

IJP 02128

## Evaluation of a permeametry technique for surface area measurements of coarse particulate materials

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(Received 9 November 1989)

(Modified version received 12 March 1990)

(Accepted 15 March 1990)

**Key words:** Coarse particulate material, Particle characterization; Permeametry surface area; Microscopy surface area; Reynolds number; Porosity

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### Summary

The air permeability of powder beds of some coarse particulate solids with varying particle size and shape characteristics were measured. The specific surface area of each powder was calculated using the Kozeny-Carman equation. The air flow conditions through the powder bed were evaluated by the Reynolds number. The materials were examined in microscope to determine particle size and shape, and powder surface areas were calculated from these data. It was found that the height and the packing intensity of the powder bed did not affect the permeametry surface area. The Reynolds number indicated that the flow type was acceptable for the use of the Kozeny-Carman equation. The permeametry and microscopy derived surface areas correlated well although slightly higher surface area values were generally obtained with the permeametry technique. It is concluded that the air permeametry method can be used for the measurement of the external surface area of coarse particulate materials. For porous materials the granule density, rather than the true density, should be used for calculating the surface area.

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### Introduction

Characterization of the specific surface area of coarse particulate solids, 100–1000  $\mu\text{m}$ , is necessary for some pharmaceutical applications. For instance, in film coating of pellets and the evaluation of the subsequent drug release characteristics, the external surface area of the powder is important. Thus, a gas adsorption method that characterizes the total surface area, i.e., internal and external, is not applicable.

Hitherto, the external surface area of coarse particulate materials has normally been evaluated by calculating the surface area from microscopy measurements of the particle size and shape. However, microscope techniques have limitations in that normally only two-dimensional measurements are made and surface irregularities are not considered in the estimate of the surface area. Possible alternatives to microscopy are light blockage and permeametry measurements. The problem with the former technique is that the powders must be dispersed in a liquid, which can cause problems with dissolution and/or swelling of particles. Consequently, permeability measurements with a gas as the flowing medium seems to be an attractive measuring principle with the potential advantages

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of giving a fast and reliable measure of the external powder surface area.

Although some of the original works on permeametry surface area measurements dealt with the permeability of liquids through beds of coarse particulate powders (e.g. Carman, 1937, 1938) the pharmaceutical application of the method is normally concerned with the estimation of the surface area of fine particulate materials (less than 50  $\mu\text{m}$ ). As discussed above, the use of a gas instead of a liquid as the flowing medium is advantageous for pharmaceutical materials. However, air is much less viscous than a liquid and the flow rate conditions might therefore not be consistent with the use of the Kozeny-Carman equation. Air permeability methods for measurements of the surface area of coarse particulate materials have been presented in the literature, (Friedrich, 1957; Gupte, 1976; Casal et al., 1989) but none of these studies compared permeametry surface area data with results from other measuring principles. In the absence of such data an evaluation of the air permeability method for measuring the external surface area of coarse particulate materials was considered necessary. A constant volume permeameter was used for this evaluation because it gives a simple but precise measure of the air flow rate through the powder bed and the pressure difference across the powder bed.

## Theoretical Considerations

### *Design of powder container and permeameter*

The flow of a gas through a powder bed is dependent on the pore structure of the bed. The use of air permeability measurements for the calculation of the powder surface area requires theoretically laminar mass flow of the gas (Kaye, 1967). For beds of coarse particulate materials there is a potential risk of turbulent air flow due to the large pore diameters. To avoid this, the air velocity can be reduced by keeping the pressure differential low and increasing the length of the powder bed. However, for constant volume permeameters, the design of the apparatus makes it difficult to reduce the head of pressure below a specified limit, therefore the length of the powder

bed will be important to achieve an adequate air velocity. Furthermore, to carry out precise measurements of the flow rates in practice, especially for constant volume permeameters, it is necessary to reduce the volumetric air flow rate. This can be achieved by using a powder container with a small diameter, while maintaining an adequate ratio between the container and particle diameters. Two reasons for this are given in the literature (Coulson, 1949; Carman, 1937, 1938): Firstly, a powder tends to pack less densely near the wall of the container which can give a flow rate through the powder bed which is higher than can be predicted from the overall bed porosity and secondly, the friction of the fluid against the container wall affects the flow rate through the powder bed. Empirical correlations for these 'wall effects' have been described by Coulson (1949) and Carman (1937, 1938). The suggested correlations have a very limited effect on the calculated surface area, if the ratio between container and particle diameter exceeds 10.

