Measurement of Powder Surface Area Using Capillary Elevation Method

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Summary

This contribution presents a neglected method of measuring powder surface area that uses the capillary elevation phenomenon. Such measurement has a potential to distinguish a fine surface texture of measured powder materials. The method has been verified experimentally on smooth steel spheres with diameters of 0.5 mm, 1.0 mm, 1.2 mm, and 1.6 mm, for which the theoretical values of specific surface area can be calculated and compared with the measurement results. The deviation of these experimentally determined values from the reference theoretical ones was less than 1%. New alternative experimental arrangements and related theoretical relations and simulations are presented.

Keywords: specific surface area, capillary elevation, powder materials, surface tension

Introduction

The intensity of the mutual interaction of two phases is directly proportional to their contact surface at their interface. In powder solid materials a relative size of this interface is represented by surface area that is usually related to a unit mass. The specific surface area depends on the surface texture (surface fractal dimension) and the size of granules [1, 2]. The granules in powder materials, having their origin in sequential mechanical fragmentation or in crystallisation from solution, follow a log-normal distribution [3]. The specific surface area parameter is a determining factor for the speed of a possible chemical reaction of these granules and the surrounding liquid, for effectiveness of heterogeneous catalysis, powder sorption capacity or other surface phenomena.

The question of definition of any material object surface is naturally related to a selected scale of structure resolution in a given surface area, as results from fractal geometry [2, 4]. In this connection three basic types of specific surfaces have to be distinguished from the point of view of practical applications.

The first one is so far generally used size of the standard specific surface area that includes fine surface texture and open pores. This quantity defines the solid material surface area as the area that can be fully covered by a continuous adsorbed monolayer of individual molecules of the reference gas adsorbate. The smallest characteristic dimension of such surface texture is, for example, the N2 molecule size, which is 0.34 nm. Measurement in this case is conducted with the BET isotherm method, which is based on the adsorption of gas molecules [5]. For practical determination of a specific surface area by the BET-isotherm method the BET analyzer is used.

The second definition of the specific surface area could be called the "geometric specific surface area" [6]. This approximation does not take into account any surface irregularities, but only the geometric shape of the granules. Such measurement may simply be performed by measuring of the granular size via optical microscopy, electron microscopy or light dispersion on the granules in laser analysers. Subsequent theoretical computations of specific surface area usually use spherical or ellipsoidal approximations. Technically more advanced methods are the Blaine method [7] or liquid permeability method [8, 9]. Both methods use dependence of surface area on gas/liquid permeability through the powder. The Blaine method can be effectively used for specific surfaces of 0.05−0.9 m^2g^{-1}. However, there is a significant problem during result interpretation of these methods, because the data have a more technical character due to the flow of gas along the deep concave pores. In spite of the mentioned limitations the Blaine method is the most practical one used today, primarily due to the simple measuring process and an easy maintenance of the equipment.

The third definition may be named "wet surface area" or "reaction surface area" using the terminology from literature [9, 10]. This definition takes into account the microscopic surface texture. Wetting of granules by liquid having a negligible angle of wetting results in creating a real phase interface between the granules and their environment. Beside the initial degassing of material the penetration of the wetting liquid to pores needs to be considered; this penetration is different from the penetration of isolated molecules of diluted gas in the case of the BET-analysis. However, in many actual technological processes the detailed texture parts and deep pores do not play a significant role and do not enter into mutual interaction with a liquid environment. The "wet surface area" is in these cases much more realistic practical parameter during chemical reactions of solid materials in liquids. The "wet surface area" is currently measured only via measurement of the dissolution heat [10-12]. The dissolution heat is released in a liquid during wetting of powder material and depends on the interface area. Practical use of this method, however, is limited to large surface areas, since if the size of a real surface is small, the temperature change of the suspension is very hard to measure with acceptable accuracy. For example BET method provides reliable results only for powder specific surface areas larger than 0.1 $m^2.g^{-1}$. Most non-porous powders with granules larger than 1 μm have the specific surface area lower than 1 m^2g^{-1}.

This work deals with the experimental method based on measuring of the capillary elevation in the column of a powder material. Foundations of this method were laid in the first half of 20th century, but the method has not been developed and there is a lack of experimental relevant data in literature [8, 9, 13-17]. Additionally, the goal of this work is to suggest new experimental arrangements and theoretical relations to the original one. These new designs are supported by theoretical simulations, on the basis of which more sorts of powder materials having large specific surface areas can be measured, including the sorts with granules having fine surface texture.

In the referred old works, the specific surface area was computed after capillary elevation measurement from equation

$$A^{sp} = \frac{\varepsilon h \rho g}{\sigma}, \qquad (\mathrm{cm}^2/\mathrm{cm}^3) \tag{1}$$

and after permeability measurement from equation

$$A^{sp} = 14\sqrt{\frac{\varepsilon^3}{K\upsilon \;\; 1-\varepsilon \;\;^{2}}}, \qquad (\mathrm{cm}^2/\mathrm{cm}^3) \tag{2}$$

where ε is the porosity, *h* is the elevation height, ρ is the density of liquid, *g* is the gravity acceleration, *K* is the permeability and υ is the kinematic viscosity of liquid. It is obvious that specific surface area A^{sp} does not have the same meaning in Eqs. 1 and 2 since the physical measurement process is different. The capillary elevation method, represented by Eq. 1, is sensitive for texture irregularities, whereas the quick permeability method is not. Unfortunately, experimental data in original works are primarily related to Eq. 2 [8, 13].

Theory

Derivation of specific surface area from the capillary elevation process

The formula that enables determination of the specific surface area of powder material through measuring the capillary elevation has been derived from force equilibrium between interface tension and liquid column gravity, where powder is located in a vertical tube (see Figure 1, for simplicity, the real granules are graphically represented by small spheres). The theoretical derivations performed are consistent with the general physical model of capillary elevation [18].

Selection of a specific liquid depends on its very good wetting capability and, at the same time, chemical inactivity toward the measured material. The space among the powder granules should be filled with an inert gas (Ar) after vacuum degassing. The measurement tube is slightly submerged under the surface of the liquid with its lower end in the wide reservoir. After submersion of the bottom tube opening the liquid starts to penetrate up to the powder column by means of the capillary force F_k. For calculation of the capillary force we need to know the total length of the wetting circumference *L*(*x*), as schematically shown in the Figure 2.

With moderate height *H* of the measured powder column we can assume a minimum influence of the static pressure on its bulk density ρB (mass of powder material/volume of powder material), and its size will be considered constant under the given conditions. Under this assumption the mean value of the total wetting circumference *L*(*x*), when circular circumference of tube wall is negligible, can be formally defined as

$$\bar{L} = \frac{1}{H}\int_0^H L \;\; x \;\; \mathrm{d}x \tag{3}$$

as well as the mean area of the free liquid level

$$\bar{S} = \frac{1}{H}\int_0^H S \;\; x \;\; \mathrm{d}x\,. \tag{4}$$

In cylindrical tube, the $\bar{S} = \pi r^2 \kappa$ where r is the radius of the tube and κ is the packing factor defined as $\kappa = \rho B/\rho G$, where ρG is true granule density (mass of granule/volume of granule). As a consequence of the constant bulk density ρB and the relatively chaotic arrangement of the granules, the fluctuations of both quantities around mean values can be neglected. The size of the capillary force $F k = |\mathbf{F} k|$ for the liquid with the surface tension σ and the small wetting angle θ is then expressed by the general relationship

$$F_k = \bar{L}\sigma\cos\theta\,. \tag{5}$$

Volume of the intergranular space flooded by liquid during its elevation is

$$V_{\mathrm{liq}} = \bar{S}h\,. \tag{6}$$

and the size of the weight force $G(h) = |\mathbf{G}(h)|$ of this column is

$$G(h) = V_{\mathrm{liq}}\rho g = \bar{S}h\rho g\,, \tag{7}$$

where ρ is the liquid density and g the gravity accel-

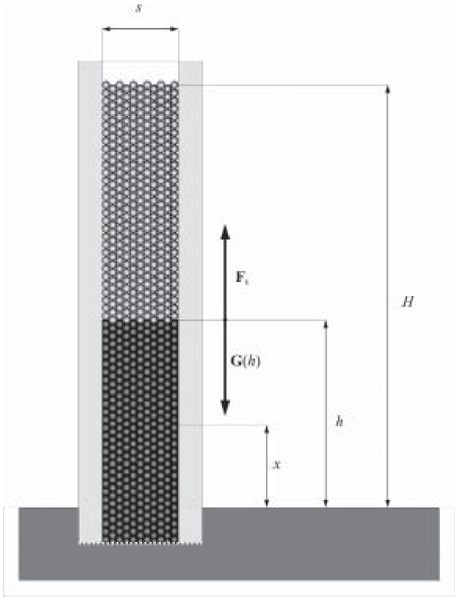

Fig. 1. The experimental set-up for the capillary elevation measurement in a powder material. The liquid level is stabilised in the height h above the reservoir level when the forces are equalised. The material is secured with a thin filtration membrane at the bottom.

Rys. 1. Odpowiednie przygotowanie materiału proszkowego do pomiaru kapilarnego. Poziom płynu jest stabilizowany na wysokości *h* powyżej poziomu wody w zbiorniku tak, aby działające siły się wyrównały. Materiał jest zabezpieczony na dnie cienką membraną filtracyjną

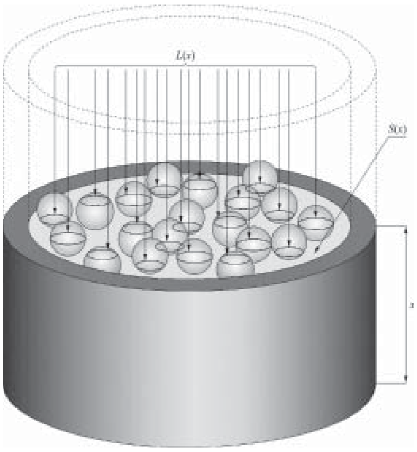

Fig. 2. The representation of the free area *S(x)* and the wetting circumference length *L(x)* of the liquid level in a general height *x* in the measuring column filled by spherical granules.

Rys. 2. Wolna przestrzeń *S(x)* oraz długość obwodu zwilżonego *L(x)* poziomu płynu na wysokości ogólnej *x* w kolumnie pomiarowej wypełnionej kulistymi granulkami eration. The total force balance of the equilibrium is apparent from the Figure 1,

$$\mathbf{F}_{\mathrm{k}} = \mathbf{G}(h). \tag{8}$$

Using Equations (5) and (7) leads to the relation

$$\bar{L}\sigma\cos\theta = \bar{S}h\rho g. \tag{9}$$

Then the following equation is valid for the mean liquid level circumference

$$\bar{L} = \frac{\bar{S}}{\sigma\cos\theta}h\rho g, \tag{10}$$

that allows to express the total surface area of all granules in the column with the height H,

$$S(H) = H\bar{L} = \frac{\bar{S}H}{\sigma\cos\theta}h\rho g. \tag{11}$$

Based on the knowledge of the granule density ρ_G, the bulk density ρ_B and the mass $m(H)$ in the column, we can calculate the flooded volume V_0 via relation

$$\bar{S}H = V_0 = \frac{m\ H}{\rho_{\mathrm{B}}} - \frac{m\ H}{\rho_{\mathrm{G}}}. \tag{12}$$

The specific surface area A^{sp} is defined by the ratio

$$A^{\mathrm{sp}} \overset{\mathrm{def}}{=} \frac{S(H)}{m(H)}, \tag{13}$$

which enables to write the final equation

$$A^{\mathrm{sp}} = \frac{1}{\sigma\cos\theta}\left(\frac{1}{\rho_{\mathrm{B}}} - \frac{1}{\rho_{\mathrm{G}}}\right)h\rho g. \tag{14}$$

Using this relationship we can calculate the specific surface area of powder in general case for the wetting angle θ and the elevation height h. In order to achieve the highest elevation effect (higher elevation enables a more accurate measurement), it is suitable to select a very well wetting specific liquid, for which the condition $\cos\theta \rightarrow 1$ can be used, and so the Eq. 14 can be rewritten more simply as

$$A^{\mathrm{sp}} = \frac{1}{\sigma}\left(\frac{1}{\rho_{\mathrm{B}}} - \frac{1}{\rho_{\mathrm{G}}}\right)h\rho g. \quad (\mathrm{m}^2/\mathrm{kg}) \tag{15}$$

For simulations see Figs. 3a and 3c. Uncertainty of A^{sp}, determined from Eq. 15, is a constant and equals 1/σ·(1/ρB – 1/ρG)ρg·uh, where uh is the uncertainty of liquid level determination, estimated to be ± 1 mm. In case of small surface area of powder, a measurement error caused by tube wall surface may occur. In this case, the term 2/(rκ)·(1/ρB – 1/ρG), which represents the specific surface area of the tube wall, needs to be substracted from Eq. 15. The Eq. 15 is also derived in original works in a different unit, see Eq. 1. Capillary elevation h in an open capillary tube may achieve a significant size for large values of surface area, and condition $h \geq H$ states the natural technical limit of such measurement. Limit value for the model bulk density spheres is bottom-limited by minimal space filling factor κ, for the close packed spheres in simple cubic structure it is

$$\left.\begin{aligned} \rho_{\mathrm{B}} &= \kappa\rho_{\mathrm{G}} \\ \kappa \geq \kappa_{\min} &= \frac{\pi}{6} \end{aligned}\right\} \quad \rho_{\mathrm{B}} \geq 0.524\rho_{\mathrm{G}}. \tag{16}$$

