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UNCERTAINTY IN DETERMINING THE APPARENT SPECIFIC SURFACE AREA OF PARTICULATE MATTER BASED ON THE POWER LAW MODEL

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Abstract: The paper analyzes the uncertainty of the determination of the apparent specific surface area (SSA) of particulate matter using the power model of particle size distribution. It compares this method with selected experimental procedures in terms of measurement uncertainty. It shows that the presented measurement uncertainty is higher due to the sensitivity of the computational model to the input parameters, therefore the method is particularly suitable for rapid estimates of the SSA of simple non-porous particles of particulate matter.

Keywords: particulate matter, apparent surface area, power model, measurement uncertainty

1 Introduction

In process engineering, particulate matter is understood as material composed of individual solid particles (granules, agglomerates) that form a separate phase or are dispersed in another medium (gas or liquid) [1]. These are therefore substances that occur in the form of particles of various sizes, and the behavior of the entire system is determined by the properties of these particles.

Several key parameters are monitored for particulate matter that affect its behavior during production and technological processes. These include the size of solid particles and their distribution (granulometry), the shape and structure of solid particles, density, surface properties, bulk properties. These parameters are used in the design and optimization of processes such as mixing, grinding, granulation, filtration, fluidization or pneumatic transport of solid particles [2], [3], [4].

When designing technological processes with particulate matters, it is often necessary to know their *specific surface area*, SSA. It is a key parameter describing the size of the active surface of particles relative to their mass or volume [5]. Its knowledge is important, for example, in assessing the reactivity of substances. It allows better control of the properties of materials and their functional use in technologies. In practice, it is used, for example, in the design of catalysts, adsorbents (e.g. activated carbon) or pharmaceutical powders, where it affects the solubility and bioavailability of drugs. In civil engineering, the SSA of cement affects the hydration and strength of the material. In environmental applications, it helps to optimize the removal of contaminants.

In any case, the methods for determining SSA are based on the measurement of certain physical quantities, the values of which are used to calculate SSA using functional relationships. It follows that, as with all measurements, the determination of SSA is also burdened by measurement uncertainty. So, for a correct evaluation of a measurement, it is not enough to

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state only an estimate of the value of the measured quantity, in our case SSA, but the corresponding measurement uncertainty must also be stated.

In this paper, we compare the uncertainty of determining SSA using a power model of particle size distribution (PSD) with the uncertainty of other selected methods suitable for the same group of particulate matters.

2 Methods for determining the specific surface area of particulate matters

There are several groups of methods that can be used to determine the SSA of particulate matter. They differ in their complexity, achieved measurement uncertainty, instrumentation requirements, etc. These include:

1. adsorption methods – belong to the most important, including the BET (Brunauer – Emmett – Teller) method, the Langmuir adsorption method and the pore analysis method,
2. porosimetry – this includes mercury intrusion porosimetry (MIP),
3. permeability methods – this includes the Blaine method / gas permeability,
4. geometric and granulometric methods – this is an indirect calculation using particle size,
5. microscopic and imaging methods – this includes, for example, image analysis,
6. chemical and sorption methods – used at room conditions,
7. other modern methods – for example, scattering techniques, inverse gas chromatography (iGC), X-ray and spectroscopic methods.

However, it should be noted that the individual methods for determining SSA are not completely compatible, because they do not determine the same parameter. The so-called envelope SSA is determined using permeability methods, BET can be used to determine the so-called true SSA including microtexture, while granulometry will provide a modeled SSA based on certain assumptions about the particle shape.

In this paper, we will discuss the determination of SSA based on the power model of particle size distribution. This is one of the geometric and granulometric methods for determining SSA, that can be employed for the following groups of particulate matter:

1. powders with relatively simple particle shape – these are spherical or approximately spherical particles (equiaxed particles), or slightly irregular particles that can be approximated by an equivalent sphere. For example, these are metal powders (atomized powders), glass beads, some ceramic powders,
2. non-aggregated, non-porous (compact) particles - particles without internal pores and without significant surface roughness,
3. dispersion systems with a relatively narrow size distribution - monodisperse or slightly polydisperse systems,
4. coarser powders (micrometer range), especially suitable for medium and coarse fractions, for example construction materials (cement, sand - coarse fractions), metallurgical powders, pharmaceutical granulates,
5. dry, loose materials, especially powders, granules and loose solids, which are commonly used in the food industry, pharmacy, powder metallurgy.

