formation of a coherent film from a thermosetting powder, a reduction in viscosity of the polymer must occur during the baking cycle. Little or no work has been done on the viscosity changes taking place during the baking of a powder coating, no doubt because of experimental difficulties. Data on the viscosity of liquids at different temperatures have been obtained with instruments such as the Weissenberg rheogoniometer, but it would be difficult to adapt this instrument to the measurement of viscosity changes taking place during the cross-linking of a thermosetting coating.

In baking, not only the flow of the polymer during transition from the solid to the liquid phase may vary, but also the rate at which cross-linking occurs. If cross-linking is rapid, the flow-out time will be short and a high degree of orange peel will result. This gives low gloss but good sharp-edge cover. With a slower curing rate, flow-out will be prolonged, and smooth, even films of high gloss will result, but sharp-edge coverage will necessarily be poor. Normally a powder coating is a compromise between these two extremes.

However, the orange peel effect is not the only cause of variability of gloss in powder coatings. Many films contain, to varying degrees, a mass of very small "pinholes", and these make a great difference to the gloss. The appearance has been referred to as "peppery," as "haze-gloss," or as "seeding-out." This will be considered in more detail in the section on surface texture.

Effect of curing agents on the gloss of an epoxy coating

To show just how important it is to consider the other components in an epoxy powder coating for their influence on gloss, the same test formulation and manufacturing technique were used as for the colour and opacity determinations with a range of hardeners. Table 3 indicates the gloss obtained

with five different hardeners, and shows quite a wide variation.

Gloss in relation to surface texture

It is clear that the surface texture of a powder coating influences gloss, and the preceding section shows that there is a marked difference in the gloss given by the various hardeners used to cross-link the epoxy resin. In an attempt to define the "orange peel" and "pinholing" effects more objectively, the surface texture or roughness (i.e. that quality of a surface caused by small departures from its general geometric form, which, occurring at regular or irregular intervals, tend to form a pattern or texture on the surface) of a series of cured epoxy powder coatings was assessed by drawing a replica of each surface using a Talysurf instrument.

The epoxy powder film surfaces examined were those using hardeners Nos. 2, 3, 4, and 5 as in Table 3. The linear magnification was X 20 of the surface under examination. Fig. 9 records the profiles of the surfaces.

The terms "primary texture" and "secondary texture" as defined in BS 1134, 1961 can be used to describe the surface characteristics of a powder coating as shown below.

Roughness (primary texture). This is due to the small irregularities in the surface.

Waviness (secondary texture). This is the component upon which the roughness is superimposed.

Study of the Talysurf traces shows that all the films exhibited waviness giving the effect known as "orange peel" and ranging in length from 2 to 4mm and in depth from 2 to 8 μ m.

Three of the four films also exhibited roughness due to pinholing, which ranged in length from 100 to 200 μ m and in depth from $\frac{1}{2}$ to 1 $\frac{1}{2}$ μ m.

When these Talysurf traces are correlated with the gloss figures it is at once seen that the film exhibiting only waviness, i.e. No. 4, gives a reasonably high gloss, whereas the greater roughness of Nos. 2, 3, and 5 corresponds to poorer gloss.

Four commercially available white epoxy powder coatings were also examined for film gloss and surface roughness. The powders were applied by spraying through an electrostatic gun at 90kV on to the phosphated steel panels as already

Table 3
Influence of hardener on gloss of a cured epoxy coating

| Hardener | Chemical type | Curing time/temperature | Gloss | |
|----------|-----------------------------------|-------------------------|-----------------|---------|
| | | | Rutile pigments | |
| | | | Type II | Type IV |
| 1 | Dicyandiamide* | 30 min/200°C | 60 | 60 |
| 2 | Accelerated dicyandiamide | 10 min/180°C | 57 | 65 |
| 3 | Accelerated dicyandiamide | 10 min/180°C | 55 | 65 |
| 4 | Substituted dicyandiamide | 12 min/180°C | 86 | 90 |
| 5 | Boron trifluoride | 30 min/150°C | 56 | 58 |

* Note—Cured coatings containing the unmodified dicyandiamide show a pronounced yellowing compared with coatings cured with the other hardeners; this is in part due to the higher temperatures required when using it.

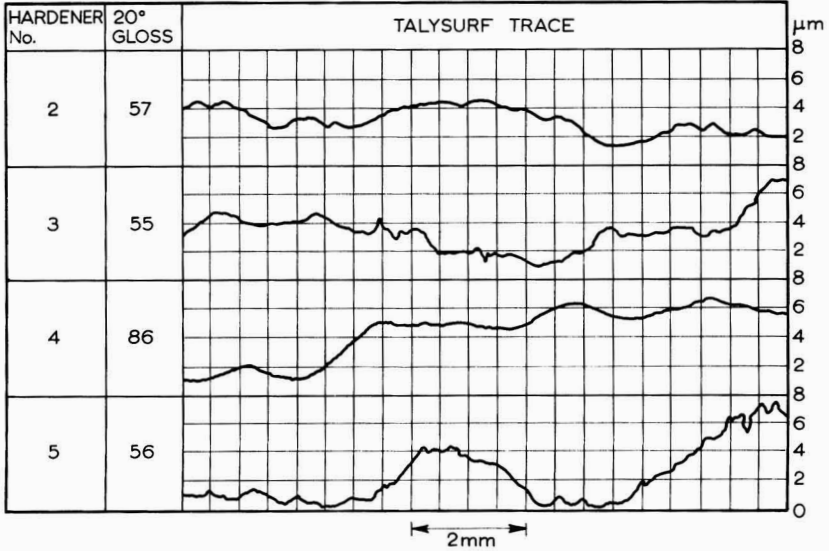


Fig. 9. Talysurf traces and gloss readings of experimental epoxy powder coatings containing different hardeners

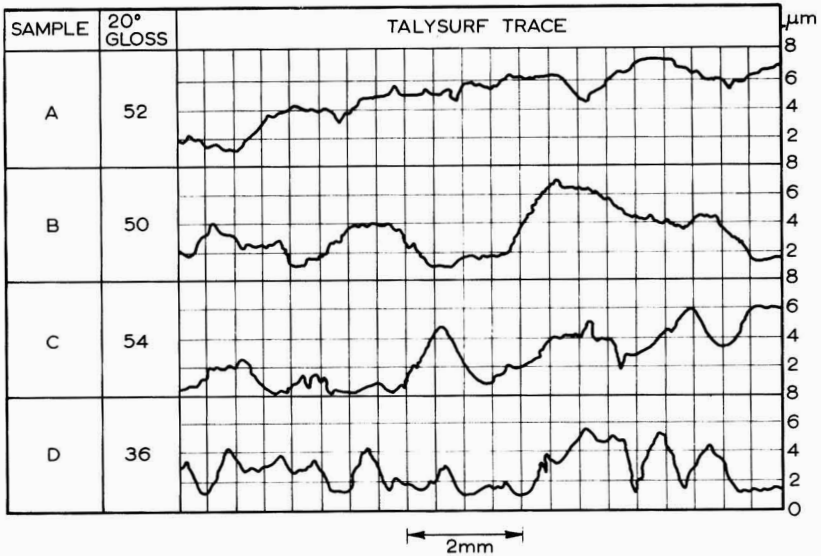
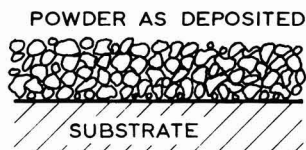


Fig. 10. Talysurf traces and gloss readings of commercial white epoxy powder coatings

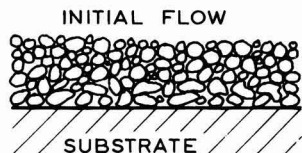
described, to give a cured film thickness of about 100µm after following the manufacturer's recommended curing cycle. Gloss and Talysurf measurements were then made as before and the results are recorded in Fig. 10, confirming that the rougher the surface the lower the gloss.

Mechanism of film formation of a powder coating

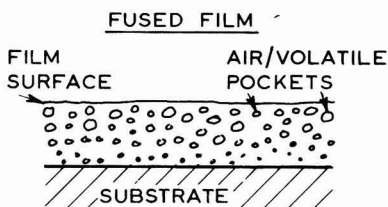
The transition of a powder coating from the "as applied" powder to the cured coating may be envisaged as taking place in the sequence shown below.



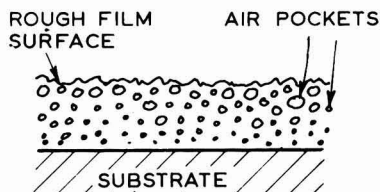
The heterogeneous mixture of powder, as deposited, forms an irregular temporary structure comprising load-bearing columns and vaults, which protect small voids.



On application of heat there is initial polymer deformation towards spherical particles, then a fusing of these with adjacent particles, and perhaps consequent squeezing of the small voids to form fewer larger ones.



The polymer particles begin to flow out to form the continuous phase whilst the entrained air/volatile pockets expand and tend to rise to the surface. If the film surface is still fluid, some or most of the pockets will erupt through the surface and the resultant hollow will be partially or wholly filled by the subsiding fluid polymer surrounding the crater, depending on the mobility of the polymer at that particular stage in the curing cycle.



However, if the rate of polymer cross-linking is rapid, then the film may set before many of the pockets have had time to diffuse from the coating. Also, there is the possibility that the coating will set just as some of the pockets escape from the film or before the coating surface has had time to flow back after erupting, so producing a rough surface with low gloss.

A series of experiments confirmed the sequence outlined above, and established the principal factors influencing the roughness of powder coatings. Each component of an epoxy powder coating was examined in succession as follows.

Epoxy resin

The epoxy resin (equivalent weight 900-1000, mp 95°-105°C) was ground by milling in three different mills as indicated

below, and the resultant powder was passed through a 200-mesh copper sieve to give a <math><75\mu\text{m}</math> cut.

- Chipper mill
- Hammer mill
- Pin-disk mill

Each powder was examined microscopically under a magnification of $\times 600$. The resin particles were all irregular in shape, and there did not appear to be any significant differences either in shape or particle-size distribution between the products from the three machines.

Particle-size measurement is not easily quantifiable, except for spheres, cubes, and other regular shapes. The problem is: to define particle size and shape,

to measure size and shape in a consistent quantifiable manner,

to relate particle size and shape to properties of the bulk powder by means of quantitative laws and equations rather than on the basis of qualitative experience with particular powders.

The vagueness of the words used to describe the shape of particles is a major obstacle to the quantitative approach to particle-size analysis; "rounded", "angular", "fibrous", "elongated", and other purely qualitative terms are of little use in quantitative work.

"Measuring" particle shape has been largely confined to microscopic techniques. However, this is not really suitable where the particles are completely irregular, as in powder coatings, and to describe particle size accurately in terms of a single unit is in practice impossible.

Sieving has been used as a versatile method of separating broad fractions quickly, and for powder coatings appears adequate at present for providing the powder in "cuts" appropriate for the application method.

Conductivity methods (for example, the Coulter counter) measure the size of small particles on passage through a small orifice. The change in resistance of an electrolyte in the orifice is used to estimate the volume of the particle. However, this instrument has been found to be rather difficult to use in determining the particle size of titanium dioxide because the concentration of electrolyte necessary to show a change in resistance brings about flocculation of the pigment. The sedimentation technique was therefore used to determine particle size distribution for the work reported here.

The Shimadzu* Sedimentograph Type SA-2 was the instrument employed, and it is illustrated schematically in Fig. 11. A 1 per cent aqueous slurry of powder containing 0.1 per cent "Teepol"† was prepared in a small glass ball

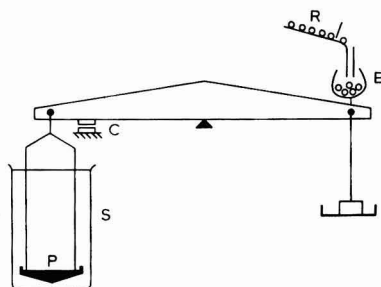


Fig. 11. Shimadzu sedimentograph

* Shimadzu Seisakusho Ltd., Tokyo, Japan. † Shell Chemicals (UK) Ltd.

mill without a grinding charge, which was trundled on rollers for 30 minutes to give an intimate mix. The powder suspension was run into a glass beaker, S, and the pan, P, of the Shimadzu balance was then immersed to the specified level in it. As the powder from the suspension settles into the pan, small identical balls are released from the reservoir, R, into the bucket, B, at the opposite end of the beam. A single ball is released from the reservoir automatically whenever the contact points, C, are closed, i.e. whenever the balance is sufficiently depressed by the settling pigment. Meanwhile, a pen records a trace on a chart attached to a drum rotating at constant speed about a vertical axis. Each time a ball is released, the pen moves vertically downwards through a height of exactly 3mm. An example of the stepped trace produced on the chart is shown in Fig. 12, and from it the particle-size distribution of the pigment is calculated.

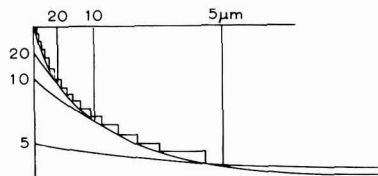


Fig. 12. Sedimentation chart

The particle size distribution of all the powders used in this work was determined, and since they all fell into the same grouping only one is reproduced as an example—Fig. 13.

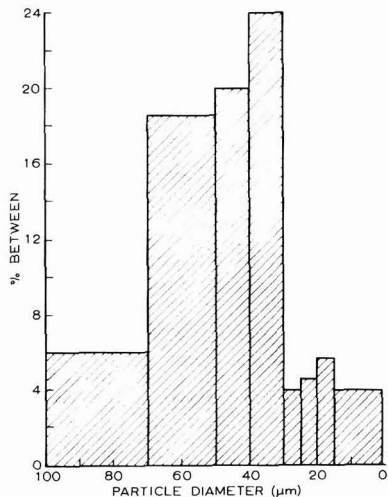


Fig. 13. Particle size distribution

Whilst the particle size distribution was being determined, another sample of each powder was sprayed electrostatically at a charge of 90kV on to solvent-degreased phosphated 20-gauge steel panels under ambient conditions (temperature 21°C, RH 55 per cent), to give a range of film thickness from 40μm to 100μm after baking the panels for 15 minutes at 180°C.

Each panel was examined visually for surface roughness in a viewing box (Fig. 14) made of wood with a window facing

the observer. Attached to the inside top of the box and running its length is a "Mazda" double-cap tubular tungsten lamp. The panels, inserted through the window, are placed on the bottom of the box and assessed visually by examining the surface at an angle of 45°.

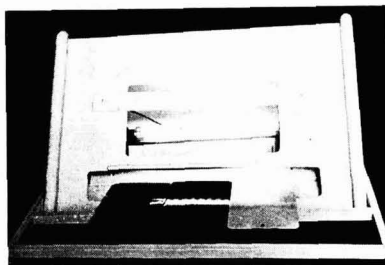


Fig. 14. Viewing box

Only the 100μm films exhibited traces of roughness. No roughness could be seen on thinner films, but all the panels showed marked cratering (cissing or crawling). Cratering in the context of powder coatings is defined as individual, fairly large circular or near-circular depressions in the baked film, generally penetrating to the substrate. They vary very widely in number and dimensions, but most of them have diameters between 1mm and 3mm.

From this series of experiments two conclusions were drawn.

1. The particle shape and size of an epoxy resin sieved to below 75μm do not depend on the processing method; the thicker the applied film the greater the roughness after baking.
2. In the absence of cross-linking, and provided that the temperature is above the flow-point of the polymer and sufficient time is allowed at this temperature, roughness can be eliminated entirely, the requisite temperature and time differing according to the polymer and film thickness.

Volatile content of raw materials

It is conceivable that residual volatile components of the resin, flow-control agent, hardener, etc. might be released during the baking cycle and contribute to roughness.

The volatile contents of the raw materials were determined by weighing a sample, heating for 15 minutes at 180°C, and reweighing after cooling in a desiccator, and were as follows.

| Material | Volatile content % |
|----------------|-----------------------|
| Epoxy resin | 1.6 |
| "Modaflow" | 4.5 |
| Hardener No. 2 | 1.0 |
| No. 3 | 1.5 |
| No. 4 | 6.1 |
| | (11.2% at 20 minutes) |

A series of powders was prepared as follows:

Epoxy resin,

Epoxy resin + 0.5 per cent Modaflow,*

Epoxy resin vacuum-dried at reduced pressure (8mm mercury) for six hours at 130°C, then cooled in a desiccator,

Epoxy resin + 0.5 per cent Modaflow* (Resin vacuum-dried as above).

All the samples were hammer-milled (immediately on removal from the desiccator for the vacuum-dried samples), sieved through 200 mesh, then applied as previously described (i.e. at a range of film thicknesses), and also a series of each at approximately 60 μ m dried film thickness. These last panels were baked for 5, 10, 15, and 20 minutes respectively at 180°C.

Examination of the panels in the viewing box showed no difference in roughness between those coated with the vacuum-dried powders and with the powders as received, so that only a minor part could be played by the volatile content of the system in contributing to roughness of the baked film.

However, marked differences in roughness were found in films baked for different lengths of time, as follows:

| Minutes at 180°C | Roughness |
|------------------|-------------------------------------|
| 5 | Extremely rough |
| 10 | Fairly rough |
| 15 | Slight traces of roughness |
| 20 | - Smooth: only waviness in evidence |

This indicated that it is the evacuation rate of the air entrained within the film that controls roughness, and this, of course, is correlated with the rate of cure of the system; the more rapid the cure the greater the degree of film roughness.

Effect of particle-size distribution of powder

The effect of particle-size distribution of the powder was determined by hammer-milling the epoxy resin and then sieving to the fractions below.

| Mesh No. | Size range μ m |
|----------|--------------------|
| 150 | 106 to 75 |
| 200 | 75 to 63 |
| 240 | 63 to 53 |
| 300 | < 53 |

The powders were sprayed, baked, and assessed as before. Little difference was noted in surface roughness between the panels, indicating that within the range of the test, particle size of powder is not a major influence on roughness in powder coatings.

Effect of preheating the substrate

If roughness is due to air entrained in the voids between the powder coating particles diffusing slowly through the film on application of heat, then with a substrate that is initially

hot the degree of roughness should be less than with a cold substrate. The milled and sieved epoxy resin was therefore electrostatically sprayed on to a series of cold steel panels, and another series was sprayed on panels preheated for 15 minutes at 180°C (spraying in the hot oven). The panels in each series were then stoved for 5, 10, and 15 minutes at 180°C and examined for roughness as before. The following results were obtained.

| Baking time minutes | Cold substrate | Hot substrate |
|---------------------|----------------|---------------------------|
| 5 | Very rough | Only a trace of roughness |
| 10 | Some roughness | Smooth |
| 15 | Smooth | Smooth |

Effect of titanium dioxide

In this study the epoxy resin was pigmented with Type IV pigment, "Modaflow," and hardener no. 2 (the least volatile). In one test, all the ingredients (except Modaflow) were vacuum-dried before mixing on the extruder, and in the other all ingredients were as received. The same technique as before was used.

After sieving to <75 μ m, the powder was electrostatically sprayed on to solvent-degreased phosphated 20-gauge steel panels to give film thicknesses ranging from 45 μ m to 120 μ m after stoving for 15 minutes at 180°C. Gloss and Talysurf measurements were made on these films as already described, and again the results indicate that the greater the film thickness the rougher the film and the lower the gloss.