A maximal value of κ for the close packed spheres in hexagonal arrangement is 0.74 [19]. The Eq. 15 may be transformed using this condition (Eq. 16) to the form

$$A^{\mathrm{sp}} \leq \frac{\rho g}{\sigma\rho_{\mathrm{G}}}\left(\frac{1}{0.524} - 1\right)h. \tag{17}$$

which expresses the technical limit for measurement tube length. Disadvantage of the long tube is that a large amount of powder must be used in measurement, for example for sandstone (ρG = 2500 kg.m^{-3}, κ = 0.57) in a 2 m long tube having diameter 5 mm it is approximately 0.17 kg of powder. To save a sample amount, a thin tube may be used for very fine powder having higher specific surface area, since the tube wall effect should not play any role (does not affect the parameter L) in this case.

A new experimental arrangement for the elevation method

Such technical limitation of tube length may restrict the utility of this method for powders with larger surface area. However, the problem may be solved via upper closing of the tube. In this case, a new force F_2, caused by gas compression among the powder granules above the liquid level, appears in the tube, and force equation has the following form: $F_k = G(h) + F_2(h)$. Using the Boyle´s law for isothermic process in ideal gas, the Eq. 15 changes to the new form

$$A^{\mathrm{sp}} = \frac{1}{\sigma}\left(\frac{1}{\rho_{\mathrm{B}}} - \frac{1}{\rho_{\mathrm{G}}}\right)\left[h\rho g + p_{\mathrm{a}}\left(\frac{H}{H-h} - 1\right)\right] \quad (\mathrm{m}^2/\mathrm{kg}) \tag{18}$$

where p_a is the atmospheric pressure. The Eq. 18 is valid when the tube is fully filled with powder. Simulations with Eq. 18 for model powder materials, steel powder and sandstone powder, are presented in Figs. 3a and 3c. Uncertainty of A^{sp}, determined from Eq. 18, is not a constant and equals 1/σ ·(1/ρB – 1/ρG)[ρg + $p_a\{H/(H-h)^2\}$] ·uh, see Figs. 3b and 3d.

A simplification of measurement represented by Eq. 18 may be performed via preparation of a device, where the liquid level is depressed and maintained on level h = 0 m using gas overpressure Δp. The force equilibrium equation is then simply $F_k = F_2$. The new elevation equation is

$$A^{sp} = \frac{\Delta p}{\sigma}\left(\frac{1}{\rho_B}-\frac{1}{\rho_G}\right). \qquad (\mathrm{m^2/kg}) \qquad (19)$$

The measurement method is schematically presented in Fig. 4. Constant uncertainty of measurement of specific surface area must namely depends on Δp and equals $1/\sigma\cdot(1/\rho B - 1/\rho G)\cdot u_{\Delta p}$, where $u_{\Delta p}$ is estimated to be 10 Pa.

The Eqs. 18 and 19 represent a change in the measurement process, compare with Eq. 15 and original Eq. 1 presented in the original literature [8, 9, 13].

Elevation speed – duration of measurement

The total flow of liquid through the pores between granules can principally be, using simplification, divided to the sum of random parallel flows in partial profiles with the same instantaneous elevation height h. The general equilibrium Eq. 9 for the given elevation h is then equivalent to the similar equation for the same elevation h in a capillary with the inner radius R. By comparison of these equations for the same elevation value h we will obtain the size of the equivalent radius R of the capillary:

$$\left.\begin{aligned}\bar{L}\sigma\cos\theta &= \bar{S}h\rho g\\ 2\pi R\ \sigma\cos\theta &= \pi R^2\ h\rho g\end{aligned}\right\}\ R=\frac{2\bar{S}}{\bar{L}}. \qquad (20)$$

As a consequence of the Equations 11-13, the equivalent capillary radius R is then defined by the

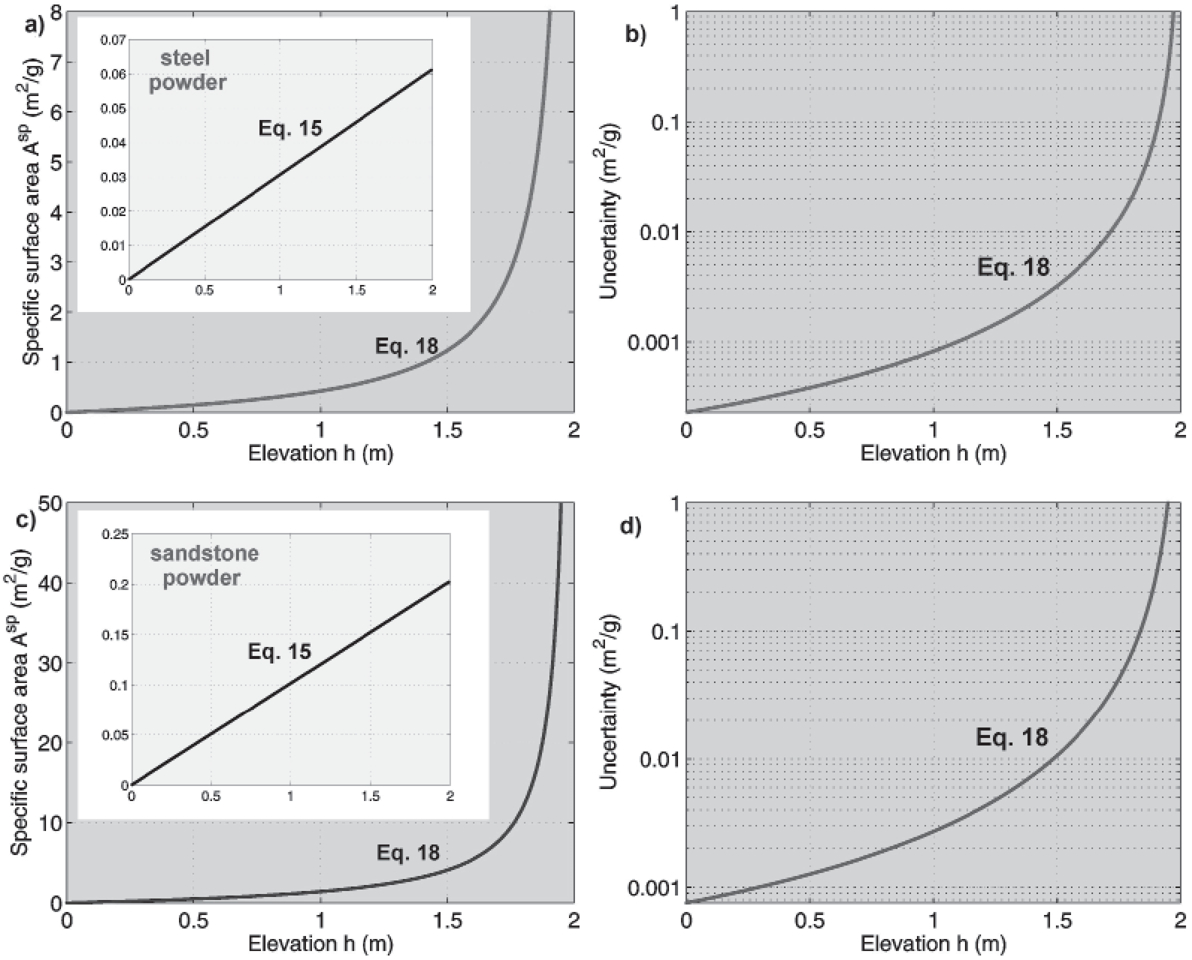

Fig. 3. Simulations of ethanol elevation through 2 m long vertical tube filled with powder material, using Eq. 15 or Eq. 18, model filling factor κ = 0.57. a) for hypothetical steel powder, ρG = 8307 kg.m^{-3} and b) its uncertainty derived from Eq. 18, where uh = 1 mm, c) for hypothetical sandstone powder, ρG = 2500 kg.m^{-3} and d) its uncertainty derived from Eq. 18, uh = 1 mm. Note: using the method represented by Eq. 18, in region of high elevation, the uncertainty in determination of the Aspe increases. Constant uncertainties determined from Eq. 15 are $3\cdot10^{-5}$ m^2/g for steel powder, and 10^{-4} m^2/g for sandstone powder respectively.

Rys. 3. Symulacja wznoszenia kapilarnego etanolu w rurze pionowej o długości 2 m, wypełnionej materiałem proszkowym zgodnie z równaniem 15 lub 18, współczynnik wypełnienia κ = 0.57, a) wypełnienie idealnym proszkiem stalowym o gestości ρG = 8307 kg.m^{-3}, b) błąd określony według równania 18, gdzie uh = 1 mm, c) dla idealnego piasku dla którego ρG = 2500 kg.m^{-3}, d). błąd określony wg równania 18, uh = 1 mm.Uwaga: zastosowanie obliczeń zgodnie z równaniem 18, w obszarze o dużej zmianie wysokości słupa etanolu powoduje zwieszenie błędu określenia Aspe. Błąd określony wg równania 15 wynosi dla proszku stalowego $3\cdot10^{-5}$ m^2/g i dla piasku 10^{-4} m^2/g parameters A^{sp}, ρB, and ρG:

$$R = \frac{2}{A^{\mathrm{sp}}}\left(\frac{1}{\rho_{\mathrm{B}}} - \frac{1}{\rho_{\mathrm{G}}}\right). \tag{21}$$

The vertical liquid movement in the space between granules can be represented by the equivalent movement in the axis of just one capillary with the radius R, and the time when equilibrium is reached can be determined. According to the Hagen-Poiseuille law [20] for the laminar volumetric flow of liquid with the viscosity η inside of a capillary with the radius R and length h, the following equation is valid:

$$\frac{\mathrm{d}V}{\mathrm{d}t} = \frac{\pi R^4}{8\eta}\frac{\mathrm{d}p}{\mathrm{d}h}. \tag{22}$$

The driving force of the elevation is a pressure gradient $\mathrm{d}p/\mathrm{d}h$ that corresponds to the difference of capillary and hydrostatic pressures in the column with the length h,

$$\frac{\mathrm{d}p}{\mathrm{d}h} = \frac{1}{h}\left(\frac{2\sigma\cos\theta}{R} - h\rho g\right). \tag{23}$$

If we consider the volume differential in the cylindrical shape $\mathrm{d}V = \pi R^2 \mathrm{d}h$, then the Hagen-Poiseuille differential equation has the specific form

$$\frac{\mathrm{d}h}{\mathrm{d}t} = \frac{R}{8\eta}\left(\frac{2\sigma\cos\theta}{h} - R\rho g\right). \tag{24}$$

The time progression of the elevation on the Figure 5 was determined by the solution of Eq. 24.

Experimental

Validity of the elevation method

The arrangement on the Figure 1 was used for the measurement of small steel sphere surface areas

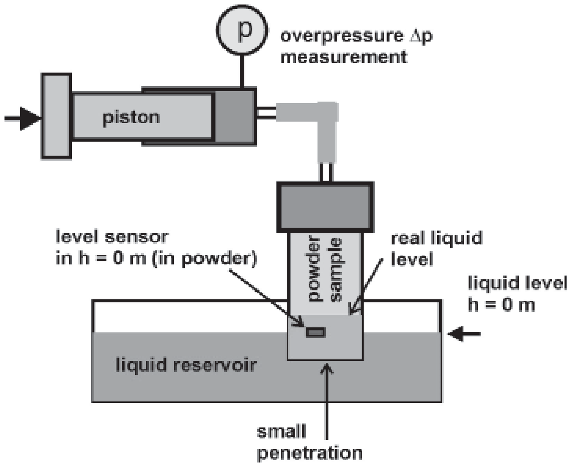

Fig. 4. A schematic arrangement of the method related to Eq. 19.

Rys. 4. Schematyczny układ metody przedstawionej w równaniu 19.

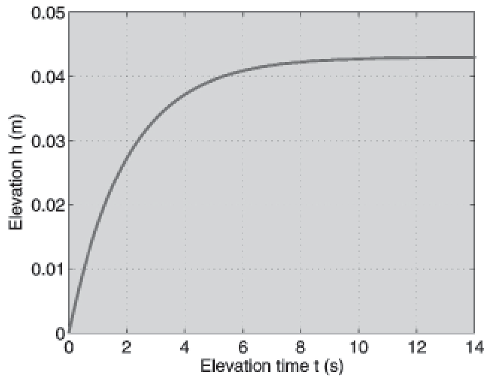

Fig. 5. The example of the theoretical time dependency of the capillary elevation h → 0.043 m in the vertical capillary with the radius R = 134 μm, see Eq. 21. The time dependency is equivalent to the elevation in pores between steel spheres with diameters 2r = 0.5 mm with the bulk density ρB = 4595 kg.m^{-3}. The force equilibrium according to Eq. 8 is achieved in a smooth and straight capillary in approx. 14 s.

Rys. 5. Przykład teoretycznej zależności wzniesienia kapilarnego od czasu dla wysokości h → 0.043 m w pionowej kapilarze o promieniu R = 134 mikrometrów (patrz równanie 21). Wznioszenie kapilarne jest równoważne wznoszeniu między kulkami stalowymi o średnicy 2r = 0,5 mm, o gęstości nasypowej ρB = 4595 kg.m^{-3}. Równowagę sił uzyskuje się (według równania 8) w gładkiej i prostej kapilarze w czasie ok. 14 s.

with the diameters 0.5 mm, 1.0 mm, 1.2 mm, and 1.6 mm. The resulting experimental values were subsequently compared to the theoretical calculated values for smooth spheres and in the end also with the specific surface area determined by the BET method.

The cylindrical measurement tube with the inner diameter 6 mm was immersed to the reservoir with ethanol. The material in the tube was degassed in vacuum (pressure approx. 0.1 hPa) before the measurement lasting for about 20 min. The elevation measurement was performed immediately after taking the tube out of the vacuum recipient. A wide Petri dish, whose area has been significantly larger than the profile of the measurement tube, served as the reservoir, and the change of a level during elevation was then negligible (the free level was covered by a free lid during the measurement in order to prevent significant evaporation of the measuring liquid). The total time of the spontaneous capillary rise and temperature stabilization was approximately 6 hours based on the first experimental experience. Such long elevation is somewhat problematic from the practical point of view, and the possibility of its shortening should be verified.

The time to achieve the force equilibrium, determined from Eq. 24, is significantly shorter than our first experimental experience with the capillary rise on measured spheres. The reason for these different results can be found in imperfect wetting due to weak degassing, presence of filtration paper at the bottom of the powder column which we used, and especially in the labyrinth of pores among granules that prevents creation of the direct vertical flow of liquid. The dynamic flow of liquid through the porous media (powder material) is originally

Tab. 1. The theoretical and measured specific surface area of the mono-dispersion steel spheres with the diameters 0.5 mm, 1.0 mm, 1.2 mm, and 1.6 mm.

Tab. 1. Wyliczona i zmierzona wartość powierzchni właściwej dla kul stalowych o średnicy 0.5 mm, 1.0 mm, 1.2 mm, and 1.6 mm.

Sphere diameter	Measured elevation	Average elevation	Measur. Eq. (15)	Theory Eq. (25)	Relative deviation
D	h_i	h	A^{sp}	A^{sp}_{teor}	$\left\| 1-A^{sp}_{teor}/A^{sp} \right\| \cdot 100$
(mm)	(mm)	(mm)	(10^{-4} m^2/g)	(10^{-4} m^2/g)	(%)
0.5	43; 49; 47	46.3	14.5	14.45	0.7
1.0	22; 23; 23	22.7	7.17	7.22	0.6
1.2	19; 18; 19	18.7	5.96	6.02	1.0
1.6	14; 14; 13	13.7	4.47	4.51	0.9

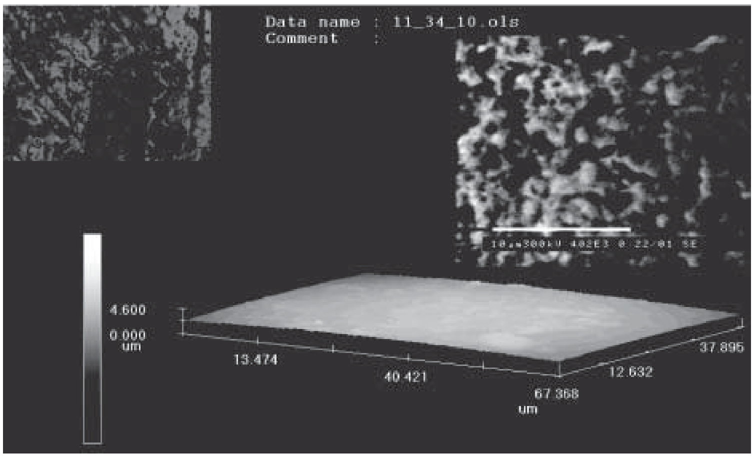

Fig. 6. The surface texture of the steel sphere with the diameter 1.6 mm depicted by the confocal microscopy, and in the upper right picture the detail of surface undulation in the regime of secondary electrons of the SEM. Based on the picture, it is possible to conclude that a possible fractal surface dimension on the scale up to 10 μm will be very close to the value of 2, which corresponds to a smooth plane.

Rys. 6. Struktura powierzchni kuli stalowej o średnicy 1,6 mm, obraz uzyskany w mikroskopie konfokalnym. W prawym górnym rogu rysunku widać szczegół powierzchni (określono za pomocą SEM). Na podstawie rysunku, można stwierdzić, że możliwy wymiar powierzchni fraktalu w skali do 10 μm jest zblizony do wartości 2, co odpowiada powierzchni gładkiej.

described in ref. [21] and advanced analyses were done by Kozeny and Carman, whose effort resulted in Kozeny-Carman equation [13, 17, 22]. In Kozeny-Carman equation, the liquid volume flow depends on a specific surface area of powder, porosity and a specific constant.

The mentioned experimental problem, however, was in case of our measurement with steel spheres very satisfactorily solved through insertion of the liquid reservoir to the ultrasound field with the intensity of approximately 80 mW.cm^{-3} during the measurement. The activation of liquid with ultrasound led to significantly shorter time to reach the equilibrium - approximately 2 minutes - with relatively well reproducible results.

The steel spheres with diameters 0.5 mm, 1 mm, 1.2 mm, a 1.6 mm have the same granule density ρG = 8307 kg.m^{-3} and the measured bulk density values during the measurement itself inside of the tube are as follows:

ρB(0.5) = 4727 kg.m^{-3} (κ = 0.569),
ρB(1.0) = 4705 kg.m^{-3} (κ = 0.566),
ρB(1.2) = 4686 kg.m^{-3} (κ = 0.564),
ρB(1.6) = 4638 kg.m^{-3} (κ = 0.558),

where κ is the coefficient presented in Eq. 16. All spheres show relatively very small deviation from sphericity (see the Fig. 6). Therefore, a smooth spherical plane was selected as a good approximation of the real surface texture for the theoretical calculation.

The theoretical value of the specific surface of a smooth sphere with the granule density ρG and the radius *r* (or diameter *d*) is given by the following equation:

$$A^{\mathrm{sp}}_{\mathrm{teor}} = \frac{4\pi r^2}{\rho_{\mathrm{G}} 4\pi r^3 / 3} = \frac{3}{\rho_{\mathrm{G}} r} = \frac{6}{\rho_{\mathrm{G}} d}. \qquad (25)$$

The theoretical values of the specific surface $A^{\mathrm{sp}}_{\mathrm{teor}}$ according to the Eq. 25 and the measured value A^{sp} according to the Eq. 15 for steel spheres are shown in the Table 1.

The measurement of the elevation height hi was performed three times. Uncertainty in individual measurements, determined as $dA^{sp}/dh \cdot (uh^2 + s^2)^{0.5}$, where s is the standard deviation of three measurements, did not exceed 10% of A^{sp} value. The relative deviation of the experimental values A^{sp} from the calculated ones A^{sp}_{teor} did not exceed 1%. For comparison, we have performed the measurement of the specific surface area of the spheres with the diameter 1.2 mm by the BET analysis. The value A^{sp}BET = 0.04·10−4 m^2/g differs from the theoretical value for smooth spheres $A^{sp}_{teor} = 6 \cdot 10^{-4}$ m^2/g significantly. The main impact on the final result of the BET measurement may be exerted also by the relatively low adsorption and desorption intensity on a small surface area, which can significantly influence the accuracy of determination of the measured BET-isotherm.

Conclusion

This paper theoretically formulates and experimentally tests a method for the measurement of specific surface area of powder materials. The method is based on utilisation of the capillary elevation phenomenon of a well wetted liquid in the space among the measured powder granules. A very good agreement between the theoretical and experimental values has been reached during the measurements of the tested materials in the form of small steel spheres with the diameters 0.5 mm, 1.0 mm, 1.2 mm, and 1.6 mm. The deviation among them was less than 1% for all dimensions. The technical limitations of the basic experimental design to the very small values of the specific surface area will be possible to eliminate applying additional well measurable forces depressing the elevation. With regard to the above mentioned definition of the so called “wet surface area”, the method of capillary elevation can be considered as an addition to the method of dissolution heat or Blaine method, primarily for non-porous but texture-rich powder materials, which could be penetrated through liquid in real time. Such penetration can be supported by ultrasound energy in case of capillary elevation through very fine powder granules. Further analyses should be focused on validity of the method, evaluated according to the measurement of capillary elevation through powder materials with granules having non-smooth surfaces.

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Pomiar powierzchni właściwej proszku przy użyciu metody kapilarnego wzniesienia

Artykuł poświęcony jest zaniedbanej metodzie pomiaru powierzchni właściwej proszku przy użyciu zjawiska kapilarnego wzniesienia. Taki pomiar jest w stanie rozróżnić miałką teksturę badanego proszku. Metoda została sprawdzona przez przeprowadzenie eksperymentu na gładkich stalowych kulach o średnicach 0,5 mm, 1,0 mm, 1,2 mm, oraz 1,6 mm, dla których można wyliczyć teoretyczne wartości powierzchni właściwej i porównać z wynikami pomiaru. Odchylenie wartości uzyskanych podczas eksperymentu w relacji do wartości teoretycznych wyniosło mniej niż 1%. Praca prezentuje nowe alternatywne metody eksperymentalne wraz z powiązaną teorią i symulacją.

Słowa kluczowe: powierzchnia właściwa, kapilarne wzniesienie, materiały proszkowe, napięcie powierzchniowe