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ABSORPTION OF OILS INTO POROUS COATINGS

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Synopsis An approach to the basic processes of ink absorption into paper coatings is made by considering the way in which oil is absorbed into kaolin-based layers (50 microns thick), formed on flexible polyester substrates. Oil layers are applied to these layers by use of an IGT printability tester. The change in gloss level with time after application of the oil film is used as a method for determining the time taken for an oil film of known thickness to be absorbed.

The capillary penetration equation of Washburn is used as a basis to explore the relationship between the physical properties of the oils and the structure of the coatings.

The basic structure of one coating is also determined by the method of mercury porosimetry and is related to the oil absorption rate by use of the equation of Millington & Quirk for the permeability of the layer.

Introduction

PAPER that is coated with a thin layer of a finely divided mineral such as china clay together with an adhesive has improved printability. Although the coating thickness may have a mean value of only 10 microns or less, it will nevertheless produce an appreciably smoother and less absorbent surface. The pore size of the coating layer and its void volume are much smaller than that of the base paper; consequently, the liquid phase from an ink layer is absorbed more slowly into its surface. This results in a lower ink demand together with a better print quality and gloss as a greater fraction of the polymerised oils and resins from the ink remain on the surface of the coating.

Recent work on oil absorption into coated papers by Hill & Mill(1) and McConnell(2) has been reported. In both cases, an estimation is made of the absorption time of a thin oil film applied to a coated paper. The data obtained allows a qualitative comparison to be made between the oil absorption properties of coated papers in a more realistic way than with the more conventional method of 'K and N' ink absorption, which, for lightweight coatings, can saturate the coating layer during an extended (2 min) contact

Under the chairmanship of Prof. M. F. Judt period. Although oils, rather than pigmented inks, were used in these methods,(1, 2) the results are obviously relevant to the initial absorption characteristics of the coating layer at a stage before any ink pigment layer is built up on the surface (in the manner of a filter cake) and capable of further slowing down the liquid phase movement.

In the work reported here, an attempt has been made to find a quantitative explanation for the rate of absorption of an oil into a coating layer in terms of the physical characteristics of the oil and the structure of the coating layer.

Preparation of the coating layers

COATINGS were prepared from commercially available paper coating grade clays together with an adhesive system consisting of equal parts by weight of an oxidised potato starch and a styrene butadiene copolymer. For this investigation, the coatings produced should have a uniform thickness great enough to enable reasonably thick oil films ($\leqslant 8$ microns) to be absorbed without saturating the layer. These were produced by applying the coating suspension to a flat, flexible substrate of polyester film by hand with the aid of a Bird applicator bar. The coatings were air dried and conditioned (65 per cent rh, 20° C) before being used for oil absorption testing. The thickness of the dried coating layer was determined by micrometer to be 50 microns. Additionally, the coating layers may be calendered, thereby producing another variable open to investigation.

Method of determining the oil absorption rate

OIL films of known mean thickness may be applied to surfaces by use of the IGT AC2 printability tester. By prior determination of the weight of oil applied to a printing disc, the fraction subsequently transferred to the experimental surface may be measured and the mean thickness of the film formed may be calculated from the density of the oil and the area laid down.

As the oil film is absorbed into the coating surface, the appearance of the surface changes; it passes progressively from that of the intact oil film, which has a high gloss, to the lower gloss of the original surface after complete absorption of the oil film.

This appearance change has formed the basis for estimating the absorption time for oils into paper (coated and uncoated) by a number of workers in the past. Thus, Larocque(3) was the first to use photoelectric measurements of the variation of surface reflectance with time to determine an absorption rate. Vallandigham(4) also used oil absorption rates, determined by a reflectance method, to correlate the printing properties of paper. Subsequently, Bowles & Silcox(5) used the method to determine absorption as a function of ink film thickness, liquid phase viscosity and the pigment volume content of a simulated ink. They used a number of different surfaces in their work, including coated and supercalendered papers. More recently, the method has been applied by Tollenaar(6) to derive a pore size distribution for a paper substrate and Hill & Mill(1) have used the method for assessing the comparative performance of coated papers.

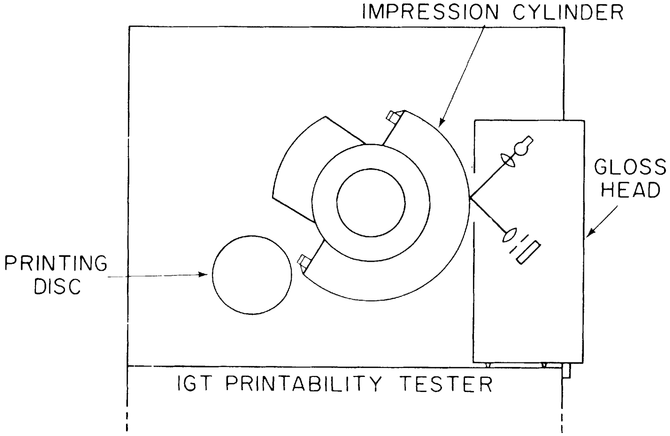


Fig. 1—Gloss head in position of IGT printability tester

Following Hill & Mill, a gloss measuring head was built that could be easily attached to the IGT AC2 printability tester. The schematic arrangement is shown in Fig. 1. The gloss head is mounted in a semi-lighttight box, which is positioned on the platform of the printability tester by means of locating pins so that it is able to monitor the light that is specularly reflected (at 40° from the normal) from a coating attached to the impression cylinder. The light source is a small 12 W tungsten filament bulb operating from a 6 V transformer supply. A selenium barrier layer photocell monitors the light specularly reflected from the surface and the output is fed through a suitable resistance to give a linear output on a 1 mV chart recorder. The printability tester was set up to give a printing pressure of 20 kgf on a 2 cm wide printing disc and with a linear impression cylinder speed of 20 cm/s. There was thus a delay of about 1 s between application of the oil to the coating and the start of reflectance monitoring. This is negligible in comparison with the absorption times encountered in this work.

Interpretation of the reflectance data

A TYPICAL example of the reflectance/time characteristics of an oil (a mineral oil with viscosity 17 poise) applied to a coating (made from clay with 80 per cent by weight less than 2 microns equivalent spherical diameter together with 15 pph adhesive) is shown in Fig. 2. In region 1, there is an initial rise in the reflectance, which is a measure of the time taken by the oil film to level out to a plane surface after the film has been split between the coating and the applicator surface. In region 2, the reflectance level remains constant until region 3 is reached, when the reflectance falls to a value equal to that of the coating before application of the oil.

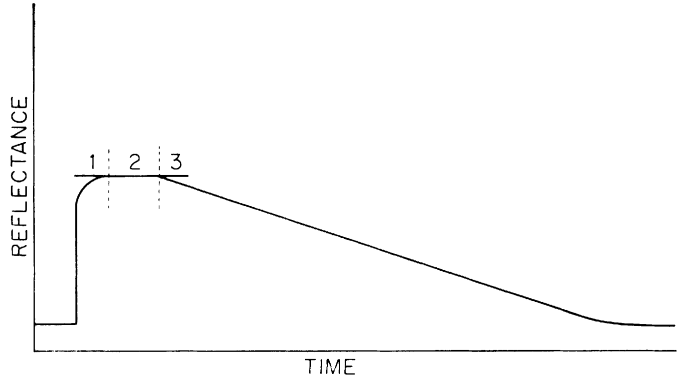


Fig. 2—Change in reflectance with time as an oil film penetrates a coating

The problem is essentially to determine, from the fall in reflectance with time, at what point an oil film of known thickness has penetrated into the coating. Hill & Mill chose to consider the absorption end point to be when the reflectance had fallen by 75 per cent from the maximum value. This is not unreasonable when considering that they applied the method to coated papers that have a point to point variation in absorption, which makes accuracy difficult to attain. In the case of the coatings considered here, however, the surfaces are uniform and it is therefore possible to consider a more accurate determination of the absorption end point.