In addition to granulometric and geometric methods, the following can also be used to determine the SSA of the above groups of particulate matter:

1. methods based on gas permeability (air/gas permeability) [6],

2. adsorption methods (BET and related) [7],
3. methods based on liquid adsorption [8],
4. methods based on density and volume measurements (pycnometry + calculations) [9],
5. microscopic methods (image-based SSA) [10],
6. dynamic scattering and optical methods (indirect SSA) [11],
7. electrokinetic and surface methods [12].

In the following chapter, we will first compare selected methods for determining the SSA of the above groups of particulate matters from the perspective of the generally achieved relative expanded measurement uncertainty U_r.

3 Uncertainty of SSA measurement using selected methods

This chapter analyses the measurement uncertainty of SSA for selected methods of its determination. The individual methods differ not only in the measurement principle, but also in the general achieved relative measurement uncertainty U_r and sensitivity to input quantities. The uncertainty of the result arises because of the propagation of uncertainties of the measured and model parameters used in the calculation of SSA.

Uncertainty of SSA measurement using the Blaine permeability method

Blaine permeability method is suitable for measuring the properties of certified reference materials, CRMs. It is a method that provides very low measurement uncertainty. As an example, for NIST reference measurements, the relative expanded measurement uncertainty U_r is around 0.7%. For instance, the NIST certified SSA value for the certified reference material SRM 46h – Portland Cement Fineness Standard –is (364.4 ± 2.68) m²/kg with a coverage factor $k = 2$. That gives a relative expanded measurement uncertainty U_r of around 0.73% [13]. For common laboratory Blaine methods, U_r is around 2% to 5%.

Uncertainty of SSA measurement using BET

In this method, the uncertainty of SSA arises from the propagation of uncertainties in the input quantities of the adsorption measurement. These uncertainties originate from the following areas:

a. sample preparation – this includes sample degassing (temperature, time, vacuum), residual moisture or contamination of the sample, sample phase change,

b. measurement of the sample mass – this includes sample mass measurement errors,

c. measurement of the adsorbed amount of gas – this includes errors associated with volume calibration (dosing volume), pressure gauge errors, especially for low pressures,

d. temperature measurement in cryogenic conditions,

e. model and calculation errors – these include the choice of the linear BET interval (P/P_0), the assumption of an ideal gas, the choice of the cross section of the adsorbate molecule,

f. material factors of the sample - surface heterogeneity, pore availability, adsorption kinetics.

The relative expanded uncertainty of SSA determination using the BET method, $k = 2$, is approximately in the range of 5% to 10% (typically). Under poor conditions (e.g. low sample mass, poor sample degassing) it is 10% to 20%. For example, NIST reports SSA values for a

certified reference material in its certificate of analysis of a sample as (10.99 ± 0.68) m²/g, which results in a relative expanded uncertainty U_r of approximately 6% [14].

Uncertainty of SSA measurement using methods based on density and volume measurements

Even in the case of determining SSA using a combined method (pycnometry and calculation from the particle size of the particulate matter), it is an indirect measurement. The magnitude of the resulting measurement uncertainty is significantly influenced by the uncertainty of the particle size determination and the model assumptions, while the contribution of pycnometry is relatively small.

The most significant sources of measurement uncertainty are:

a. particle size determination (granulometry) – this includes selection of a representative sample, dispersion /agglomeration, particle shape model, statistical variability of distribution. Typical relative expanded uncertainty from this source is approximately 1% to 10%,

b. model assumptions – assumption of spherical particle shape and homogeneous size, while real particles are three-dimensional objects with irregular shape,

c. density determination (pycnometry) – mass measurement, volume measurement, temperature measurement, pressure (less significant with a gas pycnometer). Typical relative expanded uncertainty from this source is approximately 0.05% to 0.2%.

Based on the measurement uncertainty of the input quantities, it can be stated that the typical relative expanded uncertainty U_r of SSA ($k \approx 2$) is approximately 5% to 20%. Where:

- for well-characterized powders (spherical, narrow distribution), U_r is approximately 5% (lower limit),
- U_r reaches approximately 10% to 20% for real technical powders.

4 Calculation of SSA using the power model of particle size distribution

To demonstrate the calculation of the uncertainty of SSA measurement using the power model of particle size distribution in the following text, let us first present the relations by which the SSA estimate can be calculated. First, we present the power model itself, i.e. the relation between the particle size x and the fraction of particles in the particulate matter smaller than and equal to x. The derived relations will be used to calculate the estimates of the output quantities, i.e. the parameters k, α and the apparent specific surface area, and at the same time they will serve as functional relations that will be used to calculate the uncertainties of these estimates using the law of propagation of uncertainties. Then we use this model to calculate SSA.