It was concluded from these results, therefore, that the pigment had no significant effect on the degree of roughness of the film.

Roughness

It has already been stated that the surface of a powder coating can be described in terms of the primary and secondary textures, where the secondary texture is the basic irregularity, i.e. the "orange peel," and the primary texture the small irregularities superimposed on the secondary texture.

In an attempt to define the primary texture in greater detail, pigmented epoxy and acrylic systems were sprayed on panels at different film thicknesses and baked at the appropriate temperatures.

The films were sliced by means of a scalpel and the cuts examined by a scanning electron microscope (SEM). Fig. 15 shows the cross-section at different magnifications of the epoxy film at approximately 50, 75, and 100 μ m. The thinnest film appears to be continuous, whereas the two thicker ones have numerous holes within the film, most of which appear to be in the lower one-third of the 75 μ m film and in the lower two-thirds of the 100 μ m film.

The same effect was seen with the acrylic powder coating. For comparison, therefore, Fig. 16 shows cross-sections of the acrylic and epoxy films at 50 μ m and 100 μ m film thickness as viewed by the SEM at various magnifications.

* Modaflow incorporated by fusion-blending in a Z-blade mixer.

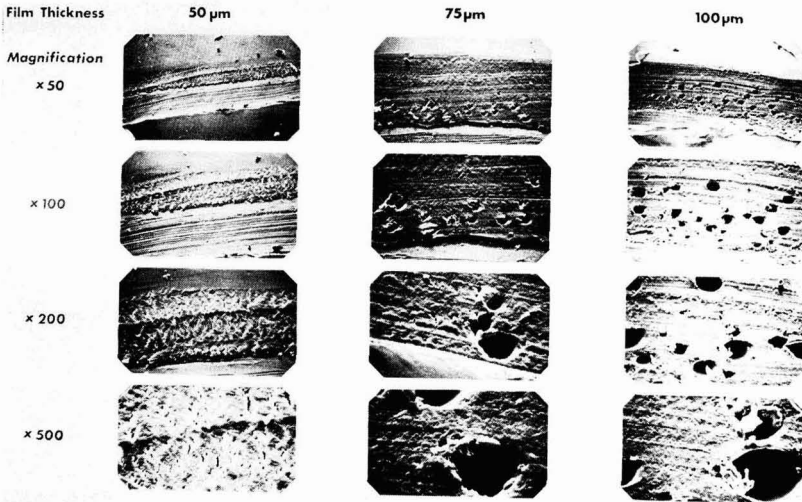


Fig. 15. Stereoscan cross-section of an epoxy powder coating at various film thicknesses

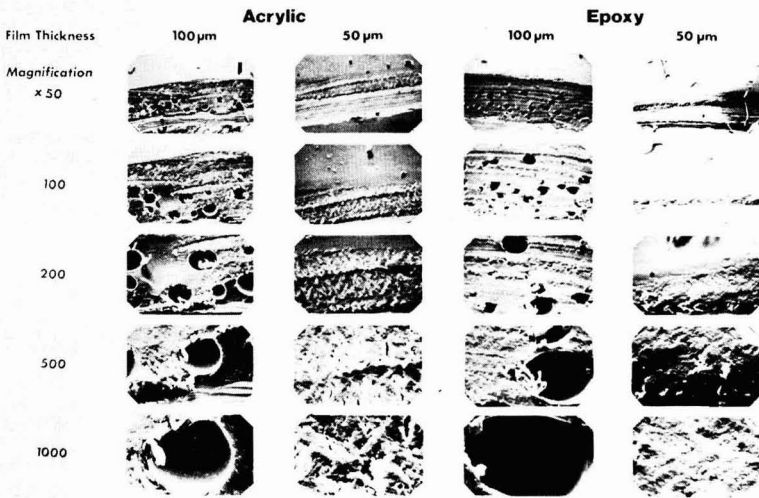


Fig. 16. Stereoscan cross-section of powder coatings

It may be concluded, therefore, that the suggested mechanism of curing of a powder coating does in fact occur, and that the two principal controlling factors in determining the surface texture are film thickness and rate of polymerisation of the coating.

Conclusions

The opacity given by a white powder coating at a specified film thickness will depend on the particular grade of titanium dioxide and on the polymer used. Coatings made with

acrylic resins have superior opacity to epoxy systems, probably because not so much aggregation of the pigment particles occurs in the acrylic type of polymer. Contrary to previous experience with solvent-based industrial finishes, the heavily coated grades of titanium dioxide give as high, if not higher, opacity than those with thinner coatings.

The tone of the white powder coating depends on the polymer and the crystal size of the rutile titanium dioxide used. The bluest tone is invariably given by rutile titanium dioxides of small crystal size.

The gloss of a powder coating is influenced by the degree of primary and secondary texture. The secondary texture is controlled by the flow of the polymer and additives used. The primary texture is influenced by the rate at which air entrained within the film during application escapes during fusion. The faster the rate of curing the greater will be the degree of primary texture and the lower the gloss. The lower the film thickness the less the primary texture and the higher the gloss.

A significant difference from conventional solvent-based industrial systems is that cured films of a powder coating do not exhibit a clear layer of resin at the coating/air interface.

Acknowledgments

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Some physical properties of pigments

I. Compaction under pressure

By M. J. Smith

ICI Plastics Division, Welwyn Garden City, Hertfordshire

Summary

The mixing operation by which plastics are coloured often leads to the production of agglomerates, which are unacceptable for a number of reasons. Mixing involves two distinct stages, powder mixing where many of the agglomerates are first produced, and paste mixing (or compounding) where they are supposedly broken down but where in practice they can be compacted and made stronger.

The prevention of agglomeration is dependent, in part, upon an understanding of the behaviour of pigment powders and their physical properties. The most important of these are the manner in which they compact and the strength of the resulting agglomerate. These can be determined by compressing powders in an instru-

mented tableting machine, a technique which is often used in pharmaceutical and powder metallurgical fields, but which has not previously been applied to the study of pigment powders.

Agglomerates have been found to vary greatly in their elasticity, the inorganic pigments being less elastic than the organic ones, and the phthalocyanines being the most elastic of all. It might be expected that elasticity would determine the ease with which the agglomerates may be dispersed during subsequent mixing operations.

Further parts of this work will demonstrate its relevance to powder coatings.

Keywords

Processes and methods primarily associated with: manufacturing and syntheses

agglomeration

Analysis, measurement and testing

compression testing

Quelques caractéristiques physiques de pigments.

Première Partie: La compacité sous pression

Résumé

Le procédé de mélange utilisé pour la coloration de matières plastiques provoque souvent le développement des agglomérés, qui, en vue de nombreuses raisons, sont insupportables. Ce procédé comprend deux étapes distinctes, le mélange des poudres où beaucoup des agglomérés se produisent, et le mélange en pâte où ils sont censément désintégrés, mais où en pratique ils peuvent devenir plus fortement tassés.

La prévention d'agglomération se dépend en partie d'une compréhension du comportement et des caractéristiques de pigments en poudre. La plus importantes de celles-ci c'est le mode de leur tassement et la force cohésive des agglomérés qui en résultent. On peut déterminer ces forces en tassant les poudres dans un

appareil de précision pour la fabrication de comprimés. On utilise cette technique très souvent dans les domaines des produits pharmaceutiques et des métaux en poudre, mais que l'on n'a pas déjà utilisé pour étudier les pigments en poudre.

On a trouvé que les agglomérés démontrent une variation importante à l'égard de leur élasticité, les pigments inorganiques ayant une élasticité plus faible que celle des pigments organiques, les phthalocyanines étant le plus élastique de tous. Il est vraisemblable que le degré d'élasticité est le facteur déterminant à l'égard de la facilité que démontrent les agglomérés à être redispersés au cours des procédés de mélange subséquents.

Einige physikalischen Eigenschaften von Pigmenten

I. Zusammenballung unter Druck

Zusammenfassung

Oft führt das für die Anfärbung plastischer Massen benutzte Mischverfahren zur Erzeugung von Agglomeraten, die aus verschiedenen Gründen unverwendbar sind. Mischung erfolgt in zwei ganz verschiedenen Zuständen; Pulvermischung, bei welcher erstmals viele der Agglomerate gebildet werden und Pastenmischung (kompoundieren), bei welcher diese annehmbarer Weise wieder verkleinert werden; in Praxis können sie aber kompaktiert und verfestigt werden.

Die Verhütung des Agglomerierens hängt teilweise davon ab, dass das Verhalten von Pigmentpulvern und deren physikalischen Eigenschaften verstanden wird. Davon ist die Art und Weise in welcher die Zusammenballung vor sich geht, sowie die Festigkeit des resultierenden Agglomerates am wichtigsten. Dieses Verhalten

kann durch Zusammenpressen von Pulvern in einer instrumentierten Tablettiermaschine bestimmt werden, eine oft auf pharmazeutischen und Pulvermetallurgie-Gebieten angewandte Technik, die jedoch bisher nicht auf das Studium von Pigmentpulvern erstreckt wurde.

Es wurde festgestellt, dass Agglomerate sich hinsichtlich Elastizität ganz verschieden verhalten, anorganische Pigmente sind weniger elastisch als organische, und die Phthalocyanine sind die allerelastischsten.

Es steht zu erwarten, dass Elastizität für den Grad der Leichtigkeit, mit welcher die Agglomerate beim sich ausschliessenden Mahl-vorgang dispergiert werden können, bestimmend ist.

Некоторые физические свойства пигментов. I. Уплотнение под давлением

Резюме

Процесс смешивания применяемый при окраске пластиков часто приводит к развитию агломератов, которые являются неприемлемыми по ряду причин. Смешивание состоит из двух стадий, порошкового смешивания в котором многие из агломератов впервые формируются, и пастового смешивания (или перемешивания), в котором эти агломераты якобы

разрушаются, но которые на практике могут уплотняться и усиливаться.

Предотвращение агломерации зависит отчасти от правильной интерпретации поведения пигментных порошков и их физических свойств. Самыми главными из этих являются процесс

их уплотнения и прочность образующихся агломератов. Последние могут быть определены сжиманием порошков в инструментальной таблетной машине, прием который часто применяется в области фармацевтики и металлургии, но который не применялся ранее в изучении пигментных порошков.

Introduction

In their natural state, plastics materials are mostly white, off-white or yellowish in colour. Since they are usually required by the consumer in a coloured form they must at some stage be coloured, usually by the incorporation of dyes or pigments^{1, 2, 3}. The reasons for this are varied but include protection from the action of light, identification by means of a code based on colour, the demands of the particular end application e.g. signs or signals, the need to make the article opaque; most frequently, however, plastics are coloured in order to improve the aesthetic appeal of the article being made.

This colouring process requires both skill and knowledge if it is to be carried out to high standards of quality, and whilst in general the incorporation of dyes presents few technical problems, pigments are more difficult to use satisfactorily. Pigments are usually supplied to the colourist as finely divided powders for mixing into the polymer by blending followed by hot compounding. Their propensity to form small hard particles (agglomerates) under compression, which cannot then readily be dispersed during the normal manufacturing cycle, creates problems of poor economics, technical weakness and unattractive appearance of the finished product.

The economic losses arise from the following factors.

The expenditure of excessive energy and loss of production time when production cycles are lengthened in attempts to disperse agglomerates, especially if reworking becomes necessary. Once agglomerates have been formed, they frequently pass through the mixing process unaltered, despite its being designed to break them down. This is not because they are too robust to be broken down by the available dispersing force, but only because the necessary level of dispersing forces has not been concentrated upon them. This is shown by their pernicious rupture to produce streaks during subsequent fabrication, when they do receive a shearing force which is sufficient to break them down, but when, of course, it is too late to introduce further mixing.

Pigment waste. In relation to their number, agglomerates account for a disproportionate quantity of the total pigment present and do not in any way contribute to the production of the desired colour. To maintain the colour, therefore, additional pigment must be added to compensate for this loss. The presence of agglomerates is thus a waste of an expensive raw material.

The technical weaknesses which arise from the presence of poorly dispersed pigment include: electrical faults; the loss of mechanical strength in mouldings, and breakages during the production of fibres and thin films; anomalous weathering performance; and difficulty in matching or maintaining a consistent colour in production.

The last of these difficulties arises when a mixture of pigments is employed. The partial loss of one pigment because of agglomeration upsets the balance to produce either an "off shade" batch, or one which varies in colour with changes in processing time as the partially agglomerated

Найдено что агломераты весьма различны в их упругости, неорганические пигменты менее эластичны чем органические а наиболее упругими являются фталоцианины. Можно было бы ожидать что упругость может определить легкость с которой агломераты могут рассеиваться в последующих операциях смешивания.

pigment gradually becomes dispersed and begins to assert its correct colour strength.

Lastly, agglomerates cause the finished article to be criticised or rejected, either because their presence is obvious or because they produce streaks of colour after becoming dispersed in the final processing stage, e.g., during moulding.

There is abundant evidence to show that agglomerates are produced in circumstances largely overlooked or unsuspected; e.g., in packages during storage and more seriously, during the early stages of those very processes which are supposed to be breaking them down. In compounding pigments with polymers it is necessary to distinguish between two separate parameters, both of which are necessary to describe the quality of mixing. "Dispersion" which is the process producing intimate contact between each unit particle of pigment and the continuous phase, and "distribution," the process which results in a homogeneous mixture.

The majority of plastics compounding equipment will distribute effectively, but will produce agglomerates in the early stages of mixing which they are subsequently unable to disperse. It is largely true to say that an agglomerate, once formed, is unlikely ever to meet conditions that will break it down. This is particularly true of batch mixers such as the Banbury or two-roll mill, in which a relatively large proportion of the total mixing volume contains only relatively low levels of shear. As a result these zones do not contribute to the development of good dispersion. An agglomerate, although weak enough to be dispersed in that part of the mixer which contains high shear forces, will not be broken down whilst it remains in the low shear zone. As the transfer from one zone to another can be slow, many agglomerates formed at the beginning of a processing cycle will not have been subjected to a high shearing force, since mixing time may be limited for technical or economic reasons. Even long dwell times in the region of low shear are not always sufficient to disperse the agglomerates. They will therefore be present in the final product. Clearly, the quality of dispersion can be most easily improved by preventing the original formation of agglomerates!

There are, therefore, two major areas of interest: the mixing of components in powder form, where agglomeration tends to occur, and the mixing of these components in paste form, where it is hoped that the agglomerates will be dispersed. Unfortunately the early stages of paste mixing (i.e., before the polymer has melted fully) frequently compact the agglomerates further. From this position two definite lines of enquiry become obvious:

An examination of the process of agglomeration, its mechanism and the reason for its occurrence.

The evaluation of strength and mechanical properties of the agglomerate so as to judge the ease with which it can later be dispersed. Here, the use of large agglomerates (especially made!) is necessary for ease of handling.

The second investigation is of particular importance, since it is more than probable that the dispersibility of pigment agglomerates will be dependent upon their strength; aggro-

merate strength (measured as tensile or compression strength) will determine the level of shear necessary in a mixer. Knowledge of this relationship should also enable us to forecast changes in dispersion, and hence changes in colour which might result where a mixture is subjected to a change in mixing intensity. The relationship between agglomerate strength and dispersibility will, in fact, be established in a later paper.

In the past, efforts aimed at examining pigment dispersion have all centred upon a study of populations of pigment particles. The quality of dispersion was determined from the particle size distribution, and modifications to the basic mixing process were monitored in terms of their effect upon the previously determined size distribution. Unfortunately, the method was unable to differentiate between the relative importance of agglomeration and dispersion.

A suitable method of examining the mechanism by which agglomerates are formed involves the direct compression of the pigment powder. Various attempts have been made in the past. Walker⁴ studied the compression of powders in a cylinder and piston assembly under both impactive and static loadings, and recorded changes in bulk volume against resistance to compression. He observed differences between materials, which he attributed to packing by deformation and plastic flow as opposed to fracture followed by repacking. This technique has been used more recently to examine the structure of carbon black pigments⁵ by relating specific volume and the way in which it varies with compaction, to the oil absorption of the pigment powder.

Birks has studied the compaction of various powders under low stress (up to about 100psi). He found⁶ the effects of wall friction (between powder and die) upon compaction to be much greater than had previously been assumed. The implications of this are considerable, as it follows that comparisons are only valid when they are made between tablets which have been produced in the same sized die.

Unfortunately, because of the static nature of these tests, none of them are immediately applicable to the study of powder flow under conditions which are equivalent to those which exist in mixers. However, a suitable method has been developed by the pharmaceutical industry using a fully instrumented tableting machine⁷ which is normally capable of operating at relatively high speeds and where loadings are of relatively short duration (0.10 second).

Experimental

Apparatus

The device consists of a cylindrical die and two closely fitting punches which enter into the die from each end and serve both to contain and compress the powder. The powder is compressed by the downward movement of the top punch, and the level of pressure developed is dependent upon the amount of material and the size of the space into which it is being compressed. Pressure is transferred by the powder from the top punch on to both the bottom punch and the die wall. The way in which the radial pressure develops on the die wall is controlled by the compressibility and elasticity of the powder, and the way in which it flows under pressure. The development of both axial and radial pressure may be monitored by means of strain gauges fixed to the top punch and the die wall. Information relating to the properties of the agglomerate produced may be obtained from the decompression cycle.

For this work a Manesty F3 Single Punch tableting machine was used, fitted with $\frac{3}{8}$ inch diameter, plain-ended punches and the appropriate die (Fig. 1). The top punch and die wall were both monitored for stress changes by fitting foil strain gauges (Fig. 2), and their output was amplified and recorded.

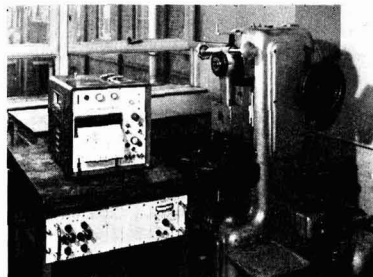


Fig. 1 Manesty F3 tableting machine complete with instrumentation

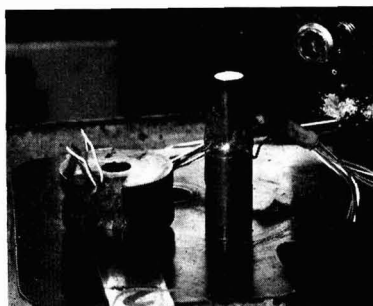


Fig. 2. The top punch and cut away die assembly, showing strain gauges

Two gauges were located on diametrically opposite sides of the punch shank. They were connected into opposite arms of a Wheatstone bridge network, which was then completed using two dummy gauges attached to a separate steel bar. These dummy gauges compensated for temperature variations, whilst the opposed gauges allowed for the possibility that bending might occur in the punch during compression, and also provided increased sensitivity. While using two gauges helps considerably, there is still the risk that the plane of bending may not be fully coincident with that of the two gauges.

High sensitivity was less easily achieved for the die assembly, since the die wall had to be thick enough to withstand tableting pressure, yet thin enough to reduce the dissipation of the strain set up in the metal. As shown in Fig. 2, this was achieved by cutting away some of the outer wall of the die. The active gauges were stuck on to the exposed wall with their active axes perpendicular to the direction of compression. To increase sensitivity further, these surfaces were curved in a circle concentric to the original die wall. Additional dummy gauges were mounted on the adjacent radial surfaces and connected into a Wheatstone bridge together with the active gauges.

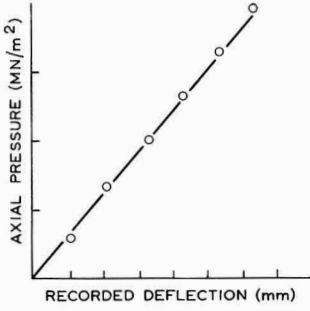


Fig. 3. Calibration of the upper punch strain gauge

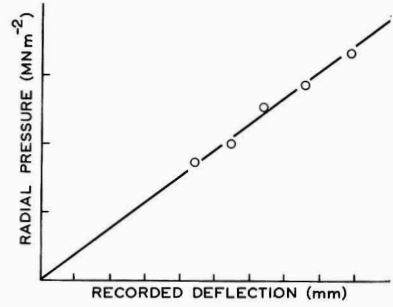


Fig. 4. Calibration of the die wall strain gauge

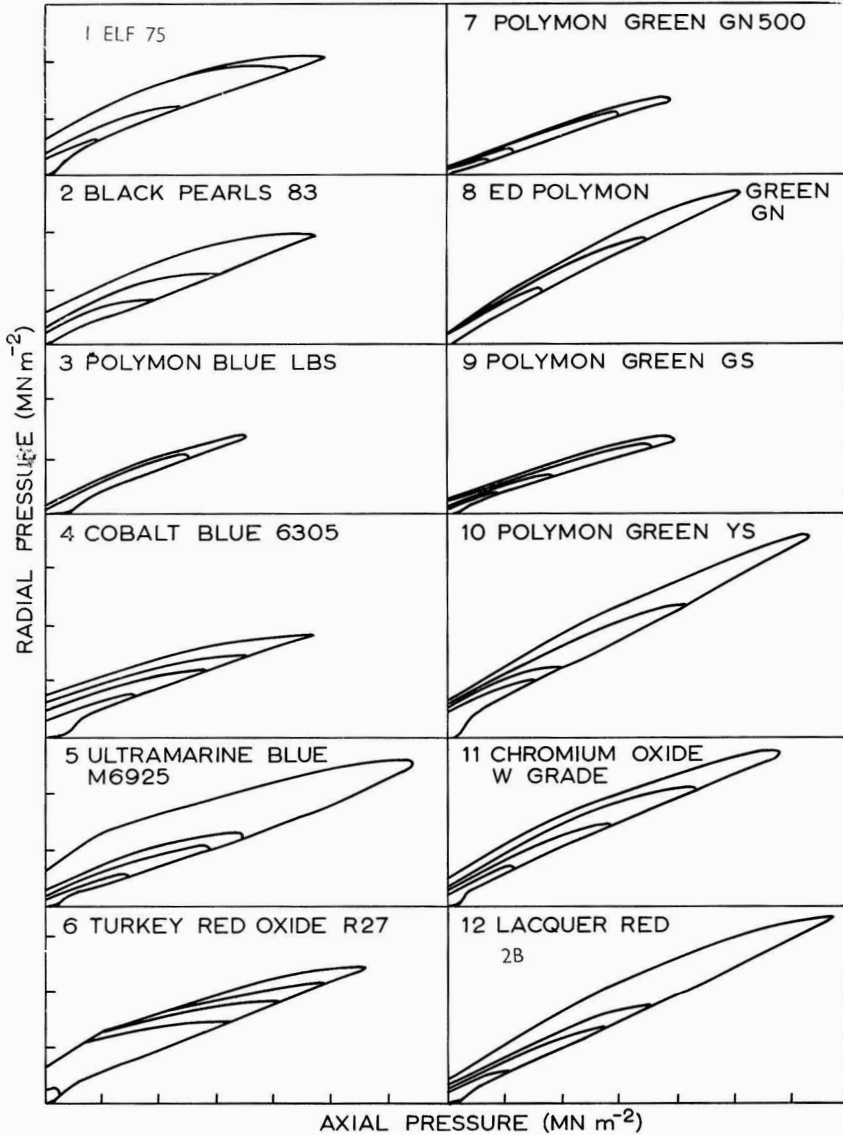


Fig. 5. Development of stress during powder compression (axial vs radial pressure)

Method

Calibration

This was conducted in two stages. First, a known load was applied to the top punch via its planar surfaces. This load was increased in steps of half a ton up to a maximum of three tons. The output from the strain gauges was recorded and the values used to construct an axial pressure calibration plot. Fig. 3 shows that the strain gauge response was linear.

Secondly, the response of the die wall and its strain gauges to known pressures was determined by compressing a rubber plug^{8, 9} in the die. Under compression, rubber has been shown to behave as a liquid¹⁰, and the axial and radial pressures may therefore be assumed to be equal. As shown in Fig. 4, the response of the strain gauges was again linear.

Varying quantities of pigment were transferred to the tableting die by hand, and compacted. The strain gauge responses were recorded producing two traces, similar in form to the standard Gaussian distribution curve. The radial pressure trace was slightly delayed in comparison to the axial pressure curve because of the frictional properties of the powder under examination. Friction resulted in "bridging" with a subsequent delay in the transference of pressure from the punch on to the die wall. At the end of the decompression cycle (lasting about 0.05 seconds), a small residual radial pressure remained. This resulted from the presence of the agglomerate or tablet, and was a measure of the apparent strength of the tablet.

The strain gauge traces were converted into pressure using the earlier calibrations. Radial pressure was plotted against the axial pressure which produced it, and the resulting compression/decompression cycles are shown in Fig. 5 and 6. (For Figs. 3, 4 and 5 see page 129)

Errors

There are several potential sources of error associated with this technique. Errors which arise owing to changes in ambient temperature and the punch bending under pressure have already been mentioned, as has the loss of sensitivity due to the finite thickness of the die wall, it being impossible to position the gauge at the point of maximum stress, i.e. on the internal surface of the die in contact with the edge of the tablet. Errors may also arise from the way in which stress is propagated outwards from the pigment tablet through the metal die. It has been found that the radial pressure, as measured on the outer die wall, is dependent upon the vertical position of the strain gauge in relation to the point at which the tablet is produced. To avoid errors from this cause, care was taken to ensure that the tablets were all produced at the same level within the die.

It is well known that fine powders flow badly and show high levels of interparticle friction. Thus the coarseness or fineness of grind of the sample under test can influence the way it compacts and variations of as much as 5-10 per cent can be encountered. Interparticle friction also influences the way in which particles rearrange themselves during compaction

Friction between the powder and the die wall results in a shearing action which aids particle packing during compression, but this same friction leads to the undesirable development of cracks in the tablet¹¹ which result in its disintegration (or capping) during ejection from the die.

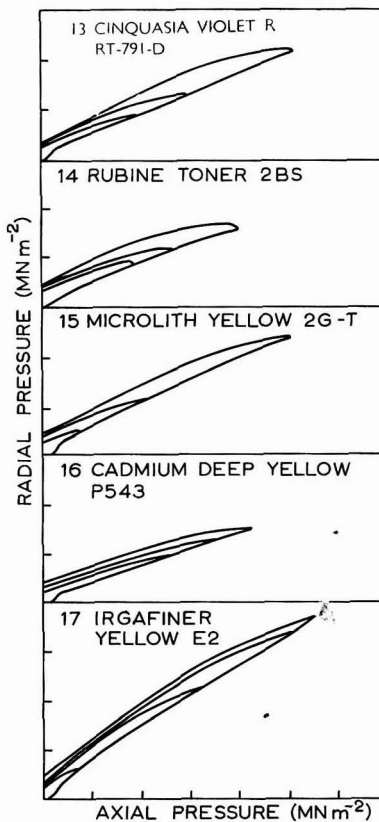


Fig. 6. Development of stress during powder compression (axial vs radial pressure)

Whilst no attempt was made to modify interparticle friction by the addition of glidants or lubricants, the friction between the pigment and the die wall was reduced as much as possible by coating the inside surface of the die with Carnauba wax before each compression cycle.

Materials

A total of 17 pigments was selected so as to cover a wide range of chemical and physical types. During this examination the local relative humidity remained between 55 and 65 per cent.

1. Elf 75—a medium flow channel black from Cabot Carbon Ltd.
2. Black Pearls 83—a densified granulated channel black from Cabot Carbon Ltd.
3. Polymon Blue LBS—a full shade α -form copper phthalocyanine from ICI Ltd., Organics Division.
4. Cobalt Blue 6305—from Blythe Colours Ltd.
5. Ultramarine Blue M6925—from Reckitts Colours Ltd.

6. Turkey Red Oxide R27—from Golden Valley Colours Ltd.
7. Polymon Green GN500—a full shade polychloro copper phthalocyanine from ICI Ltd., Organics Division.
8. ED Polymon Green GN—a predispersed version of 7, from ICI Ltd., Organics Division.
9. Polymon Green GS—a reduced form of Polymon Green GN500 from ICI Ltd., Organics Division.
10. Polymon Green YS—from ICI Ltd., Organics Division.
11. Chromium Oxide: W Grade—from Associated Chemicals Ltd.
12. Lacquer Red 2B—an anthraquinone dyestuff from ICI Ltd., Organics Division.
13. Cinquasia Violet R RT-791-D—a quinacridone from Du Pont.
14. Rubine Toner 2BS—from ICI Ltd., Organics Division.
15. Microlith Yellow 2G-T—a predispersed pigment from CIBA-GEIGY (UK) Ltd.
16. Cadmium Deep Yellow P543—from Blythe Colours Ltd.
17. Irgafiner Yellow E2—a predispersed pigment from CIBA-GEIGY (UK) Ltd.

Results

The results are shown in Figs. 5 and 6, in the form of radial vs axial pressure curves. The general form of the curves is summarised below.

The several superimposed curves for each pigment are the results of the powder being subjected to different maximum levels of compression in the various tests made.

The area of the curve gives a measure of the elasticity of the agglomerate. The narrower the curve (i.e. the lower the level of hysteresis) the greater is the elasticity shown by the pigment tablet.

The intercept on the radial pressure axis shows the residual pressure supported by the pigment tablet in the die wall. For the less elastic pigments, this gives a measure of the strength of the tablet.

The behaviour of the powder during the early part of the compression cycle (the lower line starting at the origin) shows the flowability of the compacted powder under pressure. The poor flow characteristics of fine powders, with their ability to "bridge," is shown by the low radial pressure rise during the early stages of compression.

Discussion

Considerable interest has already been devoted to powder compression in a number of industrial applications, including pharmaceuticals^{7, 16}, fuel technology¹⁸, mining¹⁷, ceramics¹⁹ and general chemicals^{4, 8, 11, 19, 20}. Attention has also centred upon theoretical aspects of the same problems, particularly the development of powder compression equations. The

progress in this field has recently been summarised by Kawita and Lüdde²¹.

The compaction behaviour of a powder system is determined by a wide range of independent physical properties, such as particle size and shape, surface characteristics, and the presence of adsorbed species such as water. This is true even for very fine powders and is the reason why different types of powder behave so differently. The complexity of the systems involved explains why so far most effort has been directed towards the solution of particular problems. As a result, little basic work applicable to powders in general has been attempted on anything other than the simplest of systems and, apart from the inclusion of an occasional sample in a series of experiments^{5, 6, 17, 20}, little attention has been paid to the problem of agglomeration in pigment powders.

When powders are subjected to compression, the applied forces are transmitted through the mass via point contacts between particles. Consolidation takes place during these early stages, and the extent to which this occurs is greatly influenced by interparticle friction^{24, 25, 26}. At higher pressures, when the possibility of further rearrangement has become limited owing to the shortage of available space, the further development of stress concentrations will result in the deformation of particles. This can arise from elastic, plastic, or destructive mechanisms, the exact nature and balance of which depends upon the rate of application, the magnitude of the induced stress, and its duration. If the application of the load is particularly rapid and its magnitude suitably high, the powder will be incapable of plastic or elastic deformation and its particles will fracture (destructive deformation).

In his work on the compaction of sulphur powder, Birks⁶ has observed that it consolidated in a series of sudden collapses, the compacted powder deforming elastically right up to the instant of collapse. This was explained as being due to a very high strain rate during the collapse, which, coupled with friction, causes the interparticle contacts to melt. Friction is thereby momentarily reduced and considerable compaction takes place before the contacts solidify again prior to the next elastic deformation and collapse. For fine particles at low rates of compaction, Birks favours plastic deformation as the most likely mechanism. It is highly probable that the compaction of materials as complex and as varied as pigment powders will involve most of these different mechanisms.

Figs. 5 and 6 show that the pigment pellets produced in this work have been deformed beyond their elastic limits, although it is difficult to determine at what point that occurred. An examination of the foot of the compaction curves shows that most powders flow badly, indicating that they are cohesive under low pressure conditions. The one exception to this generalisation is provided by Black Pearls 83, a densified carbon black which flows easily since it is produced in beaded form (up to 1.2mm diameter). The improved flow properties of this pigment are shown by the immediate development of a radial pressure as soon as the axial pressure is applied, and may be attributed partly to the free flowing characteristics of these relatively large particles and partly to their having already been compressed.

As has already been stated, the area of the hysteresis loop in the pressure cycle is a measure of the elasticity of the agglomerate; the more elastic it is, the smaller is the area of the loop. A wide variation in elasticity has been found in the pigments tested, but in general the organic pigments are more elastic than the inorganic ones, and the most elastic pellets

of all are those produced from the phthalocyanines. Since resistance to fracture is directly related to elasticity, phthalocyanine pigments would be expected to produce agglomerates that are very difficult to disperse. This is confirmed by the author's experience in pigmentsing a wide variety of plastics where the organic pigments, as a class, were found to be more difficult to disperse well than the inorganics, and phthalocyanines more difficult than most.

As has been shown, the compression of powders in a die exerts pressures on the die wall such that when the punches are removed the tablets do not fall out, but have to be forcibly removed. As they leave the die these pellets expand, showing that while remaining in the die they support a considerable radial pressure. How does this radial pressure arise? Goetzl²² considered the residual radial pressure to arise from the elastic contraction of the die after the axial pressure had been released. However, the tablet is a solid body and must also have elastic properties by means of which it can deform and at least partly release this pressure. A suggestion was put forward by Long relating to the compaction of metal powders in a die²³ which he later developed in greater detail¹¹ by considering the material while it was under compression. Once a pellet has been produced which is sufficiently firm to offer resistance to the punch, both axial and radial pressures can be recorded. From this moment onwards the tablet behaves as a solid body, and may exhibit several different types of behaviour^{9, 11}. Three distinct types of behaviour can be established.

The body is perfectly elastic and deforms, but can show recovery later because the elastic limit has not been exceeded.

The yield point has been passed and the body deforms, giving a constant yield stress in shear independent of the magnitude of the applied stress.

The yield point is again passed, but the body deforms so that the yield stress is a function of the applied stress.

This treatment assumes isotropy in the powders during compression and the absence of die wall friction. As suggested by Long¹¹, when the axial pressure has reached zero the tablet is no longer subjected to pressure from all sides. The effect of friction between the tablet and the die wall is to produce stress, and as a result strain builds up on that part of the tablet surface which meets the die at the extremities over which friction acts, i.e. at the corners of the tablet. The expansion of the tablet is resisted by the die wall but will be relatively free along its axis, and the stress concentrations will follow saucer-like contours within the tablet. Cracks of this shape, starting at the tablet corners are common under certain conditions, and the phenomenon is referred to as "capping". It was experienced with several pigments, such as carbon black, and some of the inorganics.

Train has suggested¹⁵ that, if a material resists flow, it will form arches and voids, and the uneven packing which results could cause capping. Its occurrence during these experiments suggests a high level of die wall friction despite attempts to reduce or remove it by lubricating the die wall. The greater propensity for the inorganic pigment pellets to "cap" almost certainly arises from the abrasive nature of the inorganic pigments which remove the lubricant during compression. The organic pigments are less abrasive and seem less likely to remove this coating (besides being more elastic). Indeed, phthalocyanine pigments have even been studied for their lubricating properties²⁷.

During tableting, some pigments were found to stick to the metal surfaces of the punch. Tablets made from the pre-dispersed/coated versions were also found to deform under a continuously applied stress. This is no doubt due to the relatively low melting point and softness of the surface coatings which allows the tablet to flow under pressure. Amongst the inorganic pigments, Cobalt Blue 6305 is unusual in the ease with which it sticks to the surface of the metal die. This phenomenon of sticking has a direct bearing upon the way in which these pigments behave in powder mixers such as the Henschel mixer. Once a pigment has started to stick to a metal surface (such as a blade) it will continue to build-up rapidly at that point. Such a build-up, besides constituting a considerable loss of pigment, can act as a secondary source of agglomerates should it break away into the mixture.

The way in which the powder flows during mixing²⁸ has considerable bearing upon its behaviour during mixing. Not only will it control the balance between the various mixing mechanisms and the way in which these are related to the mixer, but it will also decide the degree and type of segregation which occurs and the amount of build-up which results. Furthermore, flow during compaction must have considerable influence upon the type of packing which results during agglomeration.

Conclusions

Under compression, pigments vary considerably in the ease and readiness with which they flow during compaction and rearrangement of the particles; however, their flow under low levels of pressure is uniformly poor, as would be expected for powders of such small particle size. As is well known, increasing the size of the particles improves the flow characteristics of these systems, as shown by the results of the tests done on the beaded version of carbon black. It appears that flow is determined by particle characteristics of a physical nature such as size, size range and shape rather than its chemistry.

Tablets made by compressing organic pigments are much more elastic than those obtained from inorganic pigments; and of the organic ones, the most elastic of all are the phthalocyanines. This is generally in line with practical experience of the ease with which these classes of pigments are found to disperse into plastics, and this supports the expectation that elastic agglomerates will be difficult to disperse.

Some pigments were found, in the tableting process, to be more prone to stick to metal surfaces than others. These pigments will be more difficult to handle in powder mixers where their adhesion to blades and/or wall will result in loss of pigment, and will contribute to increased agglomeration levels should the adhering material break away into the mixture.

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The ANLAB colour system*

By A. C. Cooper and K. McLaren

ICI Ltd, Organics Division, Hexagon House, Blackley, Manchester M9 3DA

Summary

Extensive investigations carried out on dyed textiles have shown that colour difference equations based on the Adams Chromatic Value formula correlate better with the judgments of professional shade assessors to a degree which is statistically significant than any others. The uniform colour space on which this formula is

based, ANLAB, has been found to provide a colour order system possessing many advantages over other systems and it has been used to investigate the pigment technologist's variables of hue, strength and dirtiness.

Keywords

Properties, characteristics and conditions primarily associated with materials in general

colour
hue
saturation

Miscellaneous terms

ANLAB system

Le système de couleurs "ANLAB"

Résumé

On a démontré, au cours des investigations étendues effectuées sur des textiles teinturés, que les équations de différence en couleur basées sur la formule de la valeur chromatique d'Adams (Adams Chromatic Value formula) rendent une corrélation supérieure aux autres, de n'importe quelle provenance, avec les avis des appréciateurs de teintes professionnels, et à un degré ayant de la

significance statistique. On a trouvé que les intervalles de couleur uniformes, sur lesquelles se base cette formule "ANLAB," assurent un système de couleurs qui possède plusieurs avantages auprès d'autres systèmes, et l'on s'en servait pour étudier les variables que les spécialistes de la technologie de pigments appellent respectivement, la teinte, le pouvoir colorant et l'impureté.

Das ANLAB Farbsystem

Zusammenfassung

Anhand von ausgedehnten mit angefarbten Textilien ausgeführten Versuchen wurde gezeigt, dass Farbunterschiedsgleichungen auf Basis von Adams chromatischer Wertformel besser mit der Beurteilung fachmännischer Farbtonprüfer und zwar zu einem statistisch bemerkenswerten Grad übereinstimmen, als irgendwelche anderen.

Es wurde gefunden, dass ANLAB für das einheitliche Farbfeld, auf dem diese Formel beruht, ein gegenüber anderen mit vielen Vorteilen verbundenes System der Farbordnung besitzt; es wurde angewandt, um die Variablen der Pigmenttechnologien Farbstich, Farbkraft und Schmutzigkeit zu untersuchen.

Цветная система АНЛАБ (Аналитические Лаборатории)

Резюме

Пространственные испытания проведенные на окрашенных текстильных изделиях, показали что уравнения разницы цветов основанные на формуле хроматического показателя Адамса, согласуются лучше, статистически в существенной степени, со мнениями профессиональных экспертов, чем любые другие

уравнения. Найдено что единообразное цветовое пространство на котором эта формула (АНЛАБ) основана, дает порядковую цветную систему обладающую многими преимуществами над другими системами и эта система применялась для изучения пигментных вариантов оттенка, прочности и загрязнения.

Introduction

The XYZ tristimulus values of any given colour may be considered as defining its position in a three-dimensional space whose axes are X, Y and Z. Any colour which is not a perfect match to it must be located in a different position and the distance between the points can be easily calculated. If XYZ space were uniform, this distance would be a measure of the perceived difference between two colours but it is not uniform, the distances corresponding to visually equal differences varying by at least 30:1. Since 1935, numerous transformations of XYZ space have been made with the objective of devising a uniform colour space, and these transformations usually appear as colour difference equations that permit the calculation of the distance between two colours whose XYZ values are known. The reliability of these formulae is most readily assessed by seeing which best predicts either the pass/fail decisions of a number of pro-

fessional colourists working to the same nominal tolerance, or the order in which sample/standard pairs are placed when the sole criterion is the size of the perceived difference between them. Three investigations using paint panels have been carried out^{1, 2, 3}: Davidson¹ and Robinson² both found a MacAdam equation to be best, whereas Mudd and Woods³ found the Adams Chromatic Value equation to be the best of the established formulae. The latter results are of greater value because they were obtained at 30 different colour centres, compared with only six (Davidson) and one (Robinson).

Textile samples have been much more extensively studied than paint films, but as there is every reason to believe that the criteria on which pass/fail decisions are based will not be different, the conclusions from these studies should be equally applicable in the paint industry. The most important studies were made by Davidson and Friede and by Jaeckel

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and his co-workers and their results have recently been reviewed⁴. These showed that equations based on the Adams Chromatic Value formula correlate better with visual assessments than any other and to an extent which is statistically significant. As a consequence of this, the International Standards Organisation recommended (October 1971) that the original formula should be used for textiles with a scaling factor of 40 until such time as a demonstrably better formula was developed⁵. This formula has also been recommended for use in the UK for leather⁶ and plastics⁷. Its derivation has been fully described⁸ and this account showed that, as Nickerson had played a vital part in the development of the formula, it should be referred to as the Adams-Nickerson formula: as the axes of the uniform colour space on which it is based are *L*, *A* and *B* the space is conveniently termed ANLAB.

ANLAB colour space

Whilst ANLAB space is of particular interest because it is the most uniform when the criterion is the spacing of colours showing differences of commercial significance, it also has another advantage that is potentially just as important: its orientation is ideal for using it as a colour order system.

When an attempt is made to arrange a random selection of surface colours in a systematic order solely by their appearance the same array inevitably emerges. This array was first devised by the Swedish priest and astrologer Sigfrid Aron Forcius in 1611⁹ and independently by Munsell and Ostwald in this century: ANLAB space has essentially the same configuration as shown in the diagrams in Fig. 1.

These cylindrical co-ordinates are *LC* and *H* where *L* is the lightness expressed on an 0 (black) to 100 approx. (white) scale. *C* is the distance from the central axis and is thus a measure of saturation or chroma, being linearly related to the latter: the *C* scale varies from 0 (achromatic) to 120 approx. for the most saturated non-fluorescent yellow pigment. *H* is the hue angle defined as the angle the line through the locus of the colour and the origin makes with the *A*+ axis in the *AB* diagram: these angles are expressed on a 0-360° scale and pure red (i.e. neither yellowish nor bluish), pure yellow, green and blue hues occur at 27, 87, 163 and 254° respectively (mean of 24 colourists' choice)¹⁰. The fact that three of these angles are considerably displaced from the *AB* axes must not be assumed to be a weakness of ANLAB space, because it is known that if colours of the same saturation are equally spaced visually to form a complete hue circle, pure red and pure green do not lie diagonally opposite each other and neither do pure yellow and pure blue.

This fact is mainly responsible for some of the significant anomalies existing in Munsell hues. The principal blue for example, should be a pure blue, one which is neither greenish nor reddish; Munsell 5B hues, however, are decidedly greenish, and Munsell 10B hues are the purest blues.

The calculation of the ANLAB co-ordinates *LABC* and *H* from *XYZ* tristimulus values is very simple if tables are available for converting *XYZ* → *VXYZ*. These tables are given in two of the standard text books on colour measurement^{11, 12} whilst a more accurate set, which is also easier to use, is published by the Society of Dyers and Colourists⁸.

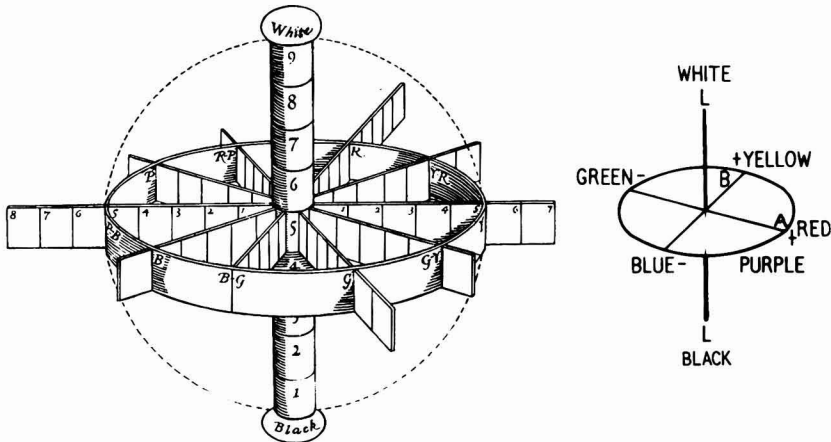


Fig. 1. Left: Munsell Space (reproduced from A Grammar of Colour. A Basic Treatment on the Color System of Albert H. Munsell, copyright 1969 by Reinhold Publishing Co.) Right: ANLAB colour space

However, although it is very much easier to deduce the appearance of a surface colour from its LAB values than from its xy Y values (which are very much better for this purpose than XYZ values), complications are introduced by the fact that three-quarters of all surface colours will have at least one negative value (*A* and or *B*). However, in 1966 one of the authors (ACC) showed that this defect could be completely overcome by converting the Cartesian co-ordinates *LAB* into cylindrical co-ordinates which are then just as easy to interpret as Munsell notations.

From *V_X*, *V_Y* and *V_Z*, the ANLAB co-ordinates are calculated from the following equations:

$$\begin{aligned}
 L &= 9.2 V_Y \\
 A &= 40 (V_X - V_Y) \\
 B &= 16 (V_Y - V_Z) \\
 C &= (A^2 + B^2)^{1/2} \\
 H &= \tan^{-1} (B/A)
 \end{aligned}$$

This is an extremely important advantage of ANLAB space over Munsell space: the calculation of the Munsell renotation of a given paint panel whose XYZ values are known is extremely difficult, even if a computer is available, and cannot be done precisely enough to satisfy the needs of industry; LAB values can, of course, be calculated as precisely as tristimulus values.

To illustrate the value of ANLAB as a colour order system, a comprehensive investigation has been carried out into the terms the dyer uses to describe the perceived difference between two colours that are similar¹⁰. Most of these terms are also used by pigment technologists, and these terms have been investigated separately; where there were no significant differences in terminology the term "colourist" will be used to cover workers in both industries.

The variable of hue

This is the simplest of the colourist's variables and his terminology differs from that of the layman merely by being much more restricted. Only one of four hue terms is normally needed to describe any perceived difference in hue, these terms being redder, yellower, greener and bluer. These are the terms which identify unique hues, i.e. hues which cannot be described in any other way, unlike the hue "orange" which can be adequately described as a combined sensation of redness and yellowness.

Differences in hue can be readily deduced from the ANLAB hue angle H . The first step is to recognise the hue of the standard so as to identify the permissible terms, e.g. a red can be yellower or bluer than another red but never redder or greener. The difference in hue angle then indicates the hue difference, e.g. in the case of reds, the sample with the larger angle will be yellower, that with the smaller angle, bluer because the $B+$ (yellow) axis is the first to be reached as one goes round the hue circle from the position of reds in the direction of increasing hue angle and the $B-$ (blue axis) is the first going the other way round; in either case one ignores the $A+$ (red) axis if this is reached first, as redder is not a permissible term.

The variable of strength

BS 1611¹³ defines the terms "stronger" and "weaker" as used in the paint industry by reference to differences "apparently due to the presence of more or less colour than in the original sample." Presumably a paint sample containing a 60:40 pigment:white ratio would be regarded as being weaker than one having a 65:35 ratio although BS 1611 defines this equally as being whiter—"a difference apparently due to the presence of more white than in the original sample."

The pigments listed in Table 1 were ground in a blend of a semi-drying oil-modified thixotropic alkyd resin with a long-oil modified alkyd resin containing pentaerythritol, using a stainer-grind technique with a "Red Devil" paint conditioner, and then mixed with a white paint based on TiO₂ RCR2 to give the required proportions.

The paints were then coated on white cards and, after drying, measured on a GE recording spectrophotometer excluding specular reflection. The XYZ values were then calculated for Illuminant C (16 weighted ordinates) and converted to the ANLAB co-ordinates LA and B .

Table 1

Coloured pigments used for strength line determinations

| Number on Figures | Colour Index number |
|-------------------|---------------------|
| 1 | Pigment Yellow 3 |
| 2 | Pigment Yellow 34 |
| 3 | Pigment Yellow 24 |
| 4 | Pigment Orange 1 |
| 5 | Pigment Orange 5 |
| 6 | Pigment Red 104 |
| 7 | Vat Orange 3 |
| 8 | Pigment Red 12 |
| 9 | Pigment Red 145 |
| 10 | Pigment Blue 15 |
| 11 | Pigment Violet 23 |
| 12 | Pigment Blue 27 |
| 13 | Pigment Green 15 |
| 14 | Pigment Green 41 |
| 15 | Pigment Green 13 |
| 16 | Pigment Green 13 |
| 17 | Pigment Yellow 34 |
| 18 | Pigment Brown 6 |

These co-ordinates define the strength lines in ANLAB space of each pigment and each line was a smooth curve, usually varying in three dimensions. The shapes of these curves cannot be readily interpreted from a perspective drawing and it is therefore necessary to plot two, two-dimensional graphs, the first being a plot of A against B , the second L against C .

The strength lines in the AB diagram are shown in Fig. 2, which is equivalent to viewing ANLAB space from directly above, differences in lightness (L) being ignored. Colours of constant Munsell hue plot as virtually straight lines in the AB diagram¹⁰ so Fig. 2 clearly shows that an increase in

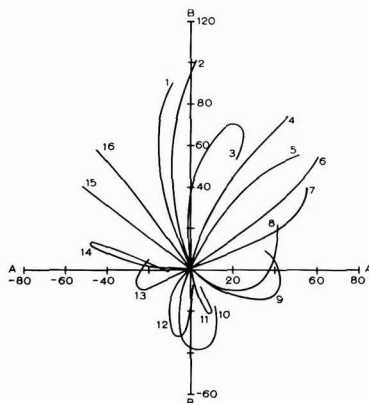


Fig. 2. Strength lines in AB diagram

strength is usually accompanied by a marked change in hue. These changes possess a certain symmetry: e.g. pigments yellower than the orange $H = 45^\circ$ yield paints which become redder with increasing strength whilst those which are redder, become yellower. A very similar pattern was observed with dyes¹⁰ and Fig. 3 shows the change in hue as a function of the mean hue angle. This shows that there are two "angles of inflection," at 55° (orange) and 200° (blue-green) and a region of minimum change in hue, at 320° (violet). These changes of hue with increasing strength do not appear to have been quantified before.

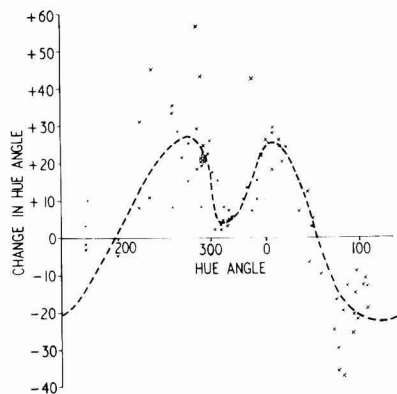


Fig. 3. Change in hue angle with increasing strength of textile dyes

The LC diagram ignores changes in hue and is thus a plot of lightness, L against saturation, C . The strength lines fell into two classes: the first consisted of those pigments which gave paints whose saturation steadily increased with increasing pigment-to-white ratio; the second consisted of those pigments which gave paints whose saturation reached a maximum well below a pigment:white ratio of 100:0. Typical examples of the first class are shown in Fig. 4 (the inclusion of all curves would have caused confusion by overlapping). Most inorganic pigments fell into this class, as did most organic yellows and oranges. Typical examples of the second class are shown in Fig. 5, and most organic

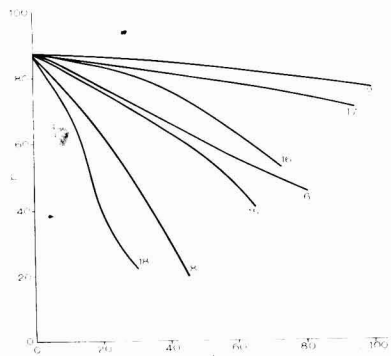


Fig. 4. Strength lines in LC diagram

pigments other than yellows and oranges fell into this class. The only inorganic pigment of the eight studied that fell into this class was Prussian Blue (Pigment Blue 27). The discovery that certain pigments behaved in this manner was first made by Tilleard in 1942¹⁴ using a visual colorimeter. It is not possible at present to offer any detailed explanation as to why pigments should vary in this manner though it is undoubtedly a consequence of differences in refractive index; most dyes give LC curves of the second class.

The variable of dirtiness

BS 1611 defines the terms "dirtier" and "cleaner" by reference to differences "apparently due to the presence of more or less black than in the original sample." This variable was investigated by the technique of computer match prediction in

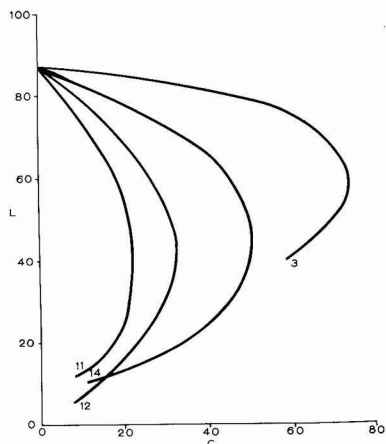


Fig. 5. Strength lines in LC diagram

reverse: the XYZ values of a number of paints containing the same chromatic pigment:white ratio but increasing amounts of carbon black were computed and converted to ANLAB co-ordinates. The results showed that the addition of small amount of black caused a negligible change in hue, except in the case of the yellow pigment which became decidedly greener. The principle effect was on the lightness and the saturation and thus most readily seen in the LC diagram of Fig. 6. Increasing dirtiness thus corresponds to a

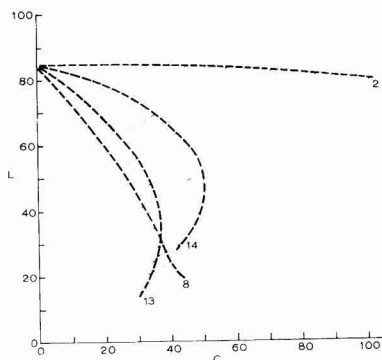


Fig. 6. Dotted lines . . . strength lines in LC diagram; solid lines dirtiness lines

decrease in both L and C , the ratio between them being dependent on the position of the paint which was free from carbon black in the LC diagram. The dyer's variable of dullness was found to be identical¹⁰.

The qualification of ΔE values

The investigation just described has revealed that, whilst the colourist's method of describing perceived colour differences is not simply related to the fundamental variables of colour measurement (i.e. XYZ or lightness, dominant wavelength and excitation purity) or even to the established variables of perceived colour, e.g. hue, value and chroma, it does form a consistent system. One of the authors (ACC) has therefore

been able to write a computer programme in BASIC for use on IBM's Call 360 time sharing service which, in addition to converting any measurements, (e.g. XYZ, RGB, 16 point reflectances, etc.), into the ANLAB values LABCH and ΔE against a nominated standard, will also print out how the dyer would describe the difference using the terms redder, yellower, greener, bluer/brighter, duller/weaker, stronger, in order of the importance of their contribution to the total colour difference.

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Discussion at the London Section

MR J. A. L. HAWKEY asked how the sensitivity of the human eye related to variations in ΔE with change in hue.

MR K. McLAREN said that the sole objective of the complex mathematics involved in ΔE calculations was to make ΔE linearly related to the sensitivity of the eye to small changes at any point in colour space. However, one had to be very careful how one interpreted sensitivity. It was widely believed that the eye was relatively insensitive to yellows, but this only applied to changes in chroma or saturation: for changes in hue, one of the two peaks of eye sensitivity occurred at 590nm.

MR HAWKEY referred to the experience of saturation decreasing with increases of strength, and cases with certain pigments where this did not occur. It had been suggested that refractive index might be one factor involved. This could be said to be confirmed by the difference in a match of an artist's water colour, which to a colour matcher appeared to have saturation parallel to strength, but his match at this strength lacked saturation.

MR McLAREN said his experience had been mainly confined to textiles, and as the dyer was never conscious of saturation as a variable of perceived colour, he would not like to comment.

MR N. M. SHEPPARD asked whether Mr McLaren

considered that colour assessments using the individual colour acuity in comparative examination of a particular colour difference would provide a more distinctive array of results than a general panel assessment?

MR McLAREN said that in his experience a professional colourist who was of reasonable health and age would be as good as anyone else throughout the colour range. There were no indications that some were better than others in particular colour areas. However, the use of a single observer could be dangerous: the whole of the "MacAdam Ellipse" data was now suspect because his observer had been found to differ markedly from other observers who agreed with each other (Wyszecki, G. and Fielder, G. H., *J. Opt. Soc. Amer.*, 1971, **61**, 1135).

DR T. M. MOYNEHAM said that he was concerned about the reliance upon the Davidson-Friede work, which used nine colourists from the same firm, all of whom might have been trained by one person and, therefore, were not a truly objective sample of observers. Could this explain the correlation between the ΔE limits of acceptance from this panel and the parameters of the Adams-Nickerson formula, whereas no correlation appears with the results from Jaeckel's observers?

MR McLAREN said that analysis of the Davidson and Friede results had shown that no formula was better than all the others to an extent which was statistically significant. Jaeckel's work, however, did and, whilst different formulae were best in each series, overall, formulae of the Adams Chromatic Value type were best. When his results and those of Davidson and Friede were combined, this type of formula was significantly better than any other.

MR C. E. WILLIAMS asked Mr McLaren to comment on whether he considered the work of Halstead, Stainsby and others on the subject of colour differences to be relevant to the present subject. These workers were concerned with the comparison of subjective differences between Munsell samples illuminated by different light sources and the corresponding objective differences, expressed in uv co-ordinates. They gave a considerable amount of statistical data on this comparison.

MR McLAREN said that these workers were interested primarily in assessing the colour-rendering properties of fluorescent lamps. They were, therefore, concerned with colour differences very much larger than those of commercial significance in the paint and textile industries. Though they found the CIE 64 formula (based on uv co-ordinates) to be reliable for this purpose, they did not compare it with any other, so there was a possibility that the Adams-Nickerson formula might be even more reliable.

MR D. E. EDDOWES (Chairman) asked whether it was possible to apply colour-difference formulae of the ANLAB type to fluorescent and metallic shades?

MR McLAREN said that, in the former case, it was theoretically possible but, as the constants had been chosen for non-fluorescent colours, it might not be the most reliable because fluorescent colours were not only brighter but also more saturated. These reservations would be equally applicable to other formulae, however, so the ANLAB formula could still be the best. The major problem concerning metallic shades was viewing geometry and, until this was solved, the choice of formula would have to be left open.

An investigation into the durability characteristics of masonry finishes*

By J. Boxall

Cementone Ltd., Chandos Road, Buckingham, Bucks

Summary

The durability characteristics of some commercially available masonry coatings have been assessed by natural and accelerated weathering and by water absorption testing.

Coatings studied were drawn from emulsion- and resin-based systems deposited at both low and high film thicknesses.

Keywords

*Types and classes of coating
masonry finish*

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

It is demonstrated that high film build coatings do not necessarily afford greater protection than do the more conventional coatings and that both emulsion and resinous binders appear to give comparable durability in correctly formulated coatings.

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

Une investigation de la durabilité des peintures pour maçonnerie

Résumé

On a apprécié la durabilité des peintures pour maçonnerie de commerce, par l'exposition aux intempéries, les essais de vieillissement accélérés, et par le dosage de l'absorption d'eau.

Les peintures utilisées au cours de cette étude étaient basées sur, soit des émulsions, soit des résines, et elles étaient appliquées en couches d'une épaisseur élevée ou basse.

On démontre que les peintures "high-build" ne rendent pas nécessairement un niveau de protection supérieur à celui des peintures plus traditionnelles, et également que les liants à base d'émulsions ou de résines semblent posséder une durabilité convenable dans le cas où les peintures sont d'une composition convenable.

Eine Untersuchung der Dauerhaftigkeitscharakteristiken von Wandfarben

Zusammenfassung

Die Dauerhaftigkeitscharakteristiken einiger im Handel befindlichen Wandanstriche wurden mittels natürlicher und beschleunigter Bewitterungssowie Wasserabsorptionsteste geprüft.

Die zur Untersuchung benutzten Anstrichfarben wurden von Systemen auf Emulsions- und Kunstharzbasis ausgewählt und sowohl in dünner als auch dicker Filmschicht aufgetragen.

Es wird gezeigt, dass dicke Filme bildende Anstrichmittel nicht notwendigerweise grösseren Schutz verleihen, als die konventionellen, und dass in richtig formulierten Vorschriften beide, d.h. solche mit Emulsions- oder mit Kunstharzbindern hinsichtlich Dauerhaftigkeit vergleichbare Resultate zu ergeben scheinen.

Исследование характеристик стойкости отделок каменной кладки

Резюме

Оцениваются характеристики устойчивости некоторых промышленно доступных покрытий кирпичных кладок, путем естественного и ускоренного выветривания и испытанием на поглощение воды.

Исследованные покрытия получены из эмульсионных и смольных систем, осажденных как при низких так и высоких толщинах пленки.

Показано что покрытия с высокой толщиной пленки не дают обязательно большую защиту чем более традиционные покрытия и что как эмульсионные так и смольные крепители дают сравнимую стойкость в правильно сформулированных покрытиях.

Introduction

The function of an exterior masonry finish is twofold: it must protect the substrate from aggressive atmospheric contaminants and it must provide a decorative finish for its useful life whilst requiring a minimum of maintenance.

The choice of masonry coatings is wide, ranging from relatively low build emulsion and resinous systems to the very high build textured materials. From theoretical considerations, it is evident that a high film build coating should

delay the ingress of contaminants more effectively than a lower thickness coating, thus providing greater protection to the substrate.

Work by Whiteley and Rothwell¹ on the durability of these high build masonry coatings in comparison with the more conventional low build coatings has indicated that high build masonry coatings produce the better overall performance.

The Department of Architecture and Civic Design of the

*Paper based on a dissertation submitted in a successful application for Licentiate ship in the Technology of Surface Coatings.

Greater London Council reports on proprietary masonry finishes of all types² and the findings are generalised in an Annual Report³. From these reports it appears that the high build textured coatings generally are capable of providing outstanding durability characteristics.

It is the purpose of the work reported here to investigate the durability characteristics of a range of exterior masonry coatings, including both high and low film build materials. Accelerated and natural weathering techniques, and measurement of the water absorption characteristics of coatings attached to a standard substrate, have been used to assess comparative film performance. These findings are related to the formulation variables of the coatings.

Experimental

Materials

The coatings applied were typical of those used for the decoration and protection of external masonry, and in all cases the colour of the paints tested was white. Prior to testing, qualitative analytical tests were made to determine the major constituent raw materials in the paint. Although the limited knowledge of the pigmentation thus obtained did not enable pigment volume concentration to be determined, it was possible to calculate the pigment : binder weight ratios of the coatings.

Coating A—Based on a styrene/acrylic emulsion, pigmented with titanium dioxide, calcareous matter and silica sands.

Pigment: aggregate: binder ratio, 2.8:1.5:1.0.

Coating B—Based on pva/acrylic emulsion, pigmented with titanium dioxide, mica, silica sands and a large particle sized mineral aggregate.

Pigment: aggregate: binder ratio, 3.3:1.1:1.0.

Coating C—Based on pva/acrylic emulsion, pigmented with titanium dioxide and calcareous matter.

Pigment: binder ratio, 2.9:1.0.

Coating D—Based on vinylidene chloride and acrylic emulsions, pigmented solely with titanium dioxide.

Pigment: binder ratio, 0.65:1.0.

Coating E—Based on a long oil alkyd, pigmented with titanium dioxide, zinc oxide, mica, asbestos fibre, and fine siliceous material.

Pigment: fibre: binder ratio, 1.7:0.9:1.0.

Coating F—Based on a long oil alkyd and polybutene, pigmented with titanium dioxide, zinc oxide, mica, asbestos fibre, large particle size exfoliated lightweight mineral aggregate and fine siliceous material.

Pigment: aggregate: fibre: binder ratio, 1.2: 0.1:0.6:1.0.

Coating G—Based on a long-oil alkyd resin, pigmented with titanium dioxide, barytes and china clay.

Pigment: binder ratio, 3.6:1.0.

Coating H—Based on chlorinated rubber and tung oil phenolic varnish, pigmented with titanium dioxide, calcareous matter and barytes.

Pigment: binder ratio, 3.1:1.0.

Panel preparation

All testing was performed using unweathered 5mm thick fully compressed asbestos cement sheet as the substrate.

Test panels were coated by spray or brush application as recommended in the information given by the paint manufacturers. Panels for the accelerated weathering tests were 15 × 10cm, for natural weathering 30 × 15cm, and for the water absorption tests 10 × 10cm.

The method of application and film characteristics of the coatings are summarised in Table 1.

Test methods

Accelerated weathering

The coated 15 × 10cm panels were aged for seven days at 20°C and 50 per cent RH in a dust- and draught-free cabinet prior to testing. During the ageing period, the backs and edges of the panels were treated with a standard water resistant, pigmented, chlorinated rubber coating.

Table 1
Characteristics of test coatings

| Coating | Application | Number of coats | Appearance of dried film | Average dry film thickness (μ) |
|---------|-------------|-----------------|--------------------------|--------------------------------------|
| A | Brush | 2 | Matt, fine textured | 250-300 |
| B | Spray | 1 | Matt, coarse textured | 1250-1500 |
| C | Brush | 2 | Matt, smooth | 125-175 |
| D | Brush | 2 | Semi-gloss, smooth | 100-125 |
| E | Spray | 1 | Matt, coarse textured | 1250-1500 |
| F | Spray | 1 | Matt, coarse textured | 1250-1500 |
| G | Brush | 2 | Eggshell, smooth | 125-150 |
| H | Brush | 2 | Matt, smooth | 125-150 |

After ageing, the test panels were placed in an accelerated weathering apparatus conforming to BS 3900 Part F.3 for 1000 hours. The panels were then removed from the apparatus and compared with unexposed specimens.

Natural weathering

The coated 30 × 15cm panels were aged for seven days at 20°C and 50 per cent RH in a dust- and draught-free cabinet prior to testing. After backing and edging with a water resistant, chlorinated rubber coating, the panels were exposed at a rural site, with the painted side uppermost, in a south westerly direction at an angle of 45° to the horizontal. At regular intervals the test panels were compared with the unexposed specimens.

After accelerated testing, and during natural weathering exposure, the panels were assessed visually for signs of chalking, checking, cracking, flaking, dirt retention, gloss and colour change. Assessment was on an arbitrary 0-10 scale, where 0 indicated no change, and 10 complete failure of the coating.

Water absorption

A series of 10 × 10cm coated panels was aged for seven days at 20°C and 50 per cent RH. During the ageing period, the panels were backed and edged with a water resistant, chlorinated rubber coating. After ageing, the panels were weighed, then totally immersed in distilled water. The panels were removed periodically, blotted free from surface moisture and reweighed. The panels were then reimmersed and the test continued.

To calculate the amount of water permeating through the coating used for backing the test panels, blanks were prepared by coating both faces and edges of asbestos cement panels with the backing paint. The blank was then tested at the same time and in an identical manner to the test specimens. The amount of water absorbed by an area corresponding to one face and the edges of the blank was determined from the total absorption of the whole area. The figure thus obtained was subtracted from each of the test specimens absorption figures to give the amount of water permeating the test face.

All tests were performed in duplicate.

Results

The water absorption of the coating and its substrate, in grams of water absorbed per 100cm² of paint film, was plotted against immersion time in hours. Since the correlation between the absorption of the duplicate panels was close,

the arithmetical mean was used when plotting the results. Fig. 1 shows the water absorption characteristics of the emulsion-based coatings, and Fig. 2 those of the resinous coatings.

From Figs. 1 and 2, it is evident that the mode of water absorption is similar for all of the coatings, with the exception of G and H. A rapid initial absorption is followed by a "plateau" effect, with a maximum occurring after 100 to 250 hours of immersion. Coatings G and H exhibited a gradual, linear increase in absorption with immersion time, see Fig. 2.

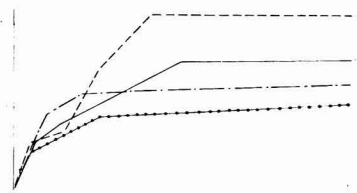


Fig. 1

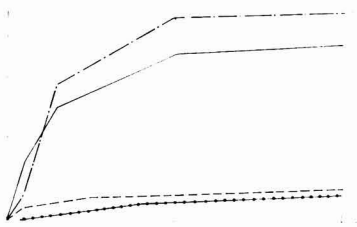


Fig. 2

It is also evident from the Figures that the higher film build coatings, A, B, E and F, also exhibited higher water absorptions than did the lower thickness coatings, C, D, G and H.

Changes in the appearance of the films due to chalking, checking, cracking, flaking, dirt retention, gloss and colour change, after accelerated weathering for 1000 hours and natural weathering for one year, are summarised in Tables 2 and 3 respectively.

From Tables 2 and 3 it can be seen that the performance of coating A under accelerated weathering conditions was extremely good, showing signs only of a uniform yellow discoloration. After natural weathering, however, some signs of film checking were evident. Dirt retention was slight considering the aggregated texture of the coating.

Table 2
Accelerated weathering

| Coating | Chalking | Checking | Cracking | Flaking | Dirt Retn. | Gloss | Colour |
|---------|----------|----------|----------|---------|------------|-------|--------|
| A | 0 | 0 | 0 | 0 | 0 | 0 | 6Y |
| B | 0 | 4 | 0 | 0 | 2WS | 0 | 4Y |
| C | 4 | 10 | 0 | 0 | 0 | 0 | 6Y |
| D | 0 | 0 | 0 | 0 | 0 | 0 | 4Y |
| E | 0 | 0 | 0 | 0 | 0 | 0 | 2/4YP |
| F | 0 | 0 | 0 | 10 | 0 | 0 | 2/4YP |
| G | 0 | 0 | 0 | 0 | 0 | 0 | 4Y |
| H | 0 | 0 | 0 | 0 | 0 | 0 | 4Y |

Y = yellowing.
YP = irregular yellow patches.
WS = water staining.

Table 3
Natural weathering

| Coating | Chalking | Checking | Cracking | Flaking | Dirt Retn. | Gloss | Colour |
|---------|----------|----------|----------|---------|------------|-------|--------|
| A | 0 | 2 | 0 | 0 | 3 | 0 | 2D |
| B | 0 | 4 | 3 | 10 | 4 | 0 | 4D |
| C | 2 | 3 | 0 | 0 | 2 | 0 | 1D |
| D | 0 | 0 | 0 | 0 | 4 | 0 | 2D |
| E | 0 | 0 | 0 | 0 | 4 | 0 | 4D |
| F | 0 | 1 | 0 | 0 | 4 | 0 | 4D |
| G | 0 | 0 | 10 | 10 | 1 | 0 | 1D |
| H | 0 | 4 | 0 | 0 | 2 | 0 | 1D |

D = colour altered by dirt.

Coating B exhibited moderate checking under accelerated weathering, but on natural weathering the film failed completely by cracking and flaking from the surface. It was noted during natural weathering that film breakdown was evident initially as checking. This checking increased in severity and ultimately led to flaking after approximately nine months' exposure.

Coating C failed during accelerated weathering by complete loss of film integrity due to checking, which was accompanied by a high degree of chalking. After one year's natural weathering, this trend towards checking and heavy chalking was evident and dirt retention was of a low order, consistent with the amount of chalking that occurred.

Coating D performed satisfactorily under both accelerated and natural weathering. However, the dirt retention of the coating was high in view of the smooth semi-gloss surface.

Coating F, after accelerated weathering, could be lifted from its substrate as a single coherent sheet. A white powdery deposit was present on the substrate after the film was removed. After one year's natural weathering, only slight signs of surface checking were evident, and film adhesion appeared to be satisfactory. The irregular yellow patches found after accelerated weathering and the degree of dirt retention on natural weathering were similar to those of coating E, the other resinous high build finish.

Coating E performed satisfactorily under accelerated and natural weathering conditions, but did exhibit irregular yellow patches after accelerated weathering and considerable dirt retention on natural weathering.

It was found that coating G performed satisfactorily on accelerated weathering, exhibiting only signs of moderate yellowing. However, after six months' natural weathering, the film integrity had failed completely by cracking and flaking. No signs of checking were evident during the weathering of the panel prior to failure by flaking.

Coating H performed satisfactorily on accelerated weathering exhibiting only moderate yellowing. On natural weathering, some film checking was evident.

Discussion

Water absorption

From the water absorption characteristics of the coating/substrate system shown in Figs. 1 and 2, it is apparent that the higher film build coatings A, B, E and F absorb more water than do the lower build coatings. The reason for this does not appear to be related to the binder type,

since both the emulsion bound coatings A and B and the resin bound coatings E and F appear to exhibit similar water absorption characteristics.

It is believed that the high water absorption characteristics of these coatings can be related to pigment formulation variables.

Coatings A, B and F contain large particle size mineral aggregate, the presence of which produces a textured effect within the dried film. Although high build emulsion bound coatings can generally be formulated with higher pigment:binder ratios than classical theory allows, it is thought that, in coatings A and B, both with pigment filler: binder ratios of over 4:1, the amount of binder is insufficient for exterior use. Subsequently upon drying, a micro-stressed or micro-cracked area occurs around each particle of aggregate, where it protrudes through the dried film surface. Water can therefore permeate through these defect areas into the bulk film structure. Such micro-cracked areas were evident upon microscopic examination of the dried coatings A and B.

The alkyd-based coating F contained an exfoliated light-weight mineral aggregate, and it is possible that water could enter into the film via the relatively open structure of this aggregate. The effect would occur regardless of the high binder content of the coating, since the exposed areas of aggregate at the pigment/water interface would be covered only with a thin layer of pigmented media. Therefore, these areas would be more susceptible to permeability effects than the surrounding film.

Both the alkyd-bound coatings, E and F, contained asbestos fibres and on immersion in water, "wicking effects" could be induced through the fibre structure and so introduce water into the film. Water, once introduced into the film structure, could react with and swell the alkyd resin owing to interaction with the carboxyl groups of the polymer. During absorption testing of these two coatings, it was observed that film swelling and blistering occurred after approximately 100 hours' immersion.

The lower absorption characteristics of the low build coatings C, D, G, and H, compared with high build coatings is considered to be primarily due to the more coherent film structure produced by closer pigment particle grading and packing within these coatings. It can be seen however, that, of the low build coatings, the emulsion-bound coatings C and D possess somewhat higher absorptions than do the resin-bound coatings G and H. This would be expected, since emulsion coatings always contain a relatively high proportion of water soluble matter, e.g. cellulosic thickeners and surfactants, compared with resin-bound coatings.

It is possible, therefore, that in coatings giving low film builds, the nature of the binder, whether emulsion or resinous,

does influence the degree of water absorption, and also that this effect may be independent of pigment type or content.

Accelerated weathering

The accelerated weathering results shown in Table 2, demonstrate that all of the coatings exhibited a yellow discoloration. This colour change is presumably due to the ultra-violet light from the arc lamp of the test apparatus affecting some constituent of the film. It is probable that the constituent affected is common to all of the coatings examined, e.g. the polymer, but further work would be needed before any conclusion could be drawn on this particular aspect of the investigation.

Both coatings B and C exhibited film checking, coating C to such an extent that the film was considered to have lost all protective or decorative value. With a pigment: binder ratio of 2.9: 1.0, and a probable pigment volume concentration of 50-55 per cent, it is possible that the coating is somewhat underbound for external use. The observed high chalking rate tends to support this theory.

The checking tendency of coating B, the high build emulsion based coating, is believed to be a progressive shrinkage on drying, as noted in the water absorption section above. The checked areas, after testing, tended to emanate from the place where aggregate protruded through the film.

The poor performance of coating F, which could be lifted as sheet from the substrate after testing, is difficult to explain, since on natural weathering its performance did not suggest this type of failure. It is possible that film saponification occurred at the paint/panel interface, owing to attack by alkali. The alkali is probably liberated from the asbestos cement in the presence of moisture, which was carried through the film by the mechanism proposed in the water absorption section above.

Natural weathering

From the natural weathering results shown in Table 3, it can be seen that all the coatings tended to retain dirt. As would be expected, the high build textured finishes A, B, E and F, retained the most dirt, with coating A retaining the least. This is presumably related to the fact that coating A contains the smallest particle size texturing aggregate of the high build coatings examined, and therefore has a correspondingly lower order of surface topography to trap dirt.

The film failure of coating B is considered to be due to a progressive shrinkage on drying, as noted in the water absorption and accelerated weathering sections, probably aggravated by moisture and other contaminants penetrating and lifting the film.

Coating D, the vinylidene chloride/acrylic emulsion-bound semi-gloss paint, retained the highest amount of dirt of the low film build coatings examined on weathering. It is believed that this is due to the relative "softness" of the film compared

with the other coatings, any dirt lying on its surface tending, over a period of time, to become engrained. In fact, it was the only low build coating which, after exposure, could not be cleaned of its retained dirt by rubbing with a damp cloth.

The failure of coating G by cracking and flaking after only six months' exposure is difficult to explain. The effect may possibly be due to saponification or to lack of adhesion to its substrate, but formulation defects were not indicated during the water absorption or accelerated weathering testing.

It is interesting to note that the yellowing observed after accelerated weathering on all coatings was not evident after natural weathering. It is possible that the absence of yellowing is due to retained dirt and moisture on the coating surface filtering or reflecting incident ultra-violet light.

With the exception of coatings F and G, it is evident that, although there was reasonable correlation between accelerated and natural weathering, film breakdown was generally more severe after natural weathering than during the accelerated weathering tests.

Conclusions

It has been demonstrated that high film build coatings are not necessarily of greater durability than are coatings of low film thicknesses.

It is apparent also that, in correctly formulated coatings, both resin and emulsion binders can give comparable durability characteristics. However, choice of pigmentation appears to be more critical in the formulation of high build textured coatings than in the more conventional low build coatings, particularly in respect of water absorption and diffusion mechanisms.

The results also indicate that accelerated weathering and water absorption tests provide a reasonably accurate guide to the probable film durability when exposed to natural weathering.

Acknowledgments

The author would like to thank the directors of Cementone Ltd. for permission to publish this work. Appreciation is also extended to Dr J. A. von Fraunhofer for the continued interest he has shown.

[Received 27 July 1972]

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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 April issue:

The surface treatment of powdered glass fibre and the stability of dispersions in polymer solutions by K. Ishii, T. Matsonaga and Y. Tamai

Paint film thickness measurement: an X-ray fluorescent technique for thin film application by R. D. Murley and H. Smith

The effect of pigments on the penetration of sulphur dioxide into coatings by H. Klicova, B. Knupek and M. Svoboda

Some physical properties of pigments. Part II. by M. J. Smith

Thames Valley Section

Powder coatings

The third technical meeting of the Thames Valley Section Student Group was held at Slough College on 7 December 1972 and attended by 50 Members and visitors. The visiting speaker, Mr H. T. Chellingsworth of Ault & Wiborg Ltd., Industrial Finishes Division, presented a lecture entitled "powder coatings."

Mr Chellingsworth opened the lecture by giving a brief history of powder coating technology. This involved the development of application methods and the materials used, ranging from early pre-heated objects with thick coatings, to electrostatically applied powders giving relatively thin films. Most of the development work on powder coatings had been done in Western Europe and, although powders had been known for many years previously, it was not until the early '60s that powder coating became a commercially viable prospect. This was due to the liaison developed between the chemical industry producing the powders and the plant manufacturers designing the application equipment.

The initial step was the selection and development of a suitable resin from which to obtain the powder. This presented many problems, particularly when one considered the requirements: non-sticky when cold—essential because of pre-mixing with pigments; a well defined melting point and good pigment melting properties when molten; good powder formation and reasonable flow. As a result of development, various generations of powders had evolved, perhaps the most dramatic improvement being the reduction of stoving time. Early powders required up to 30 minutes at 185 C

whereas recent powders required only 10 minutes at 185 C to be completely cured. This was due in the main to the use of high shear extruders in powder production as opposed to heated Z-blade mixer, consequently a fast acting catalyst could be used because the resin no longer experienced high temperatures for long periods during powder production.

As a result of this and many other contributory factors the demand for powder coating increased. One of the main advantages of using powders was the almost total lack of pollution, since no solvent was involved in the application. The essence of the application method being the formation of a cloud of low velocity charged particles propelled towards an earthed article. The particles adhered to the article and the film thickness became self-limiting owing to the insulation caused by the build-up of the powder deposit. No pre-heating of the article was required as in earlier fluidised bed systems. Once coated the article was placed in an oven where the powder would melt, flow and cure rapidly.

The speaker concluded his lecture by showing various slides of operational powder coating plants and illustrating the degree of automation that could be achieved. This was followed by a lengthy question time, during which many interesting points were raised. This illustrated the response evoked by a most interesting lecture. The Chairman concluded the meeting by calling upon Mr C. Woods to propose a vote of thanks. All those present expressed their appreciation in the usual manner.

D.A.P.
R.F.G.

Information Received

Strong profit recovery in paint industry

According to a business ratio survey of the paint and printing ink industry published by Inter Company Comparisons Ltd., a rise in pre-tax profits of 15 per cent occurred in the year 1971/72, compared with the previous year. Following the 11 per cent rise recorded in 1970/71, this is an encouraging sign for the industry.

Copies of the detailed report are available from ICC at a price of £20.00.

Schwegmann agency for CIL

Compounding Ingredients Ltd. has recently announced an agency agreement to distribute in the UK the range of paint additives made by Bernd Schwegmann K.G. Amongst the products handled will be *Antigel* viscosity stabiliser, *Korrodur* rust passivator, the *Blister-Free* products for acceleration of air-release for solvent-based systems, and the *Schwegocide* preservatives.

Both BS ranges from Hird Hastie

As well as supplying all 86 shades in the new BS 4800 range, Hird Hastie Paints Ltd. has announced that it will continue to supply colours to the BS 2660 specification, as it is felt that the changeover will take place only gradually, and large contractors will have to continue to use the BS 2660 range for some time, at least until current contracts are completed. No names will be given to the colours in the new specification, which will be known only by the BS reference.

South American office for Arnold Services

Arnold Services Ltd. has opened an office in Quito, Ecuador, with full laboratory facilities. The office will specialise in the Andean countries, and will offer market studies, *in situ* finding, technico-economic and political information, and samples and supplies of raw materials.

Kronos to handle Oncor

As from 1 February, Kronos Titanium Pigments Ltd. will distribute the Oncor range of anti-corrosive pigments previously handled by Berk Ltd. Oncor pigments are manufactured in the USA by NL Industries Inc., of which Kronos is a wholly-owned subsidiary.

Polybond merger

The three associated companies Polybond Ltd., Corrosion Ltd., and Furnascope Ltd., merged at the beginning of this year to form one company trading under the name Polybond Ltd. The company will have three divisions: Furnascope Sales Division, dealing with refractory coatings; Corrosion Sales Division, primarily concerned with sales of Sansol water-thinned epoxy coating; and Building Products Division.

This step has been taken with the aim of improving the company's efficiency and customer efficiency throughout the world.

DH Industries take Coors agency

DH Industries Ltd. is to act as UK and Irish distributor of the *Mini-Media* made by Coors Porcelain Co. of USA, it was announced recently. *Mini-Media* are for use

in sand, bead and pearl mills, and are available in two types: type "M," medium density ceramic media, and type "A," high density alumina/ceramic media.

British Decorators Association

The National Federation of Master Builders of England and Wales has changed its name to British Decorators Association, with effect from January. The change has been made because it was felt that the earlier title tended to convey the impression of an old-fashioned organisation, or a trade union, rather than the progressive employers' federation that the BDA now is.

Whitfields to supply small lots of Bayer products

Small quantity orders of surface coating materials produced by Bayer Chemicals Ltd. will now be supplied by Whitfield & Son Ltd. This arrangement has been made to ensure the best possible service for customers ordering less than 1,000kg lots. Whitfields, who will hold a full range of stock, will give customers full technical service, backed up by Bayer's own technical reserves.

Powders research unit for Leeds

A new programme of fundamental research into the solid state behaviour of dyes, pigments and pharmaceuticals, is being initiated at Leeds University, aided by a grant of £84,278 from the Wolfson Foundation. Two specific projects are planned: The technical preparation of stabilised physical form and The nucleation of pigment crystals and their technical behaviour, with a more general investigation of The general physical stability of dye, pigment, and other organic solutions. Three post-doctoral fellows will be appointed, and several postgraduate research students will work with them.

Rohm & Haas (UK) Limited

As from 1 January 1973, Lennig Chemicals Ltd., has changed its name to Rohm & Haas (UK) Limited, taking the name of its US parent company.

New products

Polymethacrylates from Degussa

A new range of fine granular polymethacrylate resins are being marketed by Degussa under the trade name *Degalan*. Particularly recommended for coatings applications, the resins are available in UK from Bush Beach & Segner Bayley Limited, which is also handling Degussa's *Degatur* methyl methacrylate based cross-linkable resin range.

Bench-top vacuum mixer

A bench-top vacuum mixer that allows the ingredients to be thoroughly mixed and de-aerated simultaneously has been developed by Cunnington & Cooper Ltd. The mixer features orbital/planetary movement of a twin-bladed mixer paddle fitted with PTFE scraper blades to allow for variations in contour of the transparent mixer bowl. Mixing can be carried out under a vacuum of 29in of mercury, and

ingredients can be added at any stage without breaking the vacuum, by means of a hopper attachment.

Addition to Antisettle range

Cray Valley Products Limited has introduced *Antisettle HT*, a bodying agent to complement the existing *Antisettle CVP*. The earlier product, although successful in most applications, proved sensitive to high temperatures during manufacture. *Antisettle HT* has been produced primarily for manufacture at high temperature, mainly necessary in the paint industry.

Cray Valley will supply samples of either product on request.

Metered spraying equipment

The *Spraymet* is a new system for spraying of precisely-controlled quantities of liquids, developed by Metering Pumps Ltd. The system is basically a motor-driven compressor coupled to up to six pumpheads, which are connected to a maximum of three sprayheads. Each pump can meter a different liquid and is fitted with a micrometer to adjust output from zero to a maximum of 0.7gph, pump output being directly proportional to micrometer setting. Each sprayhead has two separate air streams: one to vary the degree of atomisation, and one to shape the spray. By varying the individual parameters, a controlled, measurable spray with correct characteristics can be obtained.

The *Spraymet* is supplied as a single complete unit with carrying handle.

New phosphating base

Epifos is a new zinc phosphate based product for metal phosphating, introduced by the Jenolite Division of Duckhams Oils. The system is supplied as two components, *Epifos* and *Supercatalyst*, which are mixed in the correct proportions for bath or spray application. Deposited coatings are rinsed in water and sealed with a ½ per cent solution of *Controx 60*.

Advantages of the system are claimed to be speed of phosphate formation, fineness and density of coatings formed, and excellent adhesion to top coats.

Permanent Red from Hoechst

Farbwerke Hoechst AG has introduced *Permanent Red F6RK*, a new bluish red pigment for printing inks. A monoazo pigment without lake-forming groups, *Permanent Red F6RK* is said to have high tinctorial strength, good gloss and light fastness.

Literature

Total protection plan from Dixon

As part of its policy to revise all product literature, Dixon's Paints Ltd. has introduced a new form of technical brochure aimed at the paint specifier. The "total protection plan" consists of a coloured A4 size folder containing details of the four major Dixon products for buildings, *Rogston*, *Ced-O-Dress*, *Fleck Paint* and *Chemproof*.

Dixon's is also to introduce a regular bi-monthly news bulletin aimed at the specifier.

Water-soluble polymer study

The latest multi-client, techno-economic study from Skeist Laboratories Inc. is concerned with "Synthetic water-soluble polymers." The current status and emerging technical and commercial developments in the field will be analysed in the study, which will appear in June 1973.

Burrell profile

A four-page profile describing the activities of Burrell & Co. Ltd. was recently produced by the company, and circulated to shareholders, customers and trade friends.

Pigment literature from Hoechst

Farbwerke Hoechst AG has recently issued two technical information sheets: "Instructions for testing pigments on an automatic muller" and "Diaryl yellow pigments for publication gravure inks."

The transport and storage of oleochemicals

Technical Publication No. 101 from Price's Chemicals Limited deals with the transport and storage of oleochemicals. Full details of Price's bulk transport facilities, and of storage and handling methods for liquid and solid products are given.

Price's has also issued Technical Publication No. 102, "The application of Price's products in soaps, toiletries and cosmetics."

Cost reduction on Warson emulsion bulletins

H. Warson (Chemical Consultant) Ltd. has announced that, owing to widespread acceptance, the series of bulletins it produces on emulsion polymerisation and on emulsion polymer applications can now be offered at a reduced subscription.

Each series is issued as six bulletins throughout the year, and the subscription rate for either title is now £52; both series may be purchased at a cost of £99.

Index to Du Pont industrial chemicals

The industrial chemicals department of Du Pont has published a quick-reference index of more than 200 products, both commodity and proprietary, that it can supply throughout the world.

Conferences, courses, symposia

Principles of colour technology

For the ninth consecutive year, Rensselaer Polytechnic Institute of Troy, New York, USA, is to organise a series of summer courses in colour technology. The courses will be "Principles of colour technology" held 16-20 July, "Colour technology for management" held 26-27 July, and "Advances in colour technology" held 30 July-3 August.

The courses are under the direction of Dr F. J. Billmeyer Jr. and Mr M. Saltzman, both eminent in the field. Full information can be obtained from the office of Continuing Studies, Colour Technology Programme, Rensselaer Polytechnic Institute, Troy, New York 12181.

Corrosion control on structural steelwork

A one-day symposium with the above title is to be held at the Derby College of Art and Technology. The symposium, which will contain four papers under the chairmanship of Mr F. D. Timmins, is aimed at providing a platform for plain talking on the four principal methods of avoiding corrosion on structural steelwork. Full details from: Mr B. Marshall, Senior Lecturer in Materials Science, Derby College of Art and Technology, Keddleston Road Derby DE3 1GB.

Particle Workshop 1973

Loughborough University of Technology and Warren Spring Laboratory are to collaborate in organising Particle Workshop 1973, which will consist of four short courses: 29-30 March, "Measurement of particulate emissions from stacks," 2-3 April "Design of powder storage systems," 4 April "Measurement of powder strength" and 5-6 April "Cake Filtration." Full details are available from the Centre for Extension Studies, University of Technology, Loughborough, Leics. LE11 3TU.

Colour perception and control

Four courses on aspects of colour are to be held by Instrumental Colour Systems Ltd. at its Newbury premises. The first of these, on 1-3 April, is designed for colour chemists in research and industry, discussing colour perception and measurement, including a review of the mathematics involved. The second course, to be held 8-10 April, will be more advanced, and is aimed at senior scientists with a good understanding of the basic principles who require a mathematical course to bring them up to date with latest control techniques, whilst the third (2-3 May) is for technicians who require a more practical treatment. The final course, on 10 May, is for designers within the industry, and will be entirely non-quantitative.

Full details from Mr J. P. Smith, Instrumental Colour Systems Ltd., 13 Bone Lane, Newbury, Berks.

Corrosion symposium

The Materials Preservation Group (formerly the Corrosion Group) of the SCI is to hold a one-day symposium entitled "Corrosion and deterioration of metals and alternative engineering materials: some comparative assessments" at 14 Belgrave Square, London, SW1X 8PS, on Wednesday 28 March. Those interested should contact the SCI Assistant Secretary.

Bristol

The paint industry and the Common Market

Mr D. E. Eddowes, Editor of *Polymers, Paint and Colour Journal*, and Chairman of London Section, presented a paper entitled "The paint industry and the Common Market," to the Bristol Section at the Royal Hotel on Friday 27 November. A total of 19 Members were present.

After mentioning the development of the UK paint industry from 1920, Mr Eddowes outlined the formation of the larger paint groups and then established the current UK position. This was then compared and contrasted to the current European situation. The very knowledgeable and informative comparison included numerous statistics and was supported by slides illustrating the trends.

Mr Eddowes gave a personal view as to the changes which may occur when the UK joins the EEC. The effect of these changes on the cost of raw materials was also considered briefly, as were the EEC regulations on pollution.

Question time was opened by Mr F. Ruddick and a lively discussion followed, which was eventually closed by a vote of thanks by Mr L. Brooke.

R.F.N.

The economics of powder coating

Mr R. Mansell of Dunlop Ltd., presented a paper entitled "The economics of powder coating" to the Bristol Section at the Royal Hotel on Friday 8 December.

After explaining that his lecture was based on information obtained from the evaluation of a pilot automatic powder coating plant, the lecturer gave a detailed discussion on the technical merit and economics of powder coating as applied to the manufacture of car wheels.

Factors essential to the quality of the finish were mentioned, and the need to consider the less obvious economic factors stressed. The stages of conventional finishing were compared and contrasted to those required for powder coating.

The major economics necessary to offset the higher cost of powder raw materials were highlighted as: no need for demineralised water; the zinc phosphate wash could be replaced by cheaper iron phosphate; and, most important, obtaining the economies made in the electrophoretic dipping process.

A numerical example was studied, which resulted in powder coating costing fractionally more than a conventional coating system. This small increase was considered to be a very small premium for the added properties obtained by powder coating.

Further advantages, other than economic, were also claimed for powder coating, and their effect on the customer was considered.

The meeting was well attended and a vote of thanks warmly supported.

R.F.N.

Hull

Technology—kill or cure?

The third meeting of the present session was held at the College of Technology, Hull, on 4 December, when Dr C. Stoneman of Hull University gave a lecture with the above title.

Dr Stoneman took as his theme the breakdown of civilisation as a result of the exhaustion of resources essential to modern industry and discussed the prospects of technological advances providing substitutes if and when required.

The speaker expressed the view that the exponential rise in demand for most of the earth's resources was a consequence of industrialisation and technical advances, particularly in medical science, of the demand for higher living standards—itsself a result of technological advances, and of the materialistic emphasis of present day society. Technology had so far proved capable of dealing with acute shortages of vital materials, for example the invention of the Haber process for the manufacture of synthetic nitrogen fertilisers had made it possible for the earth to support a very much larger population than was considered possible hitherto. Frequently, more than one technical solution to such shortages was possible and the final choice was invariably governed by the profit motive. Such profit was calculated with respect to a particular manufacturing group and took no account

of concealed costs borne by the consumer or by society.

Transfer to substitute industries was frequently desirable on general grounds in order to conserve existing stocks of certain materials for alternative uses, but such changes were possible only over a protracted period because of the enormous capital investment in the existing activity.

In the second part of his lecture, Dr Stoneman illustrated the present rates of consumption of a variety of raw materials and presented forecasts of the number of years which remained before known reserves were exhausted; for example petroleum 20, natural gas 22, coal 110, aluminium (from bauxite) 31. The projected supply from known reserves of many less commonly occurring metals was fewer than 20 years. Dr Stoneman believed that some extension of these estimates could be expected as a result of the location of new sources, the working of leaner deposits, the recycling of scrap (presently 10-40 per cent for many metals) and of technological advances. It was through technical advances that Dr Stoneman maintained his confidence in the future, but he concluded that the time to act was now rather than when the crises were upon us.

After the discussion period, a vote of thanks for an interesting and enjoyable lecture was given by Mr J. C. Gibson. The meeting was attended by 12 Members.

J.A.H.

Manchester

New developments in clear coatings for exterior wood

A meeting of the Section was held on Friday 13 October 1972 at the Manchester Literary and Philosophical Society, 36, George Street, Manchester 1.

A lecture entitled: "New developments in clear coatings for exterior wood" was given by Dr E. R. Miller of the Building Research Establishment.

Dr Miller began with a consideration of the structure and properties of wood and of their implications for clear coatings. The shortcomings of existing types of clear coating were then discussed and recent work at the Princes Risborough Laboratory into the modification of clear coatings by the incorporation of extender pigments was described. Results were presented of investigations into the effect of extenders on the optical and mechanical properties and exterior durability of clear coatings.

A.McW.

The scanning electron microscope

A meeting of the Section was held on Friday 10 November 1972 at the Bolton Institute of Technology, Deane Road, Bolton, when a lecture entitled "The scanning electron microscope" was given by Mr R. J. Williams of BP Chemicals International Ltd.

The speaker dealt with the scanning electron microscope in an industrial research laboratory, comparing it with other forms of microscopy. Its different modes of operation were discussed, together with methods of specimen preparation. A number of slides were presented showing results obtained on a variety of paints and pigments, etc.

A.McW.

Water pollution control: objectives and methods

A meeting was held at the Royal Institution, Colquitt Street, Liverpool, on Wednesday 13 December 1972, when a lecture was given by Mr F. Buckley, Chief Water Quality Officer of the Lancashire River Authority.

To an audience of approximately 30 people, Mr Buckley spoke on "water pollution control: objectives and methods." With a staff of only 17 he was responsible for the quality of all rivers and lakes in area extending from Ainsdale/Ashton-in-Makerfield northward to Shap, including parts of Westmorland, Cumberland, and the West Riding, but not the River Irwell. He stated that 2p/person/day was spent on sewage disposal and 2p/house/day on water supply.

Midlands

Trent Valley Branch

Accelerated testing of durable coatings

On Thursday 9 November 1972, Members and guests, under the Chairmanship of Mr J. R. Bourne, attended the British Rail School of Transport, Derby, to hear a lecture on the above topic given by Mr E. Oakley, the senior technical officer responsible for co-ordinating accelerated and natural durability testing for Tioxide International Limited.

The correlation between different accelerated test methods and natural weathering had been discussed by many people for many years, but the state of the art was still such that much more work remained to be done in this field. The

lecture was a mass of facts and figures relating to various Acts of Parliament, and pollutant levels. A Royal Commission had stated that the polluters (who were often Local Authorities) should pay for the damage caused by their pollution. The maximum fine was £100 in 1951 and was now £400. There were 4,500 rivers in the UK; 22,000 miles non-tidal and 1,800 miles tidal; and 1,540 miles of canal.

The general impression gained was one of various governments since the early 1900s declaring their intentions to remedy the polluted state of many of Britain's rivers but then failing to take any action. This state of affairs might be altered when the Regional Water Authority (which would cover an area from Carlisle to Crewe and west of the Pennines) was responsible for the water, river and sewage facilities in the above area; this would take place on 1 April 1974.

A vote of thanks was proposed by Mr F. B. Windsor, and received with acclaim.

A.McW.

Design is where you find it

On Friday 12 January, 65 Members and guests attended the Manchester Literary and Philosophical Society, George Street, Manchester, to hear Mr E. Pond of WPM Ltd., give a talk entitled "Design is where you find it," discussing the psychology of colour and design.

Mr Pond based his lecture on a large number of very excellent slides. He commenced by emphasising the essential conforming nature of people, illustrating this item with pictures of bras, busts, boutiques and bikinis (and in certain cases the lack of same).

He went on to give some interesting connections between nature and designs, some accidental and some planned, and gave particular prominence to the Fibonacci series (a mathematical series whose first six numbers were 1, 1, 2, 3, 5, 8, 13 . . .), its occurrence in nature and its use in design, particularly the "golden rectangle."

Following this up with a review of fashions, particularly in furnishing, over the past 20 years, Mr Pond finished by pointing out that a good designer could produce a series of designs, one or two of which would prove very successful, although it was usually impossible to forecast which ones they would be.

After a lively discussion period, Mr Lyons proposed the vote of thanks which was received with very marked enthusiasm.

A.McW.

lecture presented the results of some work which had recently been completed in the laboratories of Tioxide International Limited.

The lecture was well illustrated by a series of slides. A full discussion period followed this very interesting and informative talk, and a vote of thanks was proposed by Mr J. R. Bourne.

It is hoped to publish this paper in full in a future edition of the *Journal*.

D.F.G.

Scottish

Eastern Branch

Leonardo Da Vinci and his times

The first meeting of the session was held in the Carlton Hotel, Edinburgh, on Wednesday 11 October, when Mr J. C. Patek of Croda Polymers Limited spoke on Leonardo Da Vinci and his times.

The question of why this particular artist was chosen by the Association from many equally famous to fulfil the role of Patron had long fascinated the speaker. He dealt with the history of the emblem, which incorporated the portrait of the artist and which had been used for the first time twenty-five years ago. The records of the Association spoke of Leonardo as the last of the mediaeval giants who managed to combine in his person the art, science and technology of his times, as few had done before and none could claim to be able to do today.

The speaker then proceeded to identify and place in perspective the so-called Renaissance period of European art and thought by reference to the broad stream of European art history, illustrating relevant highlights with slides. Then, by juxtaposing the works of other great artists of the late fifteenth and early sixteenth century, he picked out and pointed the way to the achievement of Leonardo as a man of extraordinary genius and energy and also, like the rest of us, prone to failures and disappointments.

Thames Valley

Colour measurement and control by fibre optics

At the Beech Tree in Beaconsfield on 26 October Mr D Irish of the Paint Research Association gave a lecture on the use of fibre optics in colorimetry. Mr Tatton was in the chair.

Stating that newer methods of colour measurement could make significant reductions in a manufacturer's costs by speeding output, e.g. at an output of 1.5 million gallons estimated savings of between £500-£10,000 per annum were realisable, Mr Irish discussed the basic theory and development of instrumental colour matching.

The possibility of colour measurement on the liquid sample during its manufacture, without the need to prepared dried films was made a reality by the coming of fibre optics. The arrangement developed by the PRA was described. On the whole, results had been very encouraging, although there have been problems with the thixotropic paints which could not be passed through a funnel.

In addition to their normal use for gloss and emulsion paints, fibre optics offered the possibility of viewing a sample at many different angles, and this was of particular value with gonio chromatic finishes. Further interesting applications for the equipment are foreseen.

Finally, Mr Irish took a glimpse into the future, when he envisaged a semi-automated paint factory in which a small process control computer was linked with colorimeters and metering pumps to feed proportionated amounts of tints etc.

After an interesting question time, Mr Bishop, of the Thames Valley Section Committee, gave a warm vote of thanks to the speaker.

R.E.G.

Car painting requirements of the future

The third meeting of the '72-'73 session was held on 23 November at the Beech Tree, Beaconsfield, with Mr W. Tatton in the chair. Mr W. Dabbs of the Austin/Morris

Finally, Mr Patek dealt in some detail with Leonardo's background and the vagaries of life in the city states of mediaeval Italy, each jealous of, and, more often than not, at war with, one another and simultaneously each offering its own interpretation of the enlightenment of a hunger for beauty and for the impossible.

J.H.S.

Marketing in the paper industry

The second ordinary meeting of the session took the form of a joint meeting with BPBMA North Eastern Discussion Group in the Tree Tops Hotel, Aberdeen, on 7 November, when Mr R. P. F. Shorter of Bowater UK Paper Co. Ltd. spoke on "Marketing in the paper industry."

Mr Shorter commenced his talk by defining industrial marketing. Here one was not concerned with final products but the purchase and sale of goods and services in industrial markets between organisational buyers and sellers. Marketing encompassed a wide spectrum, including market research and analysis, technical and new product development, market intelligence, industrial advertising, distribution, and selling. Each of these topics was dealt with in detail with reference to each other and to the paper industry.

J.H.S.

Body Division, BLMC, Cowley, gave a talk with the rather challenging title shown above.

He began by considering the defects of present day automobile paints, more particularly from the car manufacturer's viewpoint. First, some physical properties such as levelling (or flow), adhesion, stone chip resistance, solids content, and corrosion resistance were not at a sufficiently high performance level. Secondly, facilities required for their satisfactory application and cure were most expensive because of the need for a number of differing functional coats and long cycle or curing times. This required the provision of much valuable floor space, considerable track and oven maintenance, fire precautions, and facilities for pollutant disposal. Since mild steel was the dominant construction material for the body skin, paint systems also demanded expensive precleaning and phosphatising steps in the finishing cycle. Mr Dabbs forecast that mild steel would remain the material of construction for many years to come.

Mr Dabbs gave his views on some possible developments which could alleviate some, if not all, of the criticisms above. In the shorter term, paint systems on most of the cheaper mass production automobiles would be reduced to only two coatings on the exterior decorated surfaces. These would consist of a priming and a finishing colour coat, both applied by automatic means. The composition of the finishing coat was changing to yield higher solids, and hence the possibility of fewer coats. Already non-aqueous dispersion finishes (NAD) were in regular use and soon high molecular weight polymeric dispersions in water were likely to be available which would reduce pollutant problems. In previous attempts to employ water dispersed thermal curing resin systems, paint manufacturers had failed to overcome problems of excessive flow, which demanded longer and more accurately controlled ovens. However, Mr Dabbs expected developments in this area quite soon. Powder coatings represented yet a further stage in the cycle of potential development of improved finishing systems. He saw them as materials with minimal pollution, lower running costs requiring less floor space, but with known difficulties to be overcome, such as minimal

film weight, explosivity, higher curing temperatures, porosity in the film, absence of metallic effects, high cost of powder and high initial capital equipment cost were considerable.

As to the means for film curing, conventional ovens required much space and they suffered from delayed start-up times, though their running costs were low. More recent methods of curing included electron beam, ultra-violet and infra-red systems. All three held considerable attraction because of their improved curing speed, high efficiency, limited space, and minimal start-up time but, bearing in mind the complex shape, the frequent colour changes necessary, and the relatively short time-span of development of the newer methods, it was difficult to see any early dramatic development.

For the priming coat, the use of anodic electrodeposition was adopted by most car manufacturers. It was possible to foresee the use of an alternative cathodic deposition process which, while offering the advantage of iron-free films and no possibility of rupture of the phosphate coating, made for a

narrower choice of resin and higher capital installation costs. Another possibility lay in the application of what had become known as the "inverse process", provided three dissimilar coats were available. The conventional order of painting was reversed, some coats being applied first and then followed by the electropainting operation to those areas free of paint.

Diagrammatic illustrations of possible routes to improved systems and finishing techniques were used by Mr Dabbs and finally he suggested that, to overcome some of the difficulties raised in his talk, the most logical painting method in the near future was to adopt a two-coat system consisting of electropaint primer and high solids water-borne finish for the mass produced cheaper car. Higher quality cars would require a distinctly better standard of finish.

The discussion period was wide ranging and, after a vote of thanks proposed by Mr Bruce of Goodlass Wall Industries, the audience offered their appreciation in the usual manner.

R.E.G.

West Riding

Powder coatings

A meeting of the West Riding Section was held on 10 October 1972 at the Griffin Hotel, Leeds, at which Mr D. W. Brooker of Shell Research Limited presented a lecture on the above topic.

Mr Brooker discussed the problems of manufacture and traced the development of various methods culminating in that most commonly used at the present time, employing an extrusion technique. The development of this method enabled the use of more reactive curing agents and thus the production of fast-curing powders based on epoxy resins.

The market penetration of powders in various parts of the world was discussed and the wide variety of end-use applications was illustrated with colour slides.

Mr Brooker explained that the powder coating market was dominated by systems based on epoxy resins, which for many applications, were eminently suitable. However, they tended to lose gloss on exterior exposure and considerable development work was taking place on coatings based on acrylics, polyurethanes and polyesters. Work was also being carried out on manufacturing techniques, including a spray drying method of production which would overcome colour matching problems.

Mr Brooker finished with some thoughts on market development in the UK, which lagged behind the Continent, and suggested that UK paint manufacturers should take more interest in these developments or face the possibility of some of their traditional markets for liquid paints disappearing.

This most interesting and informative lecture provoked many questions and Mr T. Wood proposed a vote of thanks which was heartily endorsed by all present.

R.A.C.C.

Anaerobic acrylics

A meeting of the West Riding Section was held on Tuesday 14 November 1972 at the Griffin Hotel, Leeds. Dr W. A. Lees, of The Borden Chemical Co. (UK) Limited, gave a talk on the subject of anaerobic acrylics.

Dr Lees prefaced his talk by explaining that he intended to talk about two types of material, anaerobic acrylics and cyanoacrylates. The term anaerobic was not particularly accurate but arose from their ability to cure and change into

solids when the access of oxygen was cut off. This reaction was accelerated in the presence of metals. The cyanoacrylates polymerised in the presence of moisture and did not require a metal surface to act as a catalyst. They were sensitive to carbon dioxide, which inhibited polymerisation.

The anaerobic acrylics were used in a wide variety of engineering applications, e.g. permanent and semi-permanent assembly of components, sealing of threaded and non-threaded components and locking of threaded components. The cyanoacrylates were used typically for bonding plastic mouldings, where their solvent-free composition was of advantage.

These materials were of particular interest to the mechanical engineer. Cumbersome methods of assembly could often be avoided and it was possible to work to less close tolerances.

The cyanoacrylates were thermoplastic and extremely rigid with poor resistance to heat, solvents, chemicals etc., and must be used in a moderate environment.

Anaerobic acrylics, which were thermosetting, were diacrylic esters of tetraethylene glycol. A special plant was built to manufacture this material, constructed entirely of plastic and glass, dust free and with no depths greater than 2in otherwise self-polymerisation would occur very rapidly.

During question time Mrs Driver asked what temperatures the anaerobic acrylics could withstand. Dr Lees replied that they were generally satisfactory up to 120°C and could operate at up to 150°C dependent upon circumstances. Some grades could operate intermittently up to 200°C and permanently at up to 150°C. In reply to a question from Mr Bridgewood, Dr Lees said that the anaerobic acrylics were extremely resistant to most common solvents, but would swell in methylene chloride. The cyanoacrylates would dissolve in methylene chloride and dimethyl formamide and were generally less resistant.

The lecturer, when questioned about shelf-life, said that they do not have one which was determinable. Over a period of years, activity would slowly fall and reaction time become longer, but properties of the cured polymer appeared to be unimpaired.

Mr Cochrane proposed a vote of thanks for a most interesting lecture dealing with some unusual materials having a variety of useful applications.

R.A.C.C.



25th Technical Exhibition

21-24 May 1973
Empire Hall, Olympia, London

Over 100 stands

The Exhibition Committee has allocated space to AB Metal & Bergprodukt Ltd., Anderman & Co. Ltd., Instrumental Colour Systems Kingsley & Keith Ltd. and Strazdins Pty. Ltd., which means there will now be over 100 stands at the Exhibition, and 12 countries will be represented.

Official Guide

Copies of the *Official Guide* to the Exhibition will be despatched during March to all Members of the Association, and to those non-members who have made request for copies to be sent to them upon publication. They are also being sent to all Members of kindred associations on the Continent and, through the courtesy of the trade associations, to companies within the industries in the United Kingdom.

Information cards printed in six languages (English, French, German, Italian, Russian and Spanish) have been widely distributed on the Continent, both to individuals and to companies within the

industries. These are also being circulated in copies of other journals serving industries related to the surface coating industries.

Visits

Much interest has been aroused by the suggestion that Sections of the Association with the UK should arrange coach parties, on the lines of works visits, to the Exhibition, since this would afford a much cheaper form of travel for many of the younger Members. Non-members would be able to take advantage of this facility if there were places available, and those wishing to have further information on this facility should write to the Association's offices as soon as possible, in order that they may be put into contact with the Section Secretary nearest to their place of residence.

Hotel accommodation

Details of accommodation being offered by the Grand Metropolitan Hotels for those attending the Exhibition will be included in a leaflet to be inserted in each copy of the *Official Guide* being sent overseas.

Dinner

The *Official Guide* will also contain a leaflet giving details of the Dinner arranged at the Savoy Hotel on the first day of the Exhibition (21 May). This is a departure from the usual arrangements for the opening day, which will now commence at 12 noon, and is in response to the requests of many exhibitors who felt that they were not able to attend the Exhibition Luncheon in previous years as they had to complete preparation of their Exhibition Stands. Tickets are priced at £6.50 each and the function will commence at 7 p.m. for 7.30 p.m. The Guest of Honour, as announced in the February issue of the *Journal*, will be Lord Limerick, Parliamentary Under-Secretary of State for Trade to the Department of Trade and Industry, who will reply to the Address of Welcome by the President of the Association, Mr A. W. Blenkinsop.

Admission

Admission to the Exhibition is without charge and is unrestricted. The Exhibition takes place at the Empire Hall, Olympia, London, on the following days and times:—

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| Monday 21 May | 12.00-18.00 |
| Tuesday 22 May | 9.30-18.00 |
| Wednesday 23 May | 9.30-18.00 |
| Thursday 24 May | 9.30-18.00 |

Any company or non-member wishing to receive further information regarding this important display of raw materials, plant and equipment for the surface coating industries, in which over 100 companies will be taking part, from eleven overseas countries, should make application to the Association's offices for free copies of the *Official Guide*.

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

- COOKE, IAIN, BSc, Hoechst (UK) Ltd., 17 Blytheswood Square, Glasgow. (*Scottish*)
- HASTIE, COLIN, Berger Chemicals Ltd., Wellington Mills, Gateshead NE11 9HQ. (*Newcastle*)
- LYNN, STUART, GradRIC, Berger Chemicals Ltd., Wellington Mills, Dunston, Gateshead NE11 9HQ. (*Newcastle*)
- MIDCALF, CHRISTOPHER, BSc, PhD, ARIC, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)
- NAGELL, PETER A. G., c/o Columbian Carbon Deutschland GmbH, 2103 Hamburg 95, Antwerpenstrasse 1, Germany. (*General Overseas*)
- SANDS, IAN MURDOCH, Ciba-Geigy (UK) Ltd., Pigments Division, Hawkhead Road, Paisley. (*Scottish*)
- STIRLING, JOHN ANDREW, LRIC, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)
- TURNER, ALISON, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)

YEOW, KOK PENG, BSc, Kansai United Paints Pte. Ltd., 12 Jalan Lembah Kallang, Singapore 12. (*General Overseas*)

Associate Members

CONNELL, WILLIAM JOSEPH, Strathclyde Chemical Co., Beith Road, Johnstone, Renfrewshire. (*Scottish*)

Registered Students

- BLANEY, MICHAEL, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)
- FRASER, SANDRA ELIZABETH, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)
- GILES, HARRY FRASER, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)
- SCHONING, HUBERT JAMES, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)
- TAYLOR, JOHN, Ciba-Geigy (UK) Ltd., Hawkhead Road, Paisley. (*Scottish*)

OCCA Biennial Conference

towards 2000

eastbourne 19-23 June 1973

Conference Brochure

Full details of the Association's Conference were circulated to all Members of the Association and non-members who had made requests to receive these details early in January.

The full programme and summaries of the papers appeared in the December issue of the *Journal*, and biographies of the lecturers appeared in the January issue.

A considerable number of applications has now been received and readers are reminded that the closing date for registrations will be **31 March 1973**. Later applications will still be considered after that date, but those applying from United Kingdom addresses must add Value Added Tax at the standard rate to their remittances.

Preprints

Preprints of the papers are now being prepared, and it is expected that these, together with details of accommodation, badges, programmes, etc., will be sent to those who have registered in May. It is a feature of the Association's Conferences that preprints are sent well in advance of the function, in order to enable delegates to read them before the conference, at which the lecturers illustrate their topics

but do not read the papers in their entirety. This allows for a much longer discussion period than at many other conferences, and has proved of immense value to those attending previous Association Conferences.

Registration fees

The Council has fixed the registration fees for the Eastbourne Conference at £30 for Members, £10 for Wives, and £50 for non-Members. Registered Students of the Association and Retired Members are allowed to register at 50 per cent of the Member's rate.

The Chemical and Allied Products Industry Training Board will consider the payment of grant, to firms within scope, for attendance at the Conference. Payment is subject to the content of the Conference being relevant to the needs of the sponsoring company and also to the development of the individual attending.

Dates and times of sessions

The conference assembles on the evening of Tuesday 19 June, when, in accordance with its usual tradition of hospitality, the Association will hold a reception for all those attending from Overseas, which will be followed by a meeting of the lecturers

and the chairmen of sessions. The Conference sessions will be held on Wednesday and Thursday 20-21 June, morning (9.30-12.30) and afternoon (2.30-4.30), and the morning of Friday 22 June (9.30-12.30). The Association's Annual General Meeting will take place at 2.15 p.m. on the afternoon of Friday 22 June, and this will be followed by three Workshop Sessions, which have proved very popular in the past. The subjects this year will be: "Sales forecasting", "The changing influence of the do-it-yourself market", and "The measurement of research output". The number attending each Workshop Session is strictly limited and places will be allocated in order of receipt. Where the total places for a session have been filled, later applicants will be informed and, if possible, offered an alternative session.

Receptions and Dinner

There will be an informal reception for all delegates at lunchtime on Wednesday and a Civic Reception will be held, at which delegates will be welcomed by the Mayor and Mayoress of Eastbourne, on the evening of the same day. The Association's Dinner and Dance will be the closing function of the Conference on the Friday evening. Other social activities, such as coach tours, are being arranged for the benefit of ladies attending the conference.

Optional Professional Grade for Ordinary Members

The Professional Grade Committee has issued a further list of admissions to the Professional Grade. The full list showing the Sections to which the Members are attached is given below.

Fellow in the Technology of Surface Coatings (FTSC)

Balbi, Giorgio (*General Overseas—Italy*)
Carter, Eric Victor (*Midlands*)
Chessman, Clifford Reginald
(*South African*)
Cutter, John Outram (*London*)
Grover, Donald Henry (*London*)
Kut, Siegmund (*London*)
Polaine, Sydney Alan (*London*)
Roe, David Edwin (*London*)
Rouse, Robert Earnshaw (*South African*)
Slade, Harold Aitken (*West Riding*)
Walker, Alan Gordon (*Thames Valley*)
Woodbridge, Richard John (*Bristol*)

Transferred from Associate to Fellow:

Bishop, Eric Harold Abbott (*London*)
Tozer, Edwin John
(*General Overseas—Argentina*)

Associate in the Technology of Surface Coatings (ATSC)

Bloomfield, Kenneth Vincent (*London*)
Ernst, Joel (*London*)
Finlay, Cecil Newton (*Newcastle*)
Honiball, Alan Edward (*Manchester*)
Lipscombe, Charles George (*Auckland*)
Low, Charles (*South African*)
Lunt, Walter Richard (*West Riding*)

Macdonald, Arthur Gillings (*Newcastle*)
McCapra, Ronald (*Auckland*)
Marsden, Chris Eyre (*Manchester*)
Martin, Christian Pierre
(*General Overseas—France*)
Moore, William Alexander (*Auckland*)
Proudley, Philip Miles (*London*)
Quorn, Peter James (*South African*)
Smith, Harry Bertram (*London*)
Stephenson, Robert Perry (*Auckland*)
Tillyer, Richard Brian (*Wellington*)
Venus, Norman (*South African*)
Whalley, James
(*General Overseas—Kenya*)
Wood, George (*London*)

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.

Thursday 1 March

Newcastle Section: "The evolution of alkyd resins" by Mr T. I. Price of BP Chemicals, to be held at the Royal Turks Head Hotel, Grey Street, Newcastle, at 6.30 p.m.

Monday 5 March

Hull Section: Ladies Evening, "Contemporary textiles" by Professor C. S. Whewell, Head of the Department of Textile Industries, The University of Leeds, to be held at the Queens Hotel, George Street, Hull, at 7.00 p.m.

Wednesday 7 March

London Section: "Some aspects of colorimetry, particularly fibre optics" by Mr D. Irish of the Paint Research Association, to be held at the South Bank Polytechnic, at 7.00 p.m.

London Section—Southern Branch: Student symposium at the Banqueting Hall, Guildhall, Winchester, at 11 a.m.

Manchester Section: "Recent trends in the development of organic pigments" by Dr E. R. Inman of Ciba-Geigy (UK) Ltd., to be held at the Royal Institution, Liverpool, at 6.30 p.m.

Newcastle Section—Student Group: "The use of water-borne paints" by Mr B. Ridley of the International Paint Co. Ltd., to be held at Newcastle Polytechnic, Ellison Place, Newcastle, at 3.00 p.m.

Thursday 8 March

Midlands Section—Trent Valley Branch: "The origin and extraction of natural colours and minerals" by Mr E. Hare of Via Gellia Colour Co. Ltd., to be held at the British Rail School of Transport, London Road, Derby, at 7.00 p.m.

Scottish Section: "Research and innovation at the Paint Research Association" by Dr S. H. Bell of the Paint Research Association, to be held at St. Enoch Hotel, Glasgow, at 6.00 p.m.

Thames Valley Section—Student Group: "Glass reinforced plastics," speaker to be arranged. To be held at the Main Lecture Theatre, Slough College, at 4.00 p.m.

Saturday 10 March

Scottish Section—Student Group: Short talk and film on pollution by Mr Alwyn Jones of the Scottish Council Development and Industry, to be held at the St. Enoch Hotel, Glasgow, at 10.00 a.m. Followed by AGM.

Tuesday 13 March

London Section—Southern Branch: "Medicines for coatings" by a speaker from Byk-Mallinckrodt, to be held at the Pendragon Hotel, Southsea, at 7.00 p.m.

West Riding Section: "Microbiology" by Mr W. R. Springle of the Paint Research Association, to be held at the Griffin Hotel, Leeds, at 7.30 p.m.

Wednesday 14 March

Manchester Section—Student Group: "Pigment dispersion" by Mr G. R. Robson of ICI Ltd., Organics Division, to be held at the Manchester Literary and Philosophical Society at 4.30 p.m.

Friday 16 March

Irish Section: "Modern developments in packaging" by Mr E. G. O. Ridgwell of the Irish Packaging Institute, to be held at the Clarence Hotel, Dublin, at 8.00 p.m.

Midlands Section: Annual J. Newton Friend Lecture at 7.30 p.m. "Some great English potters" by Mr C. E. L. Franklin of Royal Doulton Tableware Ltd., to be held at the Birmingham Chamber of Commerce and Industry, 75 Harborne Road, Birmingham.

Wednesday 21 March

Scottish Section—Eastern Branch: AGM, followed by films (ICI Ltd.), to be held at the Carlton Hotel, North Bridge, Edinburgh, at 7.30 p.m.

Thursday 22 March

Thames Valley Section: "Recent developments in the printing and decoration of metal containers" by Mr A. D. Lott of the Metal Box Co. Ltd., to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks, at 7.00 p.m. Supper will be provided if ordered in advance from the Secretary.

Friday 30 March

Bristol Section: Informal debate, "The customer is always right," to be held at the Royal Hotel, Bristol, at 7.15 p.m.

Monday 2 April

Hull Section: "The control of polymer lifetime under environmental condition" by Professor G. Scott. The E. H. Bullock lecture theatre, College of Technology, Hull, at 7.00 p.m.

Wednesday 4 April-Thursday 5 April

London Section: Joint Symposium and exhibition with the Institute of Metal Finishing, Organic Finishing Group. "Automotive and other industrial

finishing—what is the future?", to be held at the University of Warwick, Coventry. Registration and details from Mr D. G. Gray, 36 Constance Road, Whitton, Twickenham, Middx. TW2 7HT.

Thursday 5 April

Midlands Section—Trent Valley Branch: AGM, followed by "Steel in fashion" by Mrs Coleran of the British Steel Corp., to be held at the British Rail School of Transport, London Road, Derby, at 7.00 p.m.

Newcastle Section: AGM (venue and time to be arranged).

Friday 6 April

Bristol Section: Annual Dinner Dance, to be held at the Mayfair Suite, Bristol Entertainments Centre.

Tuesday 10 April

West Riding Section: 21st AGM, to be held at the Griffin Hotel, Leeds, at 7.30 p.m.

Thursday 12 April

Thames Valley Section: AGM, followed by a talk by a speaker from HM Customs and Excise, to be held at the Beech Tree Hotel, Maxwell Road, Beaconsfield, Bucks, at 7.00 p.m.

Friday 13 April

Manchester Section: AGM, to be held at the Lancashire County Cricket Club, Old Trafford, Manchester, at 6.30 p.m. (provisional).

Midlands Section: AGM, to be held at the Birmingham Chamber of Commerce and Industry, 75 Harborne Road, Birmingham, at 6.30 p.m.

Wednesday 18 April

Scottish Section—Eastern Branch: "Restoration and conservation" by Mr R. L. Snowdon of the Restoration Centre, Department of the Environment, Edinburgh. If possible, this meeting will be held at a site where work is under way.

Friday 20 April

Irish Section: AGM, followed by an open forum discussion. To be held at the Clarence Hotel, Dublin, at 8.00 p.m.

Friday 27 April

Bristol Section: AGM, to be held at the Royal Hotel, Bristol, at 7.15 p.m.

West Riding Section: A dinner at the Bodington Hall to mark the 21st Anniversary of the Section.

Midlands Section

Trent Valley Branch

Buffet Supper Dance

A most successful supper dance was held on Friday 8 December at the Cross Keys, Turnditch, Derbyshire.

The event was extremely well supported, there being approximately 60 Members and guests present. Among the guests were Mr A. S. Gay, Chairman of the Midlands Section, and his wife and the Immediate Past Chairman of the Section, Mr H. Griffiths, and wife.

As is usual with this event, informality was the order of the day and there is no doubt that this subscribed in no small measure to the overall success of the evening.

An exceptional buffet was provided with all manner of mouth-watering delicacies to tempt the palate.

Although perhaps a little congested at times, the dance floor was nevertheless the scene of great activity, music being provided by the Roy Stone Trio.

D.F.G.

Course for Licentiatehip of the Association

The Polytechnic of the South Bank currently offers a one year part-time course leading to the Licentiatehip of the Royal Institute of Chemistry in Surface Coatings Technology. Arrangements have been made in conjunction with this course to enable students to satisfy the requirements for the Licentiatehip grade of the Oil and Colour Chemists' Association. Further details may be obtained from R. J. Ceresa, Department of Chemistry and Polymer Technology, BSc, FTSC, FRIC, FIRI, FPI.

News of Members

Mr R. N. Rea, an Associate Member attached to the Irish Section, has been promoted to sales manager of the Dyestuffs department of Hoechst Ireland Limited.

The Swiss society, Schweizerische Vereinigung der Lackund Farbenchemiker, has offered Corresponding Membership to Mr A. R. H. Tawn, the Honorary Research and Development Officer of the Association. Mr Tawn has accepted the SVLFC's invitation.

Mr J. D. Bushell, an Ordinary Member attached to the London Section, has recently transferred from Shell Chemicals UK Ltd. to take up an assignment in the base chemicals marketing division of Shell International Chemical Company.

We regret that, in the December 1972 issue, Mr D. G. Dowling was incorrectly reported as having been appointed Technical Director of Humbrol Ltd. Mr Dowling was in fact appointed Chief Chemist of Humbrol. The company's Technical Director is Mr S. Sharp, an Ordinary Member attached to the Hull Section.

Mr A. L. Pyett, an Associate Member attached to the Manchester Section, has recently been appointed to the sales staff of the general industries division of Allied Colloids Ltd.

Corrigenda

Weathering of paint films

In the paper "Weathering of paint films" by Hoffmann and Saracz which appeared in the December 1972 issue of JOCCA, the second line of *Discussion and conclusions* should read "anatase titanium dioxide at 40°C and 70 per cent relative humidity."

Some aspects of drying oils technology

The following errors have occurred in the paper "Some aspects of drying oils technology" by G. H. Hutchinson, which appeared in the January 1973 issue:

Page 48, line 10 should read "elaeostearate indicates that the fatty acid chains are not bridged by," and line 20 should read "the fact that a true polymerisation in respect of fatty acid radicals."

The legend for Fig. 1 on page 46 should include "Oxidation period five hours at 35°C; developing solvent, petroleum ether. (Modification of the method by Poxon D. W., JOCCA, 1958, 41, 291)".

The legend for Fig. 3(a) on page 47 should include "Developing solvent coloured violet with cobalt acetate drier. After oxidation and development of chromatograms, oil spots and bands are green in colour."

The legend for Fig. 3 on page 47 should include "three hours at 50°C; drier content 0.03 per cent cobalt (as metal); solvent petroleum ether."

Mr Hutchinson's present address is Croda Polymers Ltd., Ink Division, 170 Glasgow Road, Edinburgh EH12 9BE.



Oil and Colour Chemists' Association

President: A. W. BLENKINSOP, F.T.S.C.

General

The Oil and Colour Chemists' Association was formed in 1918, to cover paint, printing inks, pigments, varnishes, drying and essential oils, resins, lacquers, soaps, linoleum and treated fabrics, and the plant, apparatus and raw materials useful in their manufacture. In 1924 it absorbed the Paint and Varnish Society. The stated purpose of the Association is to promote by discussion and scientific investigation the technology of the industries concerned with the above-mentioned products, and to afford Members opportunity for the interchange of ideas. This is achieved by the regular holding of ordinary meetings at which papers are presented, and the organisation of annual technical exhibitions, biennial conferences, educational activities and practical co-operative experimental work. Details of these activities are given in the *Journal of the Oil and Colour Chemists' Association*, which is published monthly, and whose pages are open to receive communications and other pronouncements on scientific and technical matters affecting the Members of the Association and the industries concerned. The Association's meetings also afford opportunities for Members to meet informally and socially.

Sections

There are Sections of OCCA in Auckland, Bristol, Hull, Ireland, London (with a Southern Branch), Manchester, the Midlands (with a Trent Valley Branch), Newcastle upon Tyne, Scotland (with an Eastern Branch), South Africa (with Branches in the Cape, Transvaal and Natal), Thames Valley, Wellington, and the West Riding, and these are responsible for the conduct of their own local affairs. There is also a General Overseas Section. There is also a close relationship between the Association, the Federation of Societies for Paint Technology in the United States, and the Fédération d'Associations des Techniciens de l'Industrie des Peintures, Vernis, Emaux et Encres d'Imprimerie de l'Europe Continentale (FATIPEC). The Association also maintains cordial relations with the Scandinavian Federation of Paint and Varnish Technicians (SLF).

The five Sections previously maintained by the Association in Australia formed (I.I.68) the Oil and Colour Chemists' Association Australia, having the same aims and activities as, and working in close liaison with, the parent body.

Membership

Ordinary Membership is granted to scientifically trained persons, and Associate Membership to others interested in the industries covered. An optional Professional Grade, conferring designatory letters, is open to Ordinary Members. Student membership is open without restriction to persons under the age of 21 and to those up to 25 who are following a course of technical study. The annual subscription in each case is £5.25, except for registered Students, whose subscription is £1.05. An entrance fee of 50p is payable by all members. Applications for membership are invited from suitably qualified persons who are engaged or otherwise interested in the industries noted above. Applications, which should be supported by two Members of the Association (one of whom must be an Ordinary Member), should be forwarded to the Director & Secretary at the address given below. Application forms and full details of membership may be obtained from the offices of the Association.

Professional Grade

The Association recently introduced (1971) an *optional* professional grade for its Ordinary Members, giving the designatory letters FTSC (Fellow in the Technology of Surface Coatings), ATSC (Associate in the Technology of Surface Coatings), and LTSC (Licentiate in the Technology of Surface Coatings). Full details are available upon request from the Association. Where Membership has lapsed, previous periods of Ordinary Membership count towards the total required under the regulations, as set out in the January 1973 issue of *JOCCA*.

Exhibitions

A technical exhibition is held annually at Olympia; Members are sent copies of the *Official Guide* several weeks in advance, in order to plan their itineraries. Non-members should apply, in writing, to the Director and Secretary for copies of the *Official Guide*.

Conferences and Symposia

The Association organises large, biennial technical conferences, the papers for which (together with discussions) are published in the *Journal*. Sections of the Association, in the UK and abroad, hold symposia and these, too, are reported in *JOCCA*.

Publications

Journal of the Oil and Colour Chemists' Association (JOCCA) is published monthly and includes a yearly index in the December issue. The subscription rate to non-members is £10.00 p.a. in the United Kingdom and £12.00 p.a. (\$31) abroad (post free; payable in advance). Single copies may be purchased for £1.00 (£1.25 abroad).

Introduction to Paint Technology (Second Edition with additional chapter). With illustrations, 187 pages and index £1.00 (including postage).

Paint Technology Manuals (Parts 1, 2, 4, 5 at present out of print).

- Part 1 "Non-convertible Coatings"
- Part 2 "Solvents, Oils, Resins and Driers"
- Part 3 "Convertible Coatings," Second Edition, pp. 350, £2.80
- Part 4 "The Application of Surface Coatings"
- Part 5 "The Testing of Paints"
- Part 6 "Pigments, Dyestuffs and Lakes, pp. 340, £2.00
- Part 7 "Works Practice," pp. 218, £2.40

Director & Secretary: R. H. Hamblin, MA, FCIS,
Priory House, 967 Harrow Road, Wembley, Middlesex,
England HA0 2SF.

Tel. 01-908 1086; Telex 922670 (OCCA WEMBLEY)

Paint Technology Manuals



Part Seven: Works Practice

The seventh volume of the Association's series of Paint Technology Manuals, entitled "Works Practice," has now been collated, bound and is now offered for sale.

The articles which have appeared under Student Reviews each month have been so designed that the bound volume will form a complete, coherent book with full continuity. Apart from the necessary typographical changes in maintaining consistency in the *Journal*, "Works Practice" will be exactly the same as those parts of the Paint Technology Manuals already published by Chapman & Hall Ltd.

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 - (c) Processes without premixing*
 - (d) Layouts, maintenance, filling and warehouse*
- Chapter 5—Factory organisation and personnel*
- Chapter 6—Legal requirements and regulations*
- Chapter 7—Factory hazards and safety precautions*

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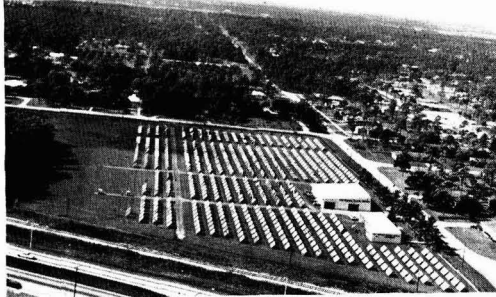
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SITUATIONS VACANT

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