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A liquid absorption model

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Abstract

The tortuosity factor is given as the ratio of the theoretical absorption coefficient calculated from the cylindrical model to the observed absorption coefficient by the Bristow method. A new formula to give mean pore radius of paper is derived by taking account of the liquid absorption into every pore size according to the Lucas-Washburn equation.

The liquid absorption model is proposed to allow calculation of transferred liquid volume to paper in an arbitrary time from initial absorption through saturation. The results of the transfer tests of ethyl alcohol to four kinds of papers by the Bristow method show good agreement between the observed and calculated liquid volumes transferred to the papers.

For other non-aqueous liquids which show the same V(t) vs. $(\gamma_L\ t/\eta)^{1/2}$ relationship in the liquid transfer tests to a particular paper as ethyl alcohol, it is assumed that the liquid volume transferred to the paper in an arbitrary time can be estimated by means of the model.

Introduction

Various testing methods for evaluating liquid absorption of paper have been proposed (1,2). The Bristow apparatus (3) is widely used for evaluating liquid absorption into paper during short time intervals, which gives us valuable information such as roughness index, absorption coefficient and wetting delay. There are many reports on application of this apparatus (4-7). But, there has not been an application of the formula which enables calculation of absorbed liquid volume into paper quantitatively as a function of time and fundamental properties of paper and liquid.

For liquid absorption into coatings, however, reference can be made to some studies on the formulation. Gate and Windle (8) gave an equation to describe oil absorption into a coating, which is obtained by modifying the Lucas-Washburn equation with tortuosity factor and porosity. As tortuosity factor, they used a value of 7.5 for the coating, referring to the result that Coulson (9) gave for plates oriented at right angles to the flow and with a diameter to thickness ratio of 8:1, and also demonstrated that it was possible to calculate the effective mean pore radius from their equation using Coulson's tortuosity factor. However, it may be supposed that the tortuosity factor of a particular coating should take its own value, so it may not be reasonable to assume a constant value for the tortuosity factor. Ranger (10) presented a useful method for coating pore structure analysis, in which effective pore radius, number of capillaries per square meter and capillary length of a coating are calculated using some equations with the K & N ink penetration test combined with the test under vacuum. Ranger also adopted Coulson's value for tortuosity factor for coatings.

In this study, new approaches for determining tortuosity factor and mean pore radius of paper and a new liquid absorption model (are proposed) which enable us to calculate absorbed liquid volume in an arbitrary time. Liquid transfer tests with non-aqueous liquid by the Bristow method and measurements of pore size distribution of paper by mercury porosimetry are applied for those calculations.

Experimental

Measurement of liquid transfer

Liquid transfer tests by the Bristow method were carried out to measure transferred volumes of liquids in various contact times. As the Bristow apparatus a KRK model was used. For sample papers three commercial woodfree papers (A1,A2 and A1C) and a mechanical paper listed in Table 1 were employed. One of the woodfree papers (A1C) was obtained from calendering A1 under a pressure of 3 kg/cm and a speed of 5m/min by a laboratory supercalender. As the test liquids ethyl alcohol (EtOH), polyethylene glycol (PEG) and mixtures (EtOH/PEG=100/0, 50/50, 30/70, 20/80, 10/90, 0/100) were employed. The liquid properties are given in table 2. 0.015 percent of malachite green was added, where shown, to the liquids for colouring. All tests were carried out under conditions 20^{o}C/65% RH.

Measurement of pore size distribution

The pore size distributions of the sample papers were measured by a mercury porosimeter (Carlo Erba porosimeter 2000 & 120). The measuring pressure range was 0 to 1000 bar, under which condition measurable pore radii are between 7.5nm and 0.3mm. The effective thicknesses of the sample papers (given in Table 1) were measured by mercury displacement method with a Macropore Unit Model 120 porosimeter.

Code	Sample	Effective thickness µm
A1	Woodfree paper	110.7
A2	Woodfree paper	79.3
B	Mechanical paper	71.1
A1C	Calendered A1	100.4

Table 1 Sample papers

EtOH/PEG ratio	Malachite green %	Surface tension mN/m	Viscosity mPa.s
100/0	0.015	21.7	0.766
50/50	0.015	27.8	6.547
30/70	0.015	32.0	13.826
20/80	-	35.0	22.248
10/90	-	39.1	35.324
0/100	-	43.6	57.981

Table 2 Properties of liquids at 20°C used for the liquid transfer tests

Theory

It is generally known that a liquid penetrates into a capillary tube according to the Lucas-Washburn equation which is expressed by

$$h = \left(\frac{r \gamma_L t \cos\theta}{2\eta} \right)^{1/2} \qquad (1)$$

where h = penetration length
η = viscosity of the liquid
γ_L = surface tension of the liquid
θ = contact angle (assuming zero for the liquids in Table 2)
r = capillary radius
t = penetration time

It is also known that when a non-aqueous liquid is transferred to paper there appears to be a linear relationship between transferred liquid volume V and square root of time $t^{1/2}$. The following relationship is utilised for evaluating liquid absorption into paper by the Bristow apparatus.

$$V = k_r + k_a t^{1/2}. \tag{2}$$

This relationship holds good in the case of the penetration of non-aqueous liquids, where k_r and k_a are referred to as the roughness index and the absorption coefficient, respectively. However, Eq.(1) and Eq.(2) are not adequate to express absorbed liquid volume into the pore structure of paper in an arbitrary time quantitatively in terms of fundamental properties of paper and liquid. The liquid absorption model to be constructed in this study is derived from more general consideration.

Cylindrical model

The simplest approach for modelling liquid absorption is to suppose a cylindrical model shown in Fig. 1(A). The pore structure of paper is assumed to compose of n capillaries (per unit area) of mean pore radius r_{ave} which pass through the paper from one side to the other. The total pore volume per unit area, V_T is given by

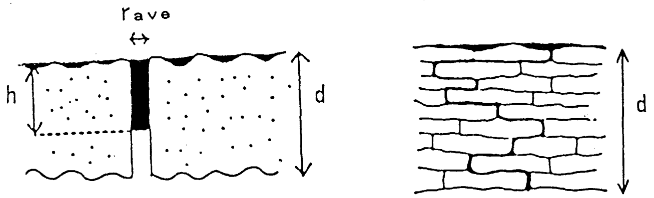
$$V_T = n\pi r_{ave}^{2} d, \tag{3}$$

where d is effective thickness of paper, Considering Eq.(1), Eq.(3) and the liquid volume retained on the paper surface per unit area, V_r, the transferred liquid volume per unit area in time t, V(t) is given by

$$V(t) = V_r + \frac{V_T}{d}\left(\frac{r_{ave}\gamma_L t}{2\eta}\right)^{1/2}, \qquad (4)$$

where V_r might be idealistically equivalent to the Bristow roughness index k_r. Accordingly, the theoretical absorption coefficient K_a' of the cylindrical model is given by

$$K_a' = \frac{V_T}{d}\left(\frac{r_{ave}\gamma_L}{2\eta}\right)^{1/2}. \qquad (5)$$


(A) Cylindrical model

(B) Tortuous model

h: Penetration depth
d: Effective thickness
r_{ave}: Mean pore radius

ξd : Actual Penetration distance
ξ : Tortuosity factor

Fig. 1 Schematic diagrams of pore structures of paper

Tortuosity factor

A more realistic model for liquid absorption into paper is constructed by introducing a tortuosity factor ξ into the model equation. As shown in Fig. 1(B), a liquid penetrates into paper through tortuous capillaries in the Z direction of paper. The tortuosity factor is considered as the ratio of the actual penetration distance to a certain depth level of paper to the direct distance to the same depth level. Since the actual penetration distance of the paper with the effective thickness d is ξ d, the actual absorption coefficient ka is given by

$$K_a = \frac{V_T}{d\xi}\left(\frac{r_{ave}\gamma_L}{2\eta}\right)^{1/2} . \tag{6}$$

Eq. (5) and Eq. (6) give

$$\xi = ka'/ka \tag{7}$$

Therefore, ξ is determined from the ratio of the theoretical absorption coefficient of the cylindrical model to the observed absorption coefficient by the Bristow method. In this case, the number of capillaries per unit area of paper, N is given by

$$N = \frac{V_T}{\pi r_{ave}^2 \xi d} . \tag{8}$$

The transferred liquid volume per unit area in time t, V(t) is given by

$$V(t) = V_r + \frac{V_T}{\xi d}\left(\frac{r_{ave}\gamma_L t}{2\eta}\right)^{1/2} . \tag{9}$$

A new definition of mean pore radius

Mean pore radius of paper, $r_{ave\,(por)}$ may be calculated from the following equation using the pore size distribution measured by a mercury porosimeter.

$$r_{ave\,(por)} = \sum_{i=1}^{end} r_i V_{Ti}/V_T \ , \tag{10}$$

where r_i = the ith largest capillary radius ($r_1 > r_2 > \ldots > r_i >$),to a value of i,

V_{Ti} = total volume of capillaries of the radius r_i per unit area.

However, no information on liquid absorption is included in Eq.(10). Therefore, it is reasonable to give a definition of mean pore radius such that both conditions of the pore size distribution of paper and the liquid absorption based on the Lucas-Washburn equation are satisfied. In the first case liquid absorption during the time intervals where the V(t) vs. $t^{1/2}$ plot shows a linearity is considered below.

If there are N_i capillaries of radius r_i per unit area, the total volume of the capillaries, V_{Ti} is given by

$$V_{Ti} = N_i \pi r_i{}^2 d\xi. \tag{11}$$

The transferred liquid volume per unit area in time t, V(t) is given by

$$V_i(t) = V_r + \frac{V_{Ti}}{\xi d}\left(\frac{r_i \gamma_L t}{2\eta}\right)^{1/2} . \tag{12}$$

By taking account of the surface roughness and the pores size distribution of paper, the total transferred liquid volume in time t, (V(t) is given by

$$V(t) = V_r + \frac{1}{\xi d}\left(\frac{\gamma_L t}{2\eta}\right)^{1/2} \sum_{i=1}^{end} V_{Ti} r_i^{1/2} \tag{13}$$

where it is assumed that each capillary has the same value of tortuosity factor ξ.

By comparing the second term of Eq.(9) with that of Eq.(13), an equation for mean pore radius r_{ave} is obtained.

$$r_{ave} = \left(\sum_{i=1}^{end} V_{Ti} r_i^{1/2} / V_T \right)^2 \tag{14}$$

This is a new definition of mean pore radius. Since V_{Ti} , r_i and V_T can be measured by a mercury porosimeter, a value of r_{ave} can be determined. By using the value of r_{ave}, the theoretical absorption coefficient of the cylindrical model K_a' can be calculated from Eq. (5). By using the value of K_a' and the observed Bristow K_a, the tortuosity factor ξ can be calculated from Eq.(7). If the value of r_{ave} is applied to Eq.(9), the transferring liquid volume in time t can be calculated during the time intervals where the V(t) vs. $t^{1/2}$ plot shows a linearity.

A liquid absorption model

It is generally known that there is a linear relationship between V(t) and $t^{1/2}$ during the initial absorption times. The linearity is reasonably understood based on Eq.(9) or Eq.(13). But, there has been no reasonable model presented to describe deviations from linearity for the longer absorption time.

In order to obtain a more general model to cover the whole time range, some modification of Eq.(13) is necessary. A new liquid absorption model is derived from including the following assumptions:-

1. The maximum liquid volume transferred to a paper is the sum of the total pore volume in the paper as measured by a mercury porosimeter and the liquid volume retained on the paper surface as expressed by the Bristow k_a.

2. All pores of a paper pass through a distance ξd from one side to the other.

3. The penetration of a liquid into an individual capillary is described by the Lucas-Washburn equation.

4. Once capillaries of a particular size are saturated with a liquid, liquid movement in the capillaries ceases.

Assumptions 1, 2 and 3 appear in the preceding discussions. Assumption 4 is a new introduction.

Now, let the number and total volume of capillaries of radius r_i be N_i and V_{Ti} ($=N_i \pi r_i{}^2 \xi d$), respectively. Let the capillaries be numbered in decreasing order of radius as $r_1>r_2>r_3 ... r_k>r_{k+1}> ...$. The penetrations in all the capillaries start at the same time. During the initial absorption period until the capillaries of radius r_1 are saturated with the liquid [that is, $0<t<t_1=2\eta(d\xi)^2/(r_1\gamma_L)$], all the pores participate in the absorption process, so that Eq.(9) or Eq.(13) can be used directly. Then, the capillaries are sequentially saturated with the liquid in decreasing order of radius. At time t_k the capillaries of radius r_k complete their absorption. Until this time the capillaries of r_1, r_2, ..., r_{k-1} have been saturated with the liquid. This absorption process is schematically shown in Fig.2.

Consequently, the equation for V(t) in time t is given by

$$V(t) = K_r + \sum_{i=1}^{K} V_{Ti} + \frac{1}{\xi d} \cdot \left(\frac{\gamma_L t}{2\eta}\right)^{1/2} \sum_{i=K+1}^{end} V_{Ti} r_i^{1/2} , \qquad (15)$$

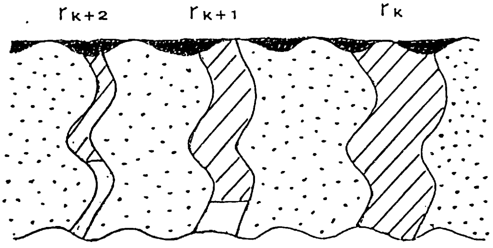


Fig.2 Schematic diagram of liquid absorption

where $t_K \leqq t \leqq t_{K+1}$,

$$t_K = \frac{2\eta(\xi d)^2}{r_K \gamma_L}$$

Results and Discussion

Liquid transfer and porosity

The results of the transfer tests of the EtOH/PEG mixtures to sample B and A1 by the Bristow method are shown in Fig.3 and Fig.4 respectively. Familiar linear relationships can be seen between V(t) and $t^{1/2}$ as given by Eq.(2). Referring to Eq.(9), it is supposed that if liquid transfer tests to the same paper using different liquids are carried out, then a linear relationship appears between V(t) and $(\gamma_L t/\eta)^{1/2}$, and that all the data points are on the same line in the V(t) vs. $(\gamma_L t/\eta)^{1/2}$ plot. The slope of the one is given by (ε/ξ) X $(r_{ave}/2)^{1/2}$ from Eq. (9) where V_T/d is replaced by ε i.e. porosity. This indicates that the value of the slope is independent of liquid properties and dependent on mean pore radius, tortuosity factor and porosity. The transfer results for sample B show such relationship in Fig.5. Other researchers have also found similar results for the transfers of various organic liquids (3, 8,11). However, sample A1 shown in Fig.6 shows different results, in which the EtOH/PEG mixtures of 0 to 70% PEG content reveal such tendency, but the EtOH/PEG mixtures

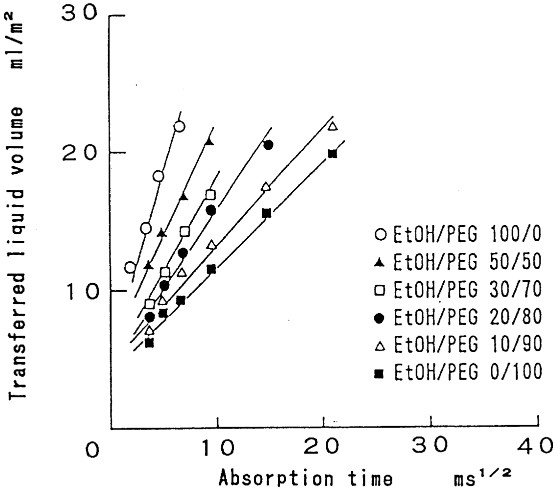


Fig.3 Transferred liquid volume as a function of $t^{1/2}$ in the case of sample B

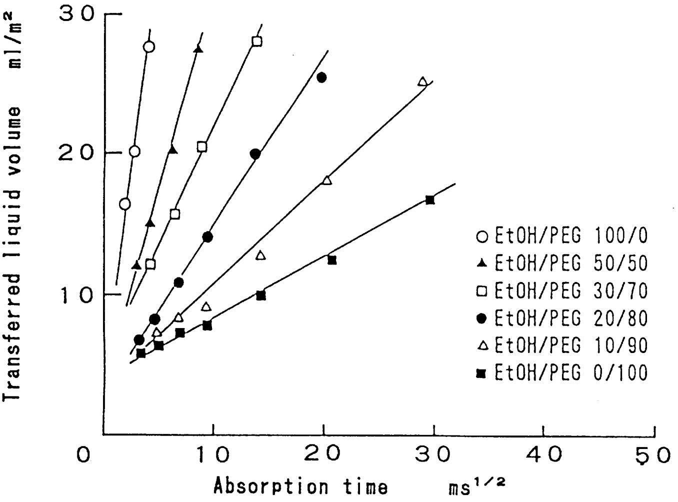


Fig.4 Transferred liquid volume as a function of $t^{1/2}$ in the case of sample A1

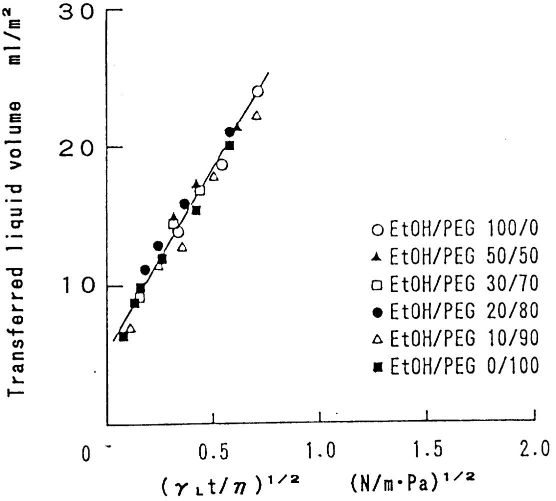


Fig.5 Transferred liquid volume vs. $(\gamma_L t/\eta)^{1/2}$ in the case of sample B

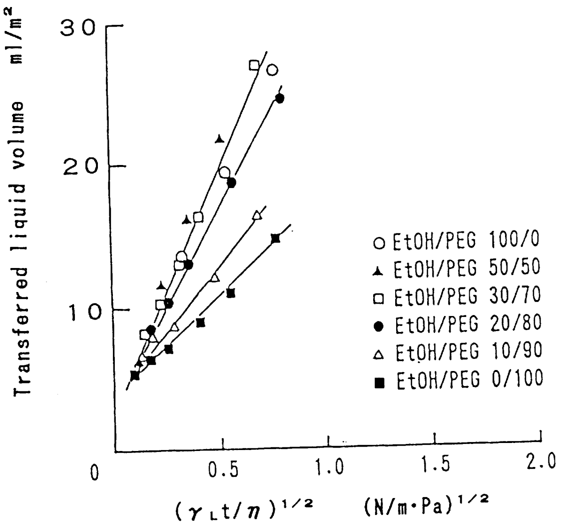


Fig.6 Transferred liquid volume vs. $(\gamma_L t/\eta)^{1/2}$ in the case of sample A1

of more than 70% PEG content show smaller values of the slope with increasing PEG content. These results indicate that liquids of high EtOH content (low PEG content) penetrate into every pore of paper, but liquids of high PEG content do not. In later discussion ethyl alcohol (EtOH) will be used as a pointer for transfer tests and for modelling.

Fig.7 shows the transfer curves obtained by the measurements of ethyl alcohol transfer in longer absorption time, where the y axis is expressed by the difference between transferred liquid volume and roughness index k_r. It is seen that the transferred liquid volume on each paper shows a linearity with $t^{1/2}$ during the initial absorption period, but over longer absorption time absorption rate decreases with time since the pores of each paper are sequentially saturated with the liquid. The dashed lines of Fig.7 show the transfer curves obtained by correcting the slit length of the head box of the apparatus. This was necessary because the absorption over such long absorption time creates spreading of the working length of

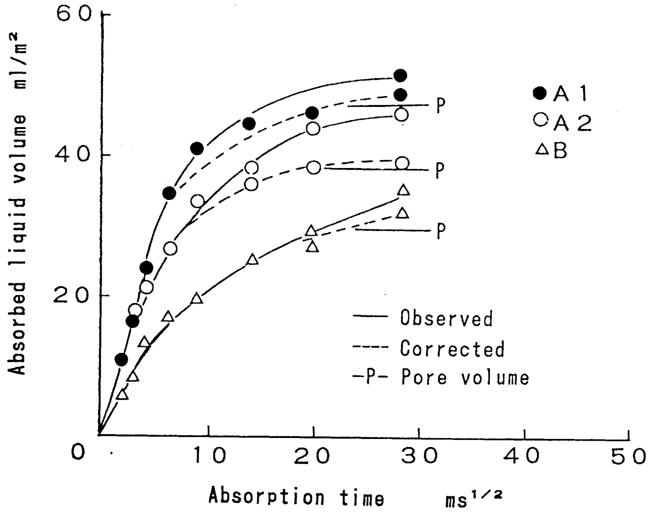


Fig.7 Transfer curves of ethyl alcohol from the initial absorption through the saturation

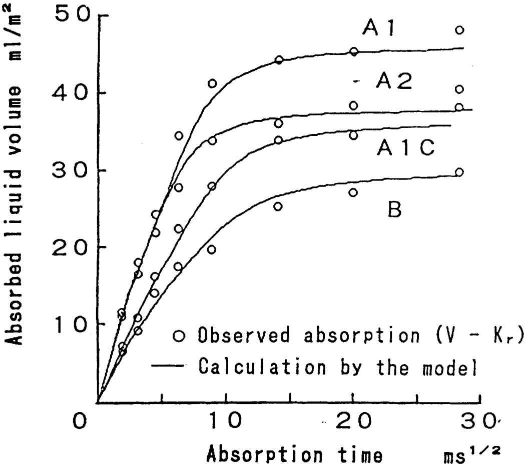


Fig.8 Comparison between observed liquid volumes transferred to papers and corresponding calculated values by the liquid absorption model

the slit. It is apparent that the maximum liquid volumes to be absorbed into the papers, excluding liquid volumes retained on the paper surfaces K_r, are almost equivalent to the total pore volumes of the papers measured by a mercury porosimeter. Accordingly, assumption 1 presented above seems to be acceptable.

Mean pore radius and tortuosity factor

The calculated results of the tortuosity factors, mean pore radii, and related properties of the sample papers are shown in Table 3. The mean pore radii r_{ave} of the papers estimated by the new definition in Eq.(14) give values of about 2 μm, which are smaller than the mean pore radii $r_{ave\,(por)}$ obtained when only using the pore size distributions. It is found that there are about 10^9 capillaries of radius r_{ave} per square meter of paper and the value of tortuosity factor ξ of each paper is more than 10.

It is obvious from Eq.(6) that when absorptions of the same liquid into different papers are compared, the factors affecting the absorption coefficients are the porosity (V_T/d), tortuosity factor and mean pore radius of paper. Comparing sample A1 with sample B, they have almost the same mean pore radius and porosity, but have different absorption coefficients. As a result, there appears to be large difference.

Code	V_T ml/m^2	porosity %	$r_{ave\,(por)}$ μm	r_{ave} μm	N $\times 10^9$/m^2	K_a ml/m^{2}ms$^{1/2}$	ξ
A1	47.0	42.5	2.38	1.83	3.19	5.37	12.7
A2	38.2	48.2	2.97	2.13	2.31	5.74	14.6
B	30.5	42.9	3.24	1.91	1.63	3.08	23.0
A1C	37.0	36.9	1.99	1.52	3.35	3.55	15.2

Table 3 Estimation of tortuosity factor, mean pore radius and related properties.

only in tortuosity factor between the two samples. In this case the main factor differentiating the two absorptions is the tortuosity. By comparing sample A1 with sample A1C, it is seen that the reduction of pore volume brought about by calendering decreases the porosity and mean pore radius, but increases the tortuosity. This indicates that, as the result of the reduction of pore volume, the effect of the micro structure of paper on the liquid absorption increases.

Accordingly, it is supposed that tortuosity factor is an essential property to describe liquid absorption.

Applicability of the liquid absorption model

Fig. 8 shows the relationship between the observed transferred liquid volumes by the Bristow method and the calculated values for various absorption times by the liquid absorption model. The y axis stands for the absorbed liquid volume, expressed as the difference between the transferred liquid volume and the roughness index. For the calculations the sum of the second and third term of Eq.(15) was utilised. It appears that the model given by Eq.(15) gives a good estimate of the liquid volume absorbed into paper in an arbitrary time for the initial absorption through the saturation.

The liquid absorption process can be reasonably understood from this model as follows. The pores of radius r_1, r_2, r_3,... are saturated with a liquid sequentially in decreasing order of radius. During the initial absorption period, the second term of Eq.(15), that is ΣV_{Ti} has relatively small effect on the liquid absorption, but the third term has a major effect on it. This leads to the apparent linear relationship between V(t) and $t^{1/2}$. During this period the transferred liquid volume is given by familiar equations such as Eq.(2) in terms of the absorption coefficient K_a and roughness index K_r. As time goes on, more pores appear which are saturated with the liquid, leading to obvious deviations of the V(t) vs. $t^{1/2}$ relationship from linearity.

Also, the liquid absorption model makes it possible to show visually how the pores of various radii of paper are saturated with the liquid with time using histograms of pore size distribution. The histograms of the ethyl alcohol absorption process are illustrated in Fig.9, 10, 11 and 12 (where the number of classes of the histograms has nothing to do with the number of pressuring steps by mercury porosimetry). For the initial absorption the pores with near mean radius and largest in volume are of most influence. Secondly, the pores with larger radii are saturated with the liquid with time. Finally, the fine structures of micropores are saturated with the liquid and then, the absorption process is complete.

As for the other non-aqueous liquids which show the same V(t) vs. $(\gamma_L t/\eta)^{1/2}$ relationship as ethyl alcohol in Fig. 5 and, partly, in Fig.6, all the pores of paper are supposed to participate in the absorption of the liquids during short time intervals in the same way. In such case it is assumed that the volume of a particular liquid to be transferred to a paper in an arbitrary time can be estimated by Eq.(15) in terms of the viscosity and surface tension of the liquid and the roughness index, tortuosity factor and pore size distribution of the paper.

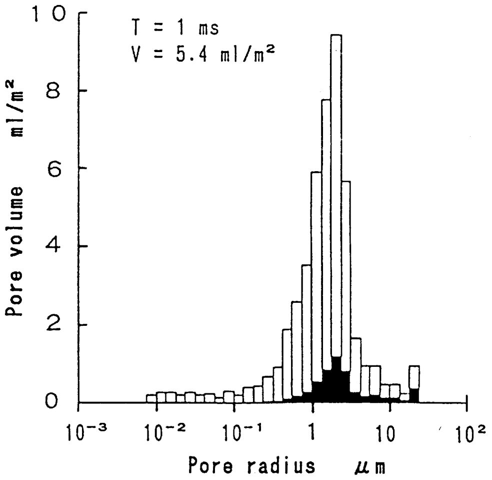


Fig.9 Ethyl alcohol absorption into sample A1 in 1 ms of absorption time

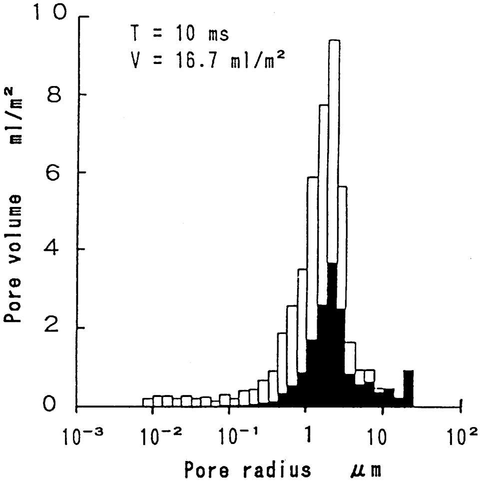


Fig.10 Ethyl alcohol absorption into sample A1 in 10 ms of absorption time

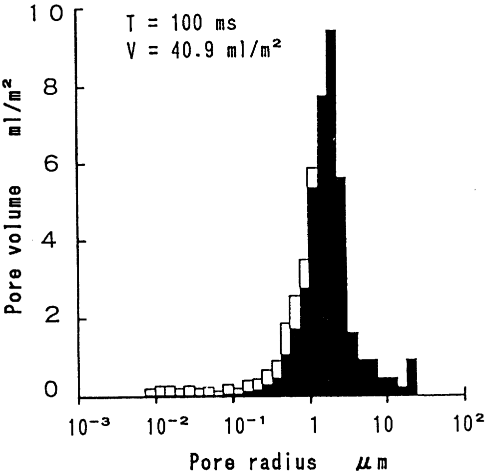


Fig.11 Ethyl alcohol absorption into sample A1 in 100 ms of absorption time

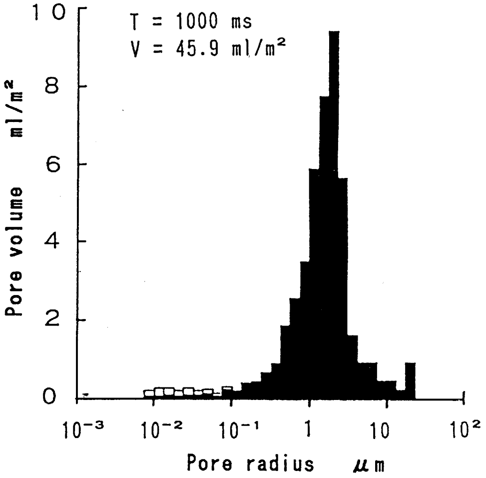


Fig.12 Ethyl alcohol absorption into sample A1 in 1000 ms of absorption time

However, for such liquids that revealed deviations from the V(t) vs. $(\gamma_L t/\eta)^{1/2}$ plot for ethyl alcohol e.g. the EtOH/PEG mixtures of high PEG content reveal in Fig.6, not all the pores of paper are supposed to participate in the liquid absorption during short time intervals. In such cases, E9.(15) is not applicable.

Conclusions

The tortuosity factor is given as the ratio of the theoretical absorption coefficient calculated from the cylindrical model to the observed absorption coefficient by the Bristow method. A new formula to give mean pore radius of paper is derived by taking account of the liquid absorption into every size of pore according to the Lucas-Washburn equation.

The liquid absorption model is proposed to allow the calculation of transferred liquid volume to paper in an arbitrary time from the initial absorption through the saturation. The results of the transfer tests of ethyl alcohol to four kinds of papers by the Bristow method show good agreement between the observed and calculated liquid volumes transferred to the papers.

For other non-aqueous liquids which show the same V(t) vs. $(\gamma_L t/\eta)^{1/2}$ relationship in the liquid transfer tests to a particular paper as ethyl alcohol does, it is assumed that the liquid volume to be transferred to the paper in an arbitrary time can be estimated by means of the model.

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Transcription of Discussion

A LIQUID ABSORPTION MODEL

H Yamazaki, Y Munakata

Dr P Kolseth, STFI, Sweden
This is a very interesting approach here. In the introduction of your paper, you quote a value of 7 1/2 for this tortuosity factor for plate materials like kaolin clay coating layers, but the calculated value you then have for paper (B) for example is as high as 23. Do you think you can give us some physical interpretation of what this structure looks like? What is the shape of the pores?

H Yamazaki
I gave a figure to help our understanding of tortuosity. This is just a model. The tortuosity factor reveals how far real pore system deviates from the idealistic cylindrical model. The tortuosity factor includes not only tortuosity but interconnection of pores and also other factors. As for the value of 7.5 for the tortuosity factor, it is not the observed value for paper but the value given by Coulson assuming such a structure as plate-like particles are oriented at right angles to the flow.

Prof J Lindsay, Institute of Paper Science & Technology, USA
I have a couple of questions about the use of mercury porosimetry to obtain an effective radius to make the Lucas Washburn equation work. Why not use the suggestion of Prof Lyne, the previous speaker, to measure absorption with one fluid and then take a value of R that makes that work and then use that as the effective radius for another fluid. There are a couple of problems with mercury porosimetry. Often you get results that give you very small pore

sizes compared to what you see optically or what you would measure with other techniques. Part of the problem is the compression of the paper that occurs at the very high pressures but even more severe is what is called the "ink bottle effect" where a pore with a small throat in the mercury porosimetry technique is viewed as being many pores with that equivalent small throat instead of one big pore that happens to have a small throat. So you tend to get a large number of small pores, based on mercury porosimetry results when in fact you may have a few large pores with small throats in them. I did a couple of calculations last night using a model pore with a small throat and a big body after the throat and I could get tortuosity factor less than 1 using your definition. Are you sure mercury porosimetry is the way to go in terms of characterising materials for absorption? Your averaging equation gives you even smaller pore sizes than the normal numerical average. I fear that the radius you use may be far too small.

H Yamazaki

I would like to re-consider Prof Lyne's suggestion myself later. The pore distribution measured by mercury porosimetry may give a distorted result. It is my view that you should not use mercury porosimetry as an absolute measure but as a comparative standard. As I answered for the previous question, it is better to think that the tortuosity factor is such a factor which reveals how far the real pore system deviates from the idealistic cylindrical model obtained from mercury porosimetry.