4.1 Relationship between particle size *x* and the fraction of particles smaller than *x*

The relationship between particle size x and the fraction of particles smaller than x is implicitly derived in [15], [16], [17], [18]. In the literature, it is called *the particle size distribution function* (PSD) and is derived from the cumulative form of the power model of particle size distribution. It has the form

$$D(x) = \left(\frac{x}{k}\right)^{\alpha} \tag{1}$$

where

$D(x)$ is the cumulative percentage, i.e. proportion of particles smaller than or equal to x, in sieve analysis it is determined as the retained fraction,

x is the particle size, in sieve analysis it is equal to the size of the sieve opening,

k is a characteristic dimension (so-called scaling parameter),

α is the PSD exponent.

The parameters k, α can be determined by values of x and $D(x)$, which are known at two points.

Determining the value of $D(x)$ for a given x using sieve analysis

One way to determine the value of $D(x)$ for a given x is the sieve analysis mentioned above. In this method, we determine $D(x)$ by measuring the masses m_i of retained fraction at a sieve with an opening size $x_{si,i}$, belonging to a system of sieves. Based on the measured masses of individual retained fractions on the sieves, the cumulative percentage $D_{si,i}$ on a given sieve i with an opening size $x_{si,i}$ can be determined (see Fig. 1). In this case

- it is assumed that a system of s sieves will be used, the parameter i indicates the serial number of the sieve, $i = 1, 2, ..., s$,
- the solid bottom of the sieve system has a serial number of $s+1$,
- the size of the opening of a given sieve is denoted by $x_{si,i}$,
- the mass of the retained fraction of the examined particulate matter on a given sieve (or on the bottom) is m_i,
- the cumulative percentage on sieve i is $D_{si,i}$.

The value of the cumulative percentage $D_{si,i}$ is calculated as the ratio of the sum of the masses of the retained fractions under the sieve i with the opening size $x_{si,i}$ and the total mass m_c, summing the mass of all fractions, thus:

$$D_{si,i} = \frac{\sum_{i+1}^{s+1} m_i}{m_c} \tag{2}$$

where

$D_{si,i}$ is the cumulative percentage passing through a sieve i with opening size $x_{si,i}$ (see Fig. 1).

The total mass m_c of all fractions represents the sum of the masses of individual fractions m_i, measured on individual sieves i

$$m_c = \sum_{i=1}^{s+1} m_i \tag{3}$$

Determining the parameters of the power model

Relation (1) is valid for any pair D, x of a given matter. It can therefore also be used to calculate the unknown parameters k, α. First, we express relation (1) in logarithmic form

$$lnD_{si} = \alpha \cdot (lnx_{si} - lnk) \tag{4}$$

After substituting the experimentally obtained values from two known points xsi,i, Dsi,i, xsi,j, Dsi,j, where i, j = 1, 2, ..., s and i ≠ j, we obtain two equations with two unknown parameters in the form (4). Based on them, we get the sought coefficients