The total volume of air that flows through the powder bed must be increased compared to a traditional constant volume permeameter. Such a modified permeameter (Fig. 1), as described by Friedrich (1957) and subsequently used by others (Gupte, 1976), was used in this study. With this construction, the measured volume of air flowing through the powder bed is comparatively large while, at the same time, the pressure drop over the bed is relatively low.

### *Calculation of powder surface area*

The powder surface area can be calculated from permeability data with the Kozeny-Carman equation which relates the volumetric flow rate of the gas to the characteristics of the powder bed (Carman, 1937, 1938). For the measurement of very fine powders, the rate of gas flow through the powder bed is greater than given by the Kozeny-Carman equation. This effect is called slip-flow and has been accounted for by a modified permeability equation (Carman and Malherbe, 1950). For fairly coarse particulate materials, such a slip flow correction is not necessary, because of the large diameter of the pores in the powder bed.

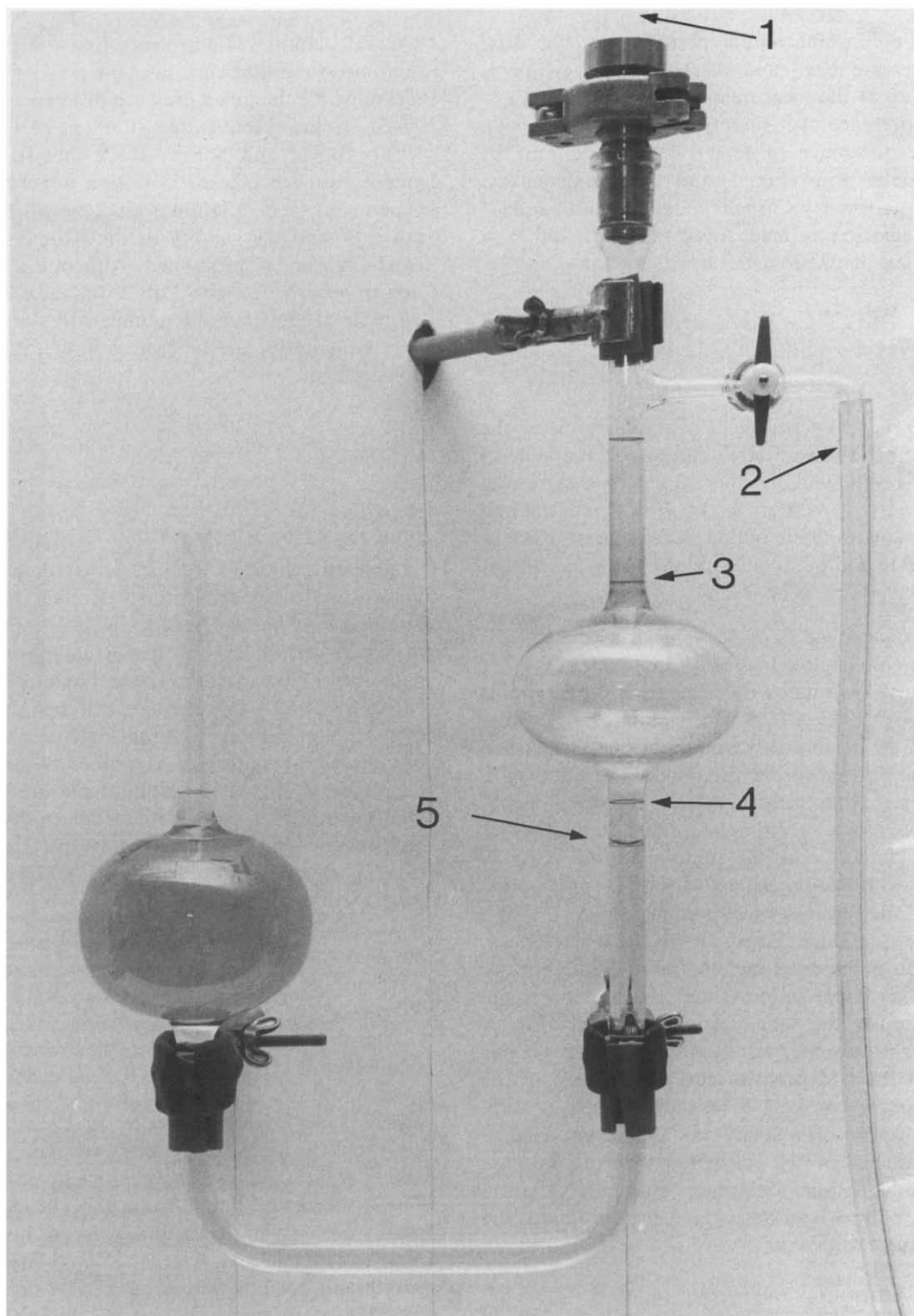


Fig. 1. The permeameter and the powder container. (1) Powder container, (2) to vacuum pump, (3) start point  $h_2$ , (4) stop point  $h_1$ , (5) equilibrium level.

For a constant volume permeameter, the driving pressure head and thereby the flow rate is changing as the measurement proceeds. The pressure difference over the powder bed is a function of the difference in level of the liquid in the manometer arms (Fig. 1) and the liquid density. Thus, the Kozeny-Carman equation can be used if an integration is made over the start and stop points on the manometer arms.

$$S_v^2 = \frac{2g\rho_w t A \epsilon^3}{kL\eta(1-\epsilon)^2} \cdot \frac{l}{\int_{h_1}^{h_2} dV/h} \quad (1)$$

Due to the geometric shape of the reservoir on the permeameter the integral cannot be mathematically solved and is therefore empirically determined, by measuring the flow time through some samples both in the permeameter used in this study and in a conventional Blaine permeameter (Alderborn et al., 1985).

#### *Calculation of the Reynolds number*

The type of flow that exists in a certain powder bed can be estimated by calculating the Reynolds number (Möller, 1972), which requires an estimation of the mean pore diameter in the powder bed and the air velocity in the pores. The mean pore diameter can be estimated from the bed porosity and the volume specific surface area of the powder (Allen, 1981a). The air velocity in the pores is related to the approach velocity by the bed porosity and the tortuosity factor (Carman, 1937, 1938). This estimation is based on the fact that only a fraction of the cross-sectional area of the powder bed is available for flow and that the pores are longer than the length of the powder bed. A reasonable estimate of the fraction available for flow is the bed porosity and an estimate of the tortuosity factor is  $\sqrt{2}$  (Carman, 1937). A measure of the approach velocity for a constant volume permeameter can be calculated with the Kozeny-Carman equation. With these assumptions the Reynolds number can be evaluated according to the following expression:

$$R_e = \frac{8hg\rho_w\rho_g(L_e/L)\epsilon^3}{kL\eta^2S_v^3(1-\epsilon)^3} \quad (2)$$

This calculation was performed firstly at the maximum pressure difference over the powder bed and secondly, for the mean pressure difference (Blaine, 1943). It has been stated (Gupte, 1976; Allen, 1981b; Büchi and Soliva, 1982) that if the Reynolds number exceeds 2 during a permeability experiment, perfect laminar air flow will probably not exist and the validity of the Kozeny-Carman equation can be questioned. Although a calculation in accordance with Eqn 2 only gives a rough estimate of the Reynolds number, it is an acceptable estimate for the type of air flow through the powder bed.

## **Experimental**

### *Materials*

Sodium chloride (cubic, crystalline puriss, Kebo, Sweden), saccharose (crystalline, K5, SSA, Sweden) and glass pellets (Kebo) represented materials with a low intraparticle porosity but with varying particle shape. Emcompress (E. Mendell, U.S.A.) represents a material consisting of porous particles. To obtain narrow size distributions of the materials, a number of sieve fractions were prepared for sodium chloride and saccharose by dry sieving with ordinary laboratory test sieves. For Emcompress only, one sieve fraction was prepared (see Tables 4–6). Glass pellets were used as supplied.

### *Methods*

#### *Particle shape*

The estimated Heywoods surface to volume shape factors (Heywood, 1954) of all materials are listed in Table 1. For sodium chloride and saccharose, literature values were used and for the glass pellets the shape factor was visually estimated from photomicrographs (Philips SEM 525, The Netherlands). For Emcompress, the Heywoods shape factor was determined according to the method reported by Nyström (1978). The particle length and breadth were measured by microscopy for approx. 100 particles and the thickness of the particles was measured in a ring-gap sizer. From

TABLE 1

Densities and estimated surface to volume shape factors for test materials

Material	Density (g/cm <sup>3</sup> )	Estimated shape factors
Sodium chloride	2.17 <sup>a</sup>	7.3 <sup>c</sup>
Saccharose	1.60 <sup>a</sup>	9.4 <sup>c</sup>
Emcompress	2.31 <sup>a</sup> 1.72 <sup>b</sup>	10.9 <sup>d</sup>
Glass pellets (200 μm, 500 μm)	2.55 <sup>a</sup>	6.0 <sup>e</sup>
Glass pellets (400 μm)	2.70 <sup>a</sup>	6.0 <sup>e</sup>

<sup>a</sup> Measured by air comparison pycnometry.

<sup>b</sup> Measured by mercury porosimetry.

<sup>c</sup> Values from Alderborn and Nyström (1982).

<sup>d</sup> Estimated according to Heywood (1954), assuming sub-angular particles.

<sup>e</sup> Visual estimation from photomicrographs.

the four geometrical forms suggested by Heywood (1954), Emcompress was fitted in with the sub-angular group.

#### Densities

The true density of all materials was measured with an air comparison pycnometer (Model 930, Beckman, U.S.A.). For Emcompress, the granule density was also determined with a mercury displacement method (Strickland et al., 1956; Wikberg and Alderborn, in press). The mean values of three determinations are given in Table 1.

#### Powder surface area by permeametry

The powder was filled manually into the powder container, a plastic tube of diameter 1.008 cm, height 60 cm and sealed at one end with a screen. The tube was fitted to the top end of the permeameter with an adapter. The pressure below the powder bed was then reduced with the aid of a pump and the time for the fluid to pass between marks  $h_2$  and  $h_1$  (see Fig. 1) on the permeameter was determined manually with a stop-watch.

Initial experiments showed that if the container was connected to the permeameter with a flexible tube instead of the powder container fixed directly to the permeameter, slightly increased flow times through the bed resulted. This is probably caused by a slight pressure drop across the flexible tube. It is thus recommended that the powder container

is directly fitted to the top end of the permeameter.

The effects of the packing intensity and the height of the powder bed on the calculated specific surface area were studied for two materials, saccharose and Emcompress, with the same size fraction, 180–250 μm, for both materials.

The effect of packing intensity was studied by measuring the permeability of 60 cm powder beds which were tapped 0, 10 or 100 times on a tap volumeter (J. Engelsman, Ludwigshafen, F.R.G.) before the container was connected to the permeameter.

The effect of the height of the powder bed was studied by measuring the permeability of powder beds of three heights: 30, 45 and 60 cm. All powders were tapped 10 times in these cases.

The permeametry surface area of all powders were subsequently measured. In these experiments the height of the powder bed was 60 cm and the powders were packed by tapping 10 times. All results presented are mean values of three determinations.

#### Powder surface area by microscopy

All powders were photographed in an optical light microscope (Vanox Universal Research Microscope, Japan). Because of the narrow size distributions of the sieve fractions, samples were taken with a spoon. The projected area diameter was measured for approx. 100 particles with a particle size analyser (Zeiss TGZ3, F.R.G.). This number of particles was considered to be sufficient to obtain a good estimate of the mean particle size.

The surface to volume diameter for each size distribution was then calculated. From these data and from the Heywood surface to volume shape factors (Table 1), the specific surface areas of the powders were calculated (Allen, 1981c).

## Results and Discussion

#### Effect of experimental parameters on the permeametry surface area

The number of taps used for packing the powder prior to the permeability measurement did not

TABLE 2

*The effect of packing intensity on the specific surface area of saccharose and Emcompress powders*

Material	Number of taps	Surface area (cm <sup>2</sup> /g)	Coefficient of variation (%)	Porosity of powder bed	Time for air flow (s)	Reynolds number	
						Maximum <sup>a</sup>	Mean <sup>a</sup>
Saccha-rose	0	226	0.63	42.7	811.6	0.11	0.05
	10	229	0.99	40.5	1064.0	0.08	0.04
	100	226	0.16	40.5	1029.5	0.08	0.04
Emcom-press	0	236	0.60	57.2	427.0	0.18	0.08
	10	236	0.32	56.3	450.5	0.18	0.08
	100	237	0.30	54.9	510.5	0.14	0.07

<sup>a</sup> Maximum and mean pressure head.

affect the calculated surface area (Table 2). The porosity of the powder bed decreased slightly with increasing number of taps. A lower porosity corresponded to a longer air flow time through the powder bed and thereby a reduced Reynolds number. However, the calculated surface area was independent of the porosity variations. Wasan et al. (1976) have pointed out that there is often a tendency for the permeametry surface area to increase with reduction in bed porosity. This has been explained as a result of a variation in aspect factor with bed porosity. However, the results here indicate that there is no change in the aspect factor within the range of porosities obtained with the packing procedure used.

The bed porosity is lower for saccharose than for Emcompress. The microscope examination indicated that Emcompress particles had a rougher

surface texture than the other materials. This can lead to higher interparticulate friction in the mass and thereby a higher bed porosity after preparation of the powder bed. For Emcompress the bed porosity was calculated by using the granule density rather than the true density as discussed below.

The variation in height of the powder bed affected neither the calculated surface area nor the porosity of the powder bed (Table 3). As expected, the time for the air to flow through the powder bed is markedly decreased with reduced height of the powder bed, which reduces the Reynolds number. Consequently, the height of the powder bed can be varied to obtain suitable experimental conditions.

The coefficient of variation was below 1% in all cases for these initial experiments (Table 2 and 3).

TABLE 3

*The effect of height of powder bed on specific surface area of saccharose and Emcompress powders*

Material	Height (cm)	Surface area (cm <sup>2</sup> /g)	Coefficient of variation (%)	Porosity of powder bed	Time for air flow (s)	Reynolds number	
						Maximum <sup>a</sup>	Mean <sup>a</sup>
Saccha-rose	60	229	0.99	40.5	1064.0	0.08	0.04
	45	229	0.41	41.2	747.7	0.11	0.05
	30	226	0.75	40.3	540.5	0.16	0.07
Emcom-press	60	236	0.32	56.3	450.5	0.18	0.08
	45	237	0.70	56.7	334.4	0.23	0.11
	30	236	0.57	56.5	233.8	0.33	0.15

<sup>a</sup> Maximum and mean pressure head.

TABLE 4

*Specific surface areas and Reynolds numbers estimated by permeametry for all powders*

Material	Sieve fraction ( $\mu\text{m}$ )	Porosity of powder bed	Surface area ( $\text{cm}^2/\text{g}$ )	Reynolds number	
				Maximum <sup>c</sup>	Mean <sup>c</sup>
Saccharose	180– 250	40.5	229	0.08	0.04
	250– 355	41.0	165	0.24	0.11
	355– 500	42.3	116	0.79	0.36
	500– 710	44.4	89	2.29	1.05
	710–1000	46.3	74	4.95	2.26
Sodium chloride	180– 250	41.6	164	0.11	0.05
	250– 355	41.2	124	0.23	0.10
	355– 500	41.8	89	0.67	0.30
	500– 710	39.6	64	1.37	0.62
Glass pellets	200	40.0	163	0.06	0.02
	400	36.9	53	0.85	0.39
	500	40.8	53	1.67	0.76
Emcompress	180– 250 <sup>a</sup>	56.3	236	0.18	0.08
	180– 250 <sup>b</sup>	67.4	415		

<sup>a</sup> Granule density: 1.72.<sup>b</sup> True density: 2.31.<sup>c</sup> Maximum and mean pressure head.

TABLE 5

*Surface to volume diameters characterized by microscopy and calculated specific surface areas of all powders*

Material	Sieve fraction ( $\mu\text{m}$ )	Size range <sup>a</sup> ( $\mu\text{m}$ )	Median value ( $\mu\text{m}$ )	Surface to volume diameter ( $\mu\text{m}$ )	Surface area <sup>b</sup> ( $\text{cm}^2/\text{g}$ )
Saccharose	180– 250	240– 325	303	304	193
	250– 355	295– 440	380	400	147
	355– 500	435– 630	543	587	100
	500– 710	620– 805	720	739	79
	710–1000	810–1000	915	933	63
Sodium chloride	180– 250	205– 285	250	261	129
	250– 355	275– 385	345	357	94
	355– 500	385– 525	459	476	71
	500– 710	515– 665	609	624	54
Glass pellets	200	105– 185	153	168	140
	400	380– 580	457	465	50
	500	385– 525	441	472	50
Emcompress	180– 250	185– 245	218	228	207

<sup>a</sup> 10–90%<sup>b</sup> Powder surface area calculated from estimated values of surface to volume shape factors (see Table 1) and surface to volume diameters.

*Comparison between surface areas obtained by microscopy and permeametry*

The permeametry surface areas (Table 4) for most powders decreased with increased particle size. The exceptions were the two coarsest fractions of glass pellets which gave the same surface area. The microscopy data (Table 5) also indicated that particle size and shape were very similar for these qualities.

For Emcompress, surface areas calculated with both granule and true densities are given. For Emcompress, the use of the granule density gave lower calculated surface areas than when the true density was used. A comparison with microscopy data (Table 5) shows that the surface area based on granule density values gives a better agreement between the methods. It is probable that the diameter of the pores between the particles are much larger than the pores within the particles of Emcompress. It seems thereby reasonable that the air flows primarily in the spaces between the particles and that the porosity of this interparticulate space is better estimated by using granule density values. It has also been observed earlier (Wasan et al., 1976) that porous materials yield unexpected values for permeametry surface area.

In Table 4, the values of the Reynolds number calculated using Eqn 2 are given. The values for the smaller particles are generally below 2, whereas the coarser powders have Reynolds numbers exceeding 2, at least at the maximum head pressure, i.e. there might be a deviation from perfect laminar flow for these powders. However, the values indicate that the flow conditions through the powder beds are generally acceptable for the use of the Kozeny-Carman equation.

A good agreement is observed between the two types of surface areas as shown in Fig. 2 and Table 5. It is thus demonstrated that the permeability method gives a reasonable estimate of the external surface area of coarse particulate materials.

There is a general tendency for the permeametry surface areas to be slightly greater than the corresponding microscopy surface areas. Furthermore, the ratios between the surface area values (Table 6) vary among the materials but appear to be independent of particle size for each material.

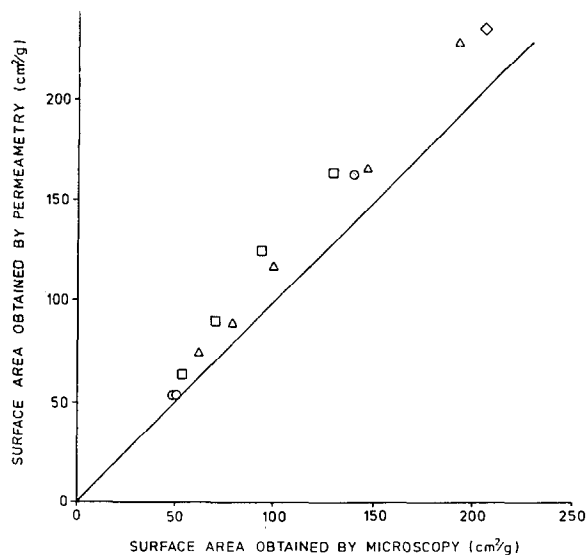


Fig. 2. The specific surface area of all powders obtained by permeametry and microscopy. A line of unit slope is drawn in the figure. ( $\Delta$ ) Saccharose, ( $\square$ ) sodium chloride, ( $\circ$ ) glass pellets and ( $\diamond$ ) Emcompress with granule density 1.72.

TABLE 6

*Ratio of permeametry surface area to microscopy surface area and values of aspect factor required to obtain a surface area ratio of unity*

Material	Sieve fractions ( $\mu\text{m}$ )	P/M <sup>a</sup>	Required aspect factor for P/M = 1
Saccharose	180- 250	1.18	7.0
	250- 355	1.13	6.3
	355- 500	1.16	6.7
	500- 710	1.12	6.3
	710-1000	1.18	6.9
Sodium chloride	180- 250	1.27	8.1
	250- 355	1.32	8.7
	355- 500	1.26	7.9
	500- 710	1.19	7.0
Glass pellets	200	1.16	6.8
	400	1.06	5.6
	500	1.07	5.6
Emcompress	180- 250	1.14	6.5

<sup>a</sup> Ratio of permeametry surface area to microscopy surface area. Permeametry surface area calculated with an aspect factor of 5.0.



These observations may be related to the variations in particle shape characteristics of the test materials, which will affect the surface areas from both microscopy and permeability measurements.

Except for some ideal materials, perfect spheres, cubes and cylinders, shape is a complex characteristic of a particle. Any deviations from a perfect principle shape, such as macro- and micro-surface irregularities, will affect the external particle surface area. The surface to volume shape factor used in this study describes a particle property, which can be denoted the geometrical or principle shape of the particle. This means that small surface irregularities are not taken into account in the value of the shape factor, which for some of the materials can give an underestimated external surface area based on microscopy.

The glass pellets represent particles with an almost perfect shape, i.e. smooth spheres, and the high surface area ratios for these powders suggest that when air permeability measurements of coarse particulate materials are used for surface area characterization, a modified aspect factor can be discussed. However, there are insufficient experimental data available to suggest an alternative value for the aspect factor.

During permeability measurements, particle shape variations between materials will affect the pore structure and thereby the calculated surface area. If an aspect factor is used which gives a surface area ratio of unity for perfect spheres, the surface area ratio for other materials will probably be different. It has been shown, for example, that the orientation of cylindrical particles can markedly affect the value of the aspect factor, i.e. values both below and above 5 have been reported (Orr and Dallavalle, 1959). The calculated powder surface area will thereby be affected if a standard value of the aspect factor is used. Shape characteristics which can be described as macro- and micro-surface irregularities will probably also affect the pore structure and contribute to the permeability of a powder bed and thereby the permeability surface area. This means that when microscopy is used as the reference surface area technique, a perfect agreement between the methods might not be theoretically expected due to the definition of the Heywood shape factor.

The aspect factor can thereby firstly be seen as a mean correlation coefficient between surface area data obtained by permeametry and by other surface area methods for a variety of powders. The present results suggest that in such a case a modification of the value of the aspect factor for coarse particulate materials is needed. However, the aspect factor can also be seen as a coefficient which correlates surface areas for powders, which consist of monodispersed, smooth spheres, i.e. the simplest and thereby the most ideal powder system concerning particle shape and packing characteristics. In such a case, discrepancies in surface areas when comparing microscopy and permeametry data for other non-ideal powders can reflect deviations in particle shape characteristics and particle size range from the ideal situation. Although materials with a narrow size range were used in this study, it is difficult to relate the degree of discrepancy to a definite particle shape characteristic. It is possible that the variation in surface area ratio is related primarily to deviations from the geometric shape, i.e. macro- and micro-surface irregularities. However, the method must be evaluated further in order to ascertain its usefulness concerning shape characterization of powders.

## Conclusions

By the use of a constant volume permeameter, the external surface areas of coarse particulate materials were measured and the generated data compared favourably with microscopy-derived data. The method is therefore applicable to rapid and routine determinations of the external surface area of such powders. However, to achieve acceptable flow conditions through the powder bed and thereby render the use of the Kozeny-Carman equation valid, the design of the powder container will be of particular importance. The limitations of the method with respect to the flow conditions for the calculation of powder surface area must be evaluated further.

The results also indicate that the properties of the individual particles can affect the relationship between surface area obtained by permeametry

and microscopy. If porous particles are characterized, it is important that a correct density value of the particles is used for the estimation of the effective porosity of the powder bed. The discrepancy between surface areas from microscopy and permeability measurements shows that the value of the aspect factor requires better determination. The possibility that this discrepancy reflects deviations in particle shape characteristics from the most simple shape of a particle, i.e. a perfect and smooth sphere, might be utilized for shape characterization of powders.

## Glossary

Symbol	Meaning
$A$	cross-sectional area of powder bed ( $\text{m}^2$ )
$g$	gravitational constant ( $9.81 \text{ m s}^{-2}$ )
$h_1$	height of stop point on manometer arm (m)
$h_2$	height of start point on manometer arm (m)
$h$	height of manometer liquid above equilibrium level (m)
$k$	aspect factor (5)
$L$	length of powder bed (m)
$L_e$	length of capillaries in the powder bed (m)
$R_e$	Reynolds number
$S_v$	powder surface area ( $\text{m}^2/\text{m}^3$ )
$t$	time for air flow through powder bed (s)
$V$	volume of air flowing through the powder bed ( $\text{m}^3$ )
$\epsilon$	porosity of powder bed
$\eta$	viscosity of air ( $1.81 \times 10^{-5} \text{ N s m}^{-2}$ )
$\rho_g$	density of air ( $1.2047 \text{ kg m}^{-3}$ )
$\rho_w$	density of manometer liquid (water) ( $1000 \text{ kg m}^{-3}$ )

## Acknowledgements

Lejus Medical AB and Pharmacia AB are gratefully acknowledged for their financial support of this work. We are grateful to Arne Kristensen

(Pharmacia AB) and Curt Appelgren and Kristina Eskilsson (Lejus Medical AB) for their contributions in our discussions. We wish to thank Elisabeth Börjesson for drawing the figure. We are also very grateful to Dr M.I. Barnett, Welsh School of Pharmacy, for valuable discussions of the manuscript.

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