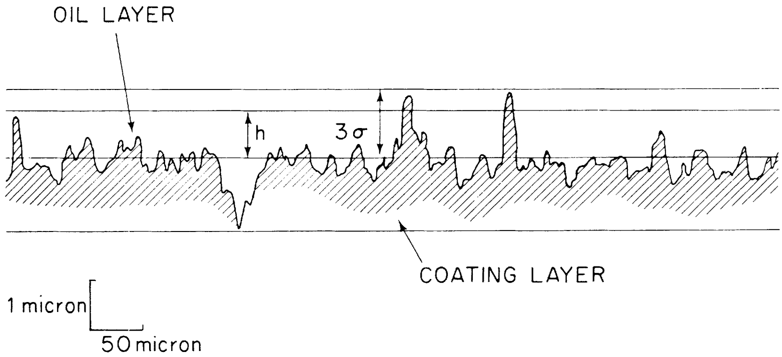


Fig. 3—Surface profile of a coating

It has recently been shown(7) that the light specularly reflected from coating surfaces such as those under consideration here can be calculated in terms of the microstructure of the surface; the microstructure can be characterised by a Gaussian distribution of peaks about the mean plane of the surface, where the standard deviation (σ) has a typical value of the order of 0·1–0·2 microns. Such a profile is illustrated in Fig. 3 and shows a Talystep (Rank Precision Industries Ltd.) trace for an uncalendered coating. Consider the application of a 1 micron mean thickness oil layer to such a surface. At the instant of application and assuming the oil is in intimate contact with all points on the surface, the mean lower surface of the oil film coincides with the mean plane of the coating. If the coating is homogeneous (with no pinholes, for example), we may assume that the oil layer advances uniformly into the coating until a time is reached when the upper surface of the oil layer lies in the mean plane of the coating surface. This will be the point at which the absorption of the oil layer will be considered to terminate for the purpose of calculating absorption times. The reflectance from the oil layer will thus remain constant until the upper surface of the layer comes into contact with the upper extremities of the coating lying approximately 3σ above the mean plane of the coating surface. The gloss will continue to drop with further penetration until the oil surface coincides completely with that of the coating.

It can be shown that, as a plane surface (the upper surface of the oil layer) moves down through the coating, the area of the plane that is intersected by surface asperities follows a Gaussian distribution.

Thus, for example, 2·3 per cent of the plane area will be intersected by surface asperities from the coating when the moving plane is at a distance of 2σ above the mean plane of the coating. It is possible in this way to calculate the fractional area of oil film remaining as a plane surface as it advances into the coating. (It is necessary to assume that localised curvature of the oil film arising from the formation of meniscii can be ignored.) In addition, the rate at which the oil surface advances into the surface structure can be calculated from the capillary equations introduced in a subsequent section. Thus, the vertical displacement of the plane is proportional to the square root of the elapsed time. In Fig. 3, h is the distance of the oil surface above the mean plane at a time t after the reflectance has started to drop and we may write—

$$(3\sigma - h)^2 = kt$$

The reflectance of the surface (R) at this point may also be written as—

$$R = R_o \cdot a + R_o(1 - a)$$

where R_c is the reflectance of the complete oil layer and R_o is the original reflectance of the coating and a is the fractional area of the oil film remaining as a plane. Hence, $a = (R - R_o)/(R_c - R_o)$.

A value for a may be calculated as a function of h from statistical tables,(8) then as a function of time from equation (*1*). A comparison may then be made between the normalised fall in reflectance found from theory and experiment. An example is shown in Fig. 4. The similarity between the shape of the curves allows us to conclude that the time at which the oil film reaches the mean plane of the coating is when the reflectance is halfway between R_o and R_c.

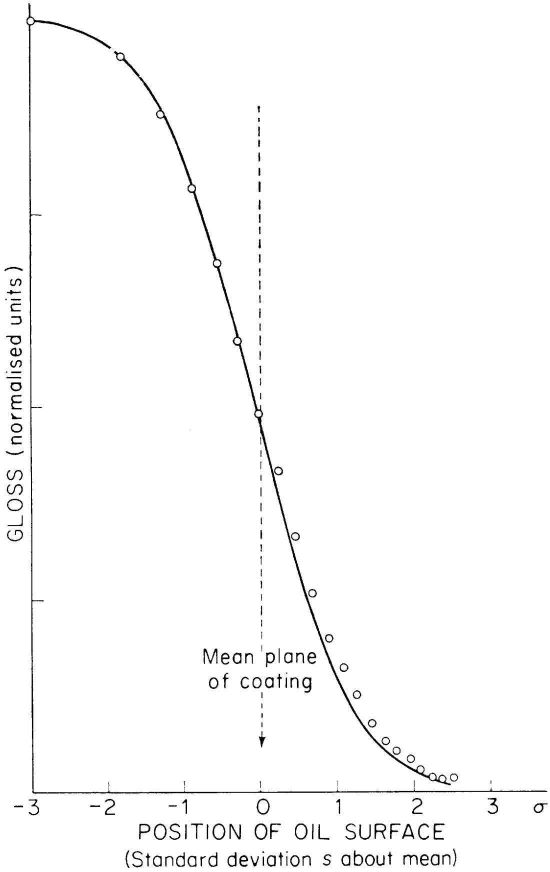


Fig. 4—Comparison of calculated and experimental reflectance curves Calculated for a surface with a normal roughness distribution ○ Measured values for mineral oil film passing through a coating surface

The penetration equation

CONSIDER an oil film of mean thickness h laid down on a coating with a porosity ϵ, where ϵ is the ratio of pore volume to total apparent volume. Then the oil will have to penetrate, on average, to a depth of h/ϵ before its upper surface is coincident with the mean plane of the surface.

The path followed by an elemental volume of the oil will be tortuous. Let this be described by a tortuosity factor τ, which is the ratio of the real distance travelled by an element to the distance h/ϵ. Hence, the total real distance travelled by an elemental volume of oil will be $h \cdot \tau/\epsilon$.

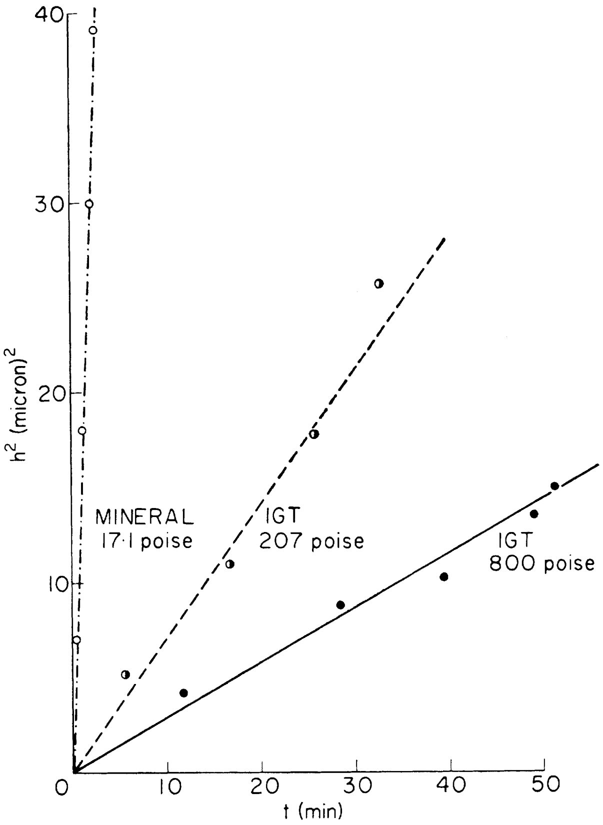


Fig. 5—Penetration time t as a function of square of the oil film thickness h for three oils

Let us characterise the pore structure of the coating by an effective pore radius r and assume that the coating behaves as a collection of parallel capillaries with this radius and with a length of $h\cdot\tau/\epsilon$. We can then apply the Washburn equation and relate the time (t) taken to penetrate the capillaries in order to drain the oil into the coating by the equation—

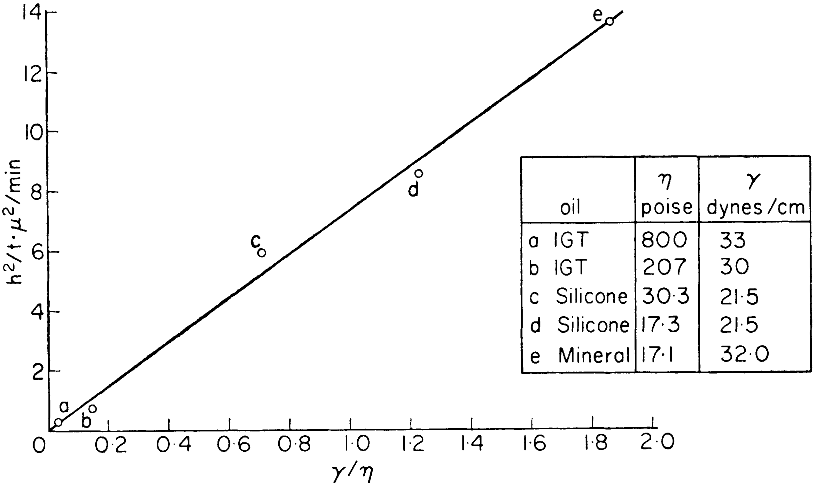
$$\left(\frac{h\cdot\tau}{\varepsilon}\right)^2 = \frac{\gamma\cdot\cos\theta}{2\eta}\cdot r\cdot t$$

where γ is the surface tension of the oil, η is the viscosity of the oil and θ is the contact angle between the oil and the coating material. It is usually justifiable to assume perfect wetting so that $\cos\theta\rightarrow 1$. We may then write—

$$h^2 = \left(\frac{\gamma}{2\eta}\right)\left(\frac{\varepsilon}{\tau}\right)^2\cdot r\cdot t\, . \qquad . \quad . \quad . \quad . \quad . \qquad (2)$$

If we then measure the time t taken for an oil layer of thickness h to be absorbed into the coating, we should find a linear relationship between h^2 and t.

By use of a coating (uncalendered) containing 15 pph adhesive, absorption times were measured with the reflectance technique described above for oil films varying in mean thickness 2–6 microns. Three different oils were used, covering a wide range in viscosity. The results obtained are illustrated in Fig. 5, in which the absorption times are plotted against the square of the oil film thickness. The linear relationship obtained illustrated the applicability of the equation and the homogeneity of the structure of the coating. A further examination of the variables in equation (*2*) indicates that the slope of the straight lines obtained in Fig. 5 (h^2/t) should be proportional to the ratio γ/η



oil	η poise	γ dynes/cm
a IGT	800	33
b IGT	207	30
c Silicone	30·3	21·5
d Silicone	17·3	21·5
e Mineral	17·1	32·0

Fig. 6—The term h^2/t as a function of γ/η for 5 oils

for a particular coating. This relationship was examined for five oils whose viscosity and surface tension were measured in the laboratory. Two silicone oils were included in this examination, as they have a significantly lower surface tension (21·5 dynes/cm) compared with the other oils ($\approx$32 dynes/cm) already used in Fig. 5. The results are illustrated in Fig. 6 and again show the linear relationship predicted by equation (*2*).

It is possible to alter the structure of a coating by changing the quantity of adhesive in the system. This can affect many of the properties of the coating; adhesive addition generally results in lower gloss, lower brightness and lower absorption. The latter was investigated at three levels of adhesive addition (5, 10 and 15 pph) for the same clay by measuring absorption times on each coating for the same range of oil thicknesses. The results are illustrated in Fig. 7 and show that a considerable increase in the oil absorption rate is produced at the lower adhesive levels.

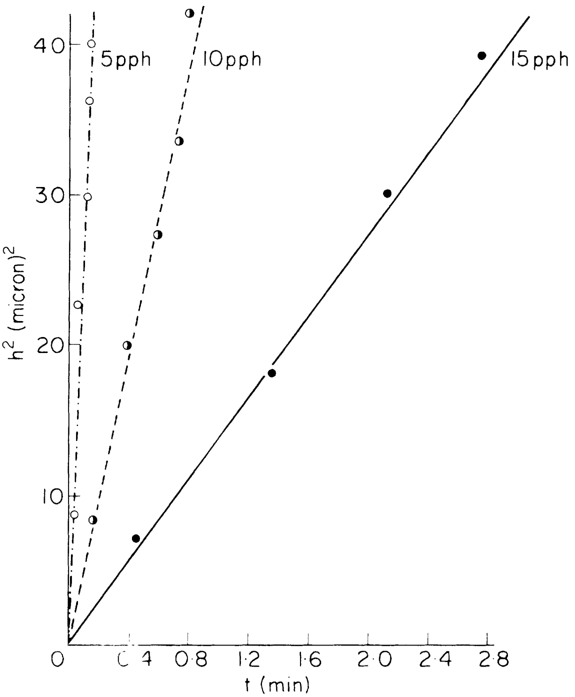


Fig. 7—The effect of adhesive levels (starch/latex 1:1) on absorption

Changing the adhesive level has the effect of changing all the variables associated with the coating structure in equation (*2*) (ϵ, τ and r). Yet, by keeping the adhesive level constant and at a relatively high level and by using a wide range of clays to produce coatings, it has been found that the absorption behaviour can be closely related to one parameter, the porosity ϵ. Thus, ten commercial coating clays from English and American sources were used to produce coatings containing 15 pph adhesive (starch/latex 1 : 1). The time t for each coating to absorb a 4 micron thick oil film was determined and the porosity ϵ measured from the volume of oil necessary to saturate the coating layer. From equation (*2*), a linear relationship should exist between t and $1/\epsilon^2$ if the other structure factors τ and r do not change substantially between coatings. The results are illustrated in Fig. 8 and show that a reasonable linear relationship is obtained from which the relative absorption behaviour can be predicted with reasonable accuracy from a knowledge of the coating porosity at this particular adhesive level and composition.

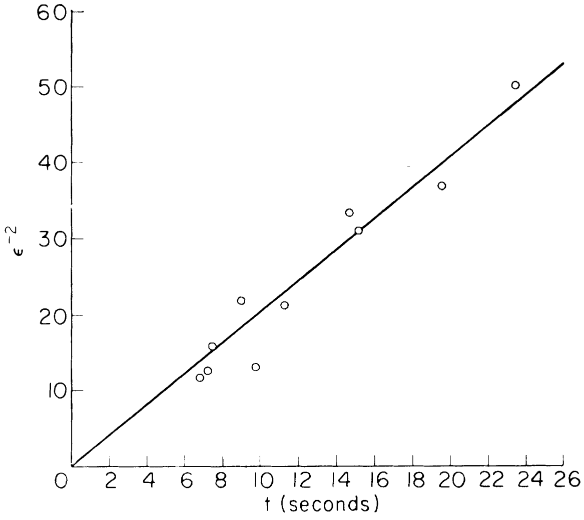


Fig. *8*—The absorption times of 10 different clays containing 15 pph adhesive as a function of $1/\varepsilon^2$

Coating structure and permeability

Of the three coating structure variables in equation (*2*), only the porosity is amenable to simple direct measurement. An approach to the other variables will now be described so that a reasonable explanation for the observed oil absorption rates may be attempted.

The implicit assumption made in the derivation of equation (*2*) is that the porous layer behaves as an array of capillaries of circular cross-section. In order to make this model more realistic, therefore more applicable to the more complex structures encountered in practice, various workers have considered adapting the model to include a distribution of capillary diameters(9) arranged in a parallel or serial manner.

Another approach, first used by Childs & Collis-George(10) is to consider the porous medium to be built up from layers; each layer contains a distribution of pore sizes that is typical of the complete layer. Then any two adjacent layers will have a statistically determinable probability for one size of pore leading into any other size of pore in the adjacent layer. With the assumption that the resistance to flow is principally controlled by the smallest of the pores in a probability sequence going through the thickness of the bed, an expression was developed for the total permeability. This expression contained a constant factor that could be determined only experimentally. This approach has been further developed by Millington & Quirk,(11) who produced an expression for the permeability K in terms of the porosity ϵ and the pore size distribution expressed in the form of m classes of pores of different radius r, each having the same fractional volume of ϵ. Then the permeability is—

$$K = \frac{\varepsilon^{4/3}}{8m^2}[r_1^2+3r_2^2+5r_3^2+\ldots+(2m-1)r_m^2] \qquad (3)$$

In the case for which equation (*2*) applies, the analogous expression for the permeability is given by $K = r^2/8$, so that equation (*3*) may be used to produce a value for r in terms of the pore size distribution of the coating layer, whose determination by mercury porosimetry will be described in a later section.

The tortuosity factor

THE tortuosity factor was first investigated by Carman,(12) who showed that, for packings of irregular powders, the tortuosity factor was approximately 2·5. More detailed work has since been undertaken to relate τ to the shape and orientation of the particles making up the bed. Thus, Fowler & Hertel(13) have related tortuosity to the mean sine of the angle ϕ between the direction of general flow and the normal of the particle surface elements—

$$\tau = \frac{3}{2(\sin^2\varphi)_{av}}.$$

The value of τ appropriate to spheres and to cylinders arranged parallel and at rightangles to the general flow direction was experimentally confirmed by Sullivan & Hertel(14) using glass beads and fibres. Coulson(15) applied this approach to a variety of other shapes in different orientations and, in particular, considered thin plates oriented with their faces at rightangles to the direction of flow. This model is appropriate to the case of coating layers composed of plate-like kaolin particles arranged with a high degree of orientation in the plane of the coating. Thus, Coulson gives a value of $\tau = 7{\cdot}5$ for plates oriented at rightangles to the flow and with a diameter to thickness ratio of 8 : 1. With the kaolin used in this work, the mean diameter to thickness ratio has a value of 10 : 1 determined from shadowed electron micrographs. The orientation of the kaolin in a coating has also been measured by X-ray methods,(7) which show a predominant alignment in the plane of the coating. For a coating with a low adhesive content (5 pph), the tortuosity factor of Coulson may be used in conjunction with the oil absorption data to calculate from equation (*2*) a value for the effective mean pore radius.

Mercury porosimetry data

THE measurement of pore size distribution by this method was originally proposed by Washburn and first demonstrated by Ritter & Drake.(16) Since then, the generally complex internal geometry of porous materials containing a wide distribution of pore sizes has recently led to a reinterpretation of the data obtained by this method. Frevel & Kressley,(17) for example, have shown that, in the case of closely packed uniform spheres, the internal voids are filled at a pressure corresponding to that of the narrowest opening between the particles, in the manner of the classic 'ink-bottle' pores. This approach has been further extended by Mayer & Stowe,(18) who have also considered the 'breakthrough' pressure as a function of the porosity (or packing geometry) of uniform spheres. Additional information may also be obtained by following the extrusion of mercury from the porous body on releasing the applied pressure. Alternate intrusion and extrusion of the mercury will produce hysteresis loops that may be interpreted to give the ratio of smallest to largest dimensions of a pore, also the total volume of blind or 'ink-bottle' pores. Thus, Svata(19) has applied the Reverberi hysteresis method to determine the structure of metal powder compacts. The type of hysteresis loop produced with a coating layer containing 5 pph adhesive is shown in Fig. 9. After the initial mercury intrusion, a closed hysteresis loop is formed. The extrusion sector of the hysteresis loop was used to estimate the pore structure available for oil flow through the layer and to produce a value for the effective pore radius by the method previously described. An effective pore radius of 0·06 micron is obtained. Compared with this, the oil absorption data and a tortuosity factor of 7·5 gives an effective pore radius of 0·09 microns. It should be pointed out, however, that the latter estimate is particularly sensitive to errors in the value of τ, which occurs as a squared term in the calculation and that agreement between the two methods would be produced by $\tau = 6{\cdot}0$.

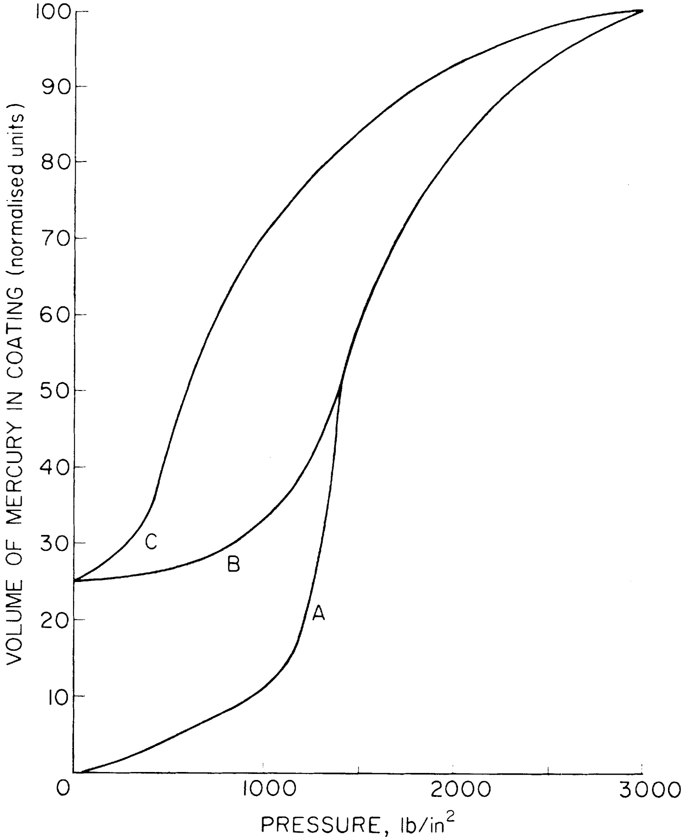


Fig. 9—Mercury porosimetry hysteresis curve for a coating containing 5 pph adhesive

A—initial pressure increase B—second pressure increase C—pressure decrease

A reasonable model can therefore be established for the absorption of oils into paper coatings. A better test of the Millington & Quirk equation will be produced by a direct measurement of the permeability of the coating measured by passing oil through it under a known pressure differential.

Conclusions

THE rate of oil absorption into coating layers can be determined quantitatively from reflectance measurements; by measurement of the time for oil films of different thicknesses to be absorbed into coatings, it has been established that a simple model for absorption based on the Washburn capillary absorption equation can be used to relate absorption times to the film thickness and the viscosity and surface tension of oils. For coatings containing the same amount of adhesive (15 pph), but made from a wide variety of coating clays, it has been established that the absorption time for an oil is proportional to $1/\epsilon^2$, where ϵ is the coating porosity.

Estimates of the effective pore radius of a coating for absorption have been made using mercury porosimetry data applied to the method of Millington & Quirk. This estimate is comparable with one based on oil absorption data and a tortuosity factor taken from Coulson.

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Discussion

Chairman The coating of paper is an ancient art—the Chinese did it 2 000 years ago (surface pigmenting their paper), also the Turks in the 15th and 16th centuries (treble treatment). Machine coating was introduced in this country for the first time in the sixties of last century. Now the surface treatment of paper and board is a growth industry, with web widths on or off the machine up to 10 m for printing papers on an airknife coater and speeds up to 1 000 m/min on a blade coater. What is our scientific understanding of these paper treatments after doing them for the last 2 000 years?

Prof. B. G. Rånby I have one comment about the possibilities of making paper less permeable. As pointed out by Prof. Stannett, the only reasonable way is to put a coating on as a continuous surface layer. Any attempt to disperse particles or latex in the fibre suspension or impregnate the paper to decrease permeability are much less effective.

There is one more point I wish to make related to the problem of having polymers other than those now commercially used to decrease the permeability by surface coating. The new polymers that have been brought on to the market recently—plastics like Barex and Lopac containing acrylonitrile—are very impermeable, they have a high mechanical modulus and they can also be used within a wide temperature range, which makes them favourable as packaging materials. It has also been stated that these new materials have advantages from an environmental point of view: we have to be careful there. PVC develops hydrogen chloride when burned, which is not really poisonous, although it is an acid. We must look also at the burning of the acrylonitrile polymers: at low temperature with insufficient oxygen, they give hydrogen cyanide, which *is* poisonous. If you burn at high temperatures with an excess of oxygen present, these plastics develop nitrous oxides, which are rather detrimental to the environment. The same is true for other polymers containing nitrogen—polyamides, polyurethanes and native products like leather and wool. We have to define the burning conditions carefully for temperature and oxygen flow to prevent formation of poisonous and otherwise undesirable gases.

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Mr J. R. Parker I would like first of all to thank Dr Youngs for so kindly talking to papermakers about this rather complicated subject. I think his approach is of great value and this will become more apparent in the further papers to be presented.

Here, we have in fact a macroscopic measure of the behaviour of fluids in unsaturated porous materials. This has great advantages over the approach for which a model is needed. This is not to say that models are not of great interest and importance, but here is a means of describing the movement of liquids without making any assumptions about the nature of the material.

Dr Youngs' approach also gives the answer to a question posed by Dr Tollenaar many years ago, 'How can we explain the distribution of oil up a strip of paper during a capillary rise experiment?' He had to do this in terms of a rather complicated model based on interconnected capillaries. This experimental result, as we see now, can be subjected to quite a different sort of treatment and useful information can be obtained for application to other problems. This approach is relevant to the setting of inks on printing papers.

May I say a word of appreciation about Dr Oliver's contribution. His work reveals phenomena that are of great importance in the printability field.

Dr J. D. Peel Mr Gate, referring to your graphs from the mercury porosimeter apparatus, did your analysis of the results take account of the effect of the pressure of the mercury on the pore size distribution in the clay? The pressures were fairly high and would probably alter the pore size distribution as the experiment progressed.

Mr L. F. Gate This is quite a valid point. My answer to it is that we only get to about 20·7 MPa (3 000 lbf/in^2), which may sound a lot, but the local distribution of pressure inside a coating will in fact be much less than that.

We have also looked at the forces necessary to disrupt a coating by using the Talystep. By increasing the loading on the stylus, a point is reached when you begin to disrupt the coating and a track is left behind as the stylus passes over it. From this, you can, from the loading on the stylus, the area of contact and the time of contact, get an estimate of the forces necessary to start disrupting the coating—and these in fact work out to be something of the order of 69 MPa (10 000 lbf/in^2). By the fact that you can get pool values that agree with other methods, using materials you might expect to be crushed, you could come to the conclusion that it is not particularly important, as the local variation pressures within the coating under the intrusion pressures are not great.

Dr E. L. Back With respect to both papers by Dr Youngs and Mr Gate, it might be appropriate to say a few words about the directional anisotropy of pores in paper. It means comparing the porosity and flow restriction in the Z-direction with that in the sheet direction. One way to quantify this comparison is by the effective pore radius for capillary flow by the Lucas-Washburn equation in these two directions. Fig. J shows the ratios of these two effective pore radii, that in the Z-direction divided by that in the machine-direction, for boards over a range of densities. These are rather thick boards made on Fourdrinier machines and the layered structure of paper increases this anisotropy. The ratio for the papers we tested is in the range of 0·01–0·04.

In these measurements, the same liquid is used in both directions, so the wettability does not enter this ratio. We found this to be true when we compared oil and water, so long as the papers were unsized or free from inherent resin.

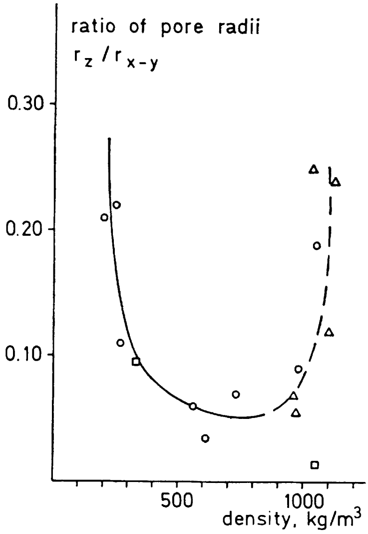

Fig. J

The ratios in this case were the same, within ± 10 per cent. For sized papers and papers with a large amount of mechanical pulp with inherent resin(such as newsprint), even if a surface agent was added, the ratios fell another one or two decades with water compared with oil. This might indicate that there is an additional anisotropy superimposed when resin or rosin is added to the paper. The distribution of this oleophobic compound will thus change the pore anisotropy.

Pore anistropy is important when evaluating the paper by impregnation, as well as for all printing.