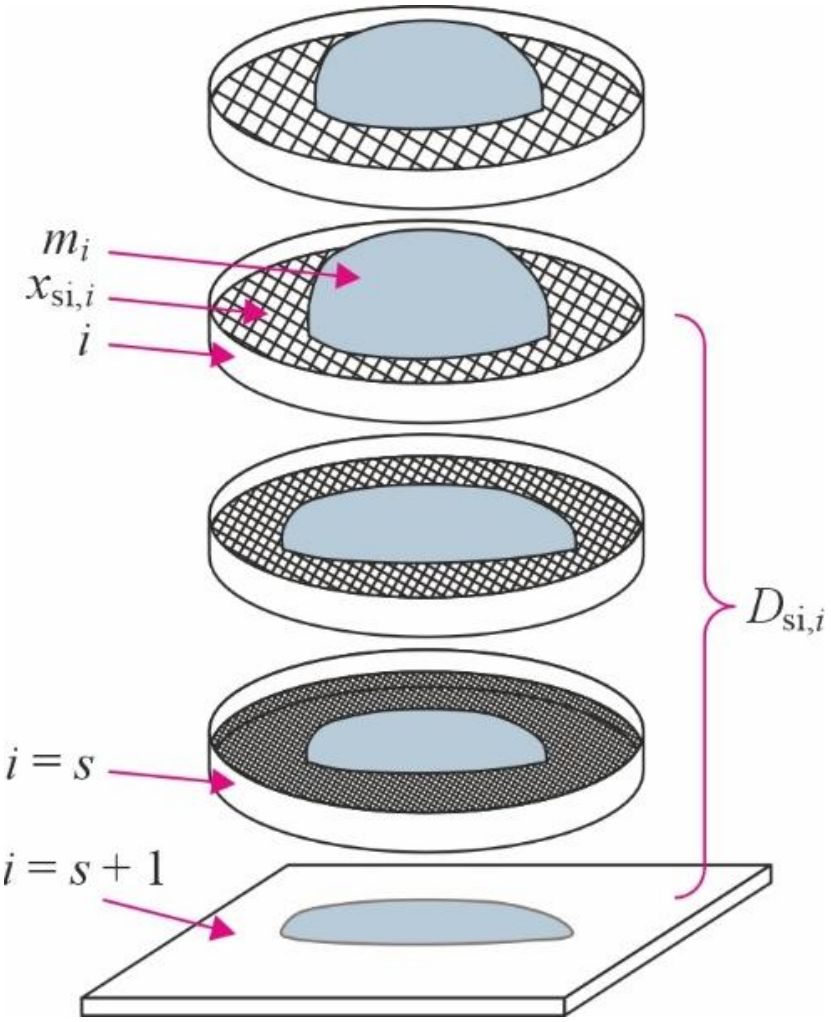


Fig. 1 Schematic designation of the sieves

$$k = e^{p} \tag{5}$$

$$\alpha = \frac{lnD_{si,i}}{lnx_{si,i} - p} \tag{6}$$

where p is a substitution

$$p = \frac{lnD_{si,i} \cdot lnx_{si,j} - lnD_{si,j} \cdot lnx_{si,i}}{lnD_{si,i} - lnD_{si,j}} \tag{7}$$

4.2 SSA calculation based on the power model for particle size distribution

The relationship for the apparent specific surface area a_z of a particulate matter, based on the power model (1) of the particle size distribution, has the form [19], [20]

$$a_z = \frac{6 \cdot \alpha \cdot \varkappa}{k^{\alpha} \cdot (\alpha - 1)} \cdot (x_{max}^{\alpha-1} - x_{min}^{\alpha-1}) \tag{8}$$

in which, in addition to the parameters appearing in relation (1), there are also

a_z is the apparent specific surface area (SSA) sought,

$\varkappa$ is a normalization constant. For spherical particles it is equal to 1, which will be used in sample calculation,

x_{min} is the minimum particle size of the particulate matter,

x_{max} is the maximum particle size of the particulate matter,

6 is the derived geometric factor for spherical particles.

This is an integral calculation of the apparent specific surface area of a material with a power model distribution of particle sizes in the interval $\langle x_{min}, x_{max}\rangle$. Integration in the interval $\langle x_{min}, x_{max}\rangle$ ensures that the contribution of all particle size classes is included in the calculation.

Equation (8) is based on the

- calculation of SSA from PSD (see also [21]),
- integration of distribution moments (see also [22]), and
- particle size distribution using a power model (e.g. [18]), considering the spherical geometry of the particles of the particulate matter.

The lower limit x_{min} and the upper limit x_{max} of the particle size of the particulate matter can be derived from the condition of the minimum and maximum cumulative percentage D corresponding to these particle sizes. We purposely chose $D(x_{min}) = 0.01$ and $D(x_{max}) = 1$. In this case, we can express the values of x_{min}, x_{max} from the following relations:

$$D(x_{min}) = 0{,}01 = \left(\frac{x_{min}}{k}\right)^{\alpha};\ D(x_{max}) = 1 = \left(\frac{x_{max}}{k}\right)^{\alpha} \tag{9}$$

5 Measurement uncertainty of output estimates

According to the International Dictionary of Metrology (VIM), see [23], the result of a measurement is generally expressed as a single measured value of a quantity (an estimate of the quantity value) and the uncertainty of the measurement (see Article 2.9). At the same time, VIM in Article 2.26 defines the uncertainty of measurement as a *non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used.* Article 2.32 states that the relative standard uncertainty of measurement is the *standard measurement uncertainty divided by the absolute value of the measured quantity value.*

The basic procedures for calculating measurement uncertainty are defined in an international document [24]. In general, an analytical method using the law of propagation of uncertainties or a numerical Monte Carlo simulation using the law of propagation of distributions can be used.

The law of propagation of uncertainties defines that for uncorrelated (unrelated to each other) input quantities X_1, X_2, ..., X_N, the combined measurement uncertainty $u(y)$, associated with the estimate y of the measured quantity Y, is obtained by combining the standard uncertainties $u(x_1)$, $u(x_2)$, ..., $u(x_N)$, associated with the estimates x_1, x_2, ..., x_N of the input quantities X_1, X_2, ..., X_N:

$$u^2(y) = \sum_{i=1}^{N} \left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i) \tag{10}$$

where f is a function defining the relationship between the measured quantity Y and the input quantities $X_1, X_2, ..., X_N$, i.e. $Y = f(X_1, X_2, ..., X_N)$. For example, in the case of relation (3), $D = f(x, k, \alpha)$, so the function f defines the relationship between the output quantity D and the input quantities x, k, α.

Partial derivatives of the function f in relation (10) are also called *sensitivity coefficients* and are often denoted by the letter A. They are evaluated using estimates x_1, x_2, ..., x_N of the input quantities X_1, X_2, ..., X_N:

$$A_i = \frac{\partial f}{\partial x_i} = \frac{\partial f}{\partial X_i}\Big|_{x_1, x_2, \ldots, x_N} \tag{11}$$

In the following text, we will use equation (10) to calculate the relevant measurement uncertainties, including the measurement uncertainty of the apparent specific surface.

5.1 Uncertainties of power model parameters estimates

The law of propagation of uncertainties in the form (10), together with the calculation of sensitivity coefficients in the form (11), is applied to relations (5), (6), i.e. to the relations defining the calculation of parameter estimates k, α. It holds that $k = f_k(x_{si,i}, x_{si,j}, D_{si,i}, D_{si,j}), \alpha = f_\alpha(x_{si,i}, x_{si,j}, D_{si,i}, D_{si,j})$.

First, we obtain the relation for calculating the uncertainty $u(k)$ of the parameter k

$$u^2(k) = A^2_{k,x_{si,i}} u^2(x_{si,i}) + A^2_{k,x_{si,j}} u^2(x_{si,j}) + A^2_{k,D_{si,i}} u^2(D_{si,i}) + A^2_{k,D_{si,j}} u^2(D_{si,j}) \tag{12}$$

The relevant sensitivity coefficients are

$$A_{k,x_{si,i}} = \frac{\partial f_k}{\partial x_{si,i}}; A_{k,x_{si,j}} = \frac{\partial f_k}{\partial x_{si,j}}; A_{k,D_{si,i}} = \frac{\partial f_k}{\partial D_{si,i}}; A_{k,D_{si,j}} = \frac{\partial f_k}{\partial D_{si,i}} \tag{13}$$

The uncertainty $u(\alpha)$ of the parameter α is also based on the law of propagation of uncertainties (10) as

$$u^2(\alpha) = A^2_{\alpha,x_{si,i}} u^2(x_{si,i}) + A^2_{\alpha,x_{si,j}} u^2(x_{si,j}) + A^2_{\alpha,D_{si,i}} u^2(D_{si,i}) + A^2_{\alpha,D_{si,j}} u^2(D_{si,j}) \tag{14}$$

In this case, the sensitivity coefficients are

$$A_{\alpha,x_{si,i}} = \frac{\partial f_\alpha}{\partial x_{si,i}}; A_{\alpha,x_{si,j}} = \frac{\partial f_\alpha}{\partial x_{si,j}}; A_{\alpha,D_{si,i}} = \frac{\partial f_\alpha}{\partial D_{si,i}}; A_{\alpha,D_{si,j}} = \frac{\partial f_\alpha}{\partial D_{si,i}} \tag{15}$$

5.2 Uncertainty of SSA calculation using a power model of particle size distribution

Similarly, we apply the law of propagation of uncertainties in the form (10) to relation (8), which gives us the equation for calculating the uncertainty $u(a_z)$ of the estimate of the apparent specific surface a_z

$$\begin{aligned} u^2(a_z) = {} & A^2_{a_z,k} u^2(k) + A^2_{a_z,\alpha} u^2(\alpha) + A^2_{a_z,\varkappa} u^2(\varkappa) + A^2_{a_z,x_{max}} u^2(x_{max}) \\ & + A^2_{a_z,x_{min}} u^2(x_{min}) \end{aligned} \tag{16}$$

The sensitivity coefficients will be

$$A_{a_z,k} = \frac{\partial f_{a_z}}{\partial k}; A_{a_z,\alpha} = \frac{\partial f_{a_z}}{\partial \alpha}; A_{a_z,\varkappa} = \frac{\partial f_{a_z}}{\partial \varkappa}; A_{a_z,x_{max}} = \frac{\partial f_{a_z}}{\partial x_{max}}; A_{a_z,x_{min}} = \frac{\partial f_{a_z}}{\partial x_{min}} \tag{17}$$

Based on relation (8), $a_z = f(k, \alpha, \varkappa, x_{max}, x_{min})$. The individual standard uncertainties u in relation (16) are obtained as follows:

a. $u(k)$, $u(\alpha)$ from relation (12), respectively (14),

b. $u(\varkappa) = 0$, since $\varkappa$ is a constant,

c. $u(x_{max})$, $u(x_{min})$ by applying the law of propagation of uncertainties in the form (10) to relations (9), substituting the uncertainties $u(k)$, $u(\alpha)$, calculated in the previous step.

6 Example of calculating apparent specific surface area

To determine the estimate of the apparent specific surface area a_z according to relation (8) and the associated uncertainty $u(a_z)$, first we need to know the estimates of the parameters k, α, $\varkappa$, x_{max}, x_{min} and the associated uncertainties $u(k)$, $u(\alpha)$, $u(\varkappa)$, $u(x_{max})$, $u(x_{min})$. To calculate these parameters (except $\varkappa$), we first need to know the value of the parameter D for the relevant x - see relations (5), (6), (9). To determine the value of the parameter D for a given x, we will use the already mentioned *sieve analysis* of particulate matter - see relations (2), (3).

To demonstrate the calculation, we performed a sieve analysis of the given particulate matter using seven sieves. We received input values according to Table 1. This table also contains the measurement uncertainties of the input quantities, determined according to the literature [24]. In doing so, we considered the metrological properties of the used Radwag WLY balance with a measuring range of 0 to 100 g, with a resolution (interval) d = 0.01 g, a repeatability of 0.03 g, and a linearity of ±0.03 g. The uncertainties of the sieve openings were determined with the use of the permissible errors given in [25]. The last two columns contain the values of the cumulative percentage $D_{si,i}$ on sieve i, calculated according to relations (2), (3) and the corresponding uncertainties $u(D_{si,i})$, determined based on the law of propagation of uncertainties [24].

Table 1. Input parameters and their uncertainties, calculated cumulative percentage $D_{si,i}$ and its uncertainty

	Nominal sieve opening size	Uncertainty of sieve opening size	Mass of the fraction on the sieve (on the bottom resp.)	Uncertainty of measurement of mass of substance	Calculated cumulative percentage on sieve i	Uncertainty of calculated cumulative percentage
i	$x_{si,i}$ / mm	$u(x_{si,i})$ / mm	m_i / g	$u(m_i)$ / g	$D_{si,i}$	$u(D_{si,i})$
1	1.6	0.027135463	16.28	$24.66 \cdot 10^{-3}$	0.9457	$80 \cdot 10^{-6}$
2	1.4	0.024248711	32.81	$24.66 \cdot 10^{-3}$	0.8362	$105 \cdot 10^{-6}$
3	1.25	0.02193931	24.65	$24.66 \cdot 10^{-3}$	0.7540	$115 \cdot 10^{-6}$
4	1.0	0.017320508	41.89	$24.66 \cdot 10^{-3}$	0.6143	$120 \cdot 10^{-6}$
5	0.8	0.014433757	34.16	$24.66 \cdot 10^{-3}$	0.5003	$115 \cdot 10^{-6}$
6	0.5	0.009237604	52.75	$24.66 \cdot 10^{-3}$	0.3244	$100 \cdot 10^{-6}$
7	0.2	0.004041452	55.28	$24.66 \cdot 10^{-3}$	0.1399	$75 \cdot 10^{-6}$
8	-		41.96	$24.66 \cdot 10^{-3}$		

To calculate parameter estimates k, α, a power model in the form (1) will be used, employing the calculated values of the cumulative percentage on a sieve with a known size of openings. In relations (5), (6), we will employ the values xsi,i, xsi,j, Dsi,i, Dsi,j from table 1, related to sieves 2 and 5, i.e. i = 2 and j = 5. The uncertainties u(k), u(α) are calculated using relations (12), (14).

In the next step, xmax, xmin are calculated using relations (9). Their uncertainties u(xmax), u(xmin) are calculated based on the law of propagation of uncertainties (10). The values u(k), u(α), calculated in the previous step, were used. The values D(xmax), D(xmin) are considered constant, i.e. they are not affected by measurement uncertainty. The calculated parameters and their uncertainties are listed in Table 2. These parameters serve as inputs for calculating the apparent specific surface according to relation (8).

In addition to the standard uncertainty associated with the estimate of the relevant parameter, Table 2 also shows the expanded uncertainty and the relative expanded uncertainty of the respective parameter. We see an interesting phenomenon here. The relative expanded uncertainty of individual parameters is in the order of units of percent, but the relative expanded uncertainty of the minimum value of the particle size x_{min} reaches approximately 45%. This indicates the limitations of the power model in the area of very small values of the particle size

of the particulate matter. In this case, even the generally small standard uncertainty $u(x_{min})$ will be significantly reflected in relation to the small value of the estimate of the parameter x_{min}. However, we note that it will not be the largest contribution to the combined uncertainty in determining the estimate of a_z (see Table 4).

Table 2. Input parameters and their uncertainties for calculating the apparent specific surface area SSA

Parameters	**Unit**	**Parameter estimate**	**Standard uncertainty**	**Expanded uncertainty U (k = 2)**	**Relative expanded uncertainty U_r / %**
k, $u(k)$	mm	1.7012	0.0411	0.0822	4.83
α, $u(\alpha)$	-	0.9179	0.0411	0.0822	8.96
x_{max}, $u(x_{max})$	mm	1.7012	0.0411	0.0822	4.83
x_{min}, $u(x_{min})$	mm	0.01127	$2.55 \cdot 10^{-3}$	0.0051	45.25
$\varkappa$	-	1			

The input data from Table 1 were inserted into equation (8) to obtain an estimate of a_z. The measurement uncertainty $u(a_z)$, associated with this estimate, was calculated using equation (16). The results are presented in Table 3. For comparison with the analytical calculation, the values of the estimate a_z and the corresponding measurement uncertainty $u(a_z)$ obtained using numerical Monte Carlo simulation are also presented.

Table 3 Estimate *of* a_z and uncertainty $u(a_z)$

	Unit	**Parameter estimate**	**Standard uncertainty**	**Expanded uncertainty (k = 2)**	**Relative expanded uncertainty / %**
		a_z	$u(a_z)$	$U(a_z)$	U_r
Analytical calculation	mm^{-1}	20.10	1.79	3.58	17.81
Numerical Monte Carlo simulation	mm^{-1}	20.30	1.90	3.80	18.72

Table 4 Uncertainty balance for analytical calculation a_z

Parameter	**Estimate**	**Standard uncertainty**	**Probability distribution**	**Sensitivity coefficient**	**Contribution to combined uncertainty**	**Proportional contribution to combined uncertainty**
k	1.7012 mm	0.0411 mm	Normal	-11	-0.45 mm^{-1}	6.2%
α	0.9179	0.0411	Normal	-32	-1.3 mm^{-1}	53.9 %
$\varkappa$	1.0					
x_{max}	1.7012 mm	0.0411 mm	Normal	1.9	0.078 mm^{-1}	0.2%
x_{min}	0.01127 mm	$2.55 \cdot 10^{-3}$ mm	Normal	433.66	-1.1 mm^{-1}	39.7%

Let us now compare the estimate a_z and its uncertainty, which we obtained by analytical calculation and numerical Monte Carlo simulation. Looking at Table 3, the estimate and the corresponding uncertainty differ, which is then reflected in the different value of the relative uncertainty U_r. A graphical representation of the comparison of the results of the analytical calculation is shown in Fig. 2 The red lines show the analytical calculation (vertical lines indicate the estimate, the uncertainty limits, the assumed course is also shown in red), the blue lines indicate the result of the Monte Carlo simulation and the narrow vertical gray columns placed close to each other represent the relative frequency of the values a_z in a given interval.

In the figure, one grey column represents 10 thousand repetitions of the numerical simulation, a total of approximately 2 million repetitions of the simulation were performed.

We see that the red line, i.e. the analytical simulation, assumes a certain course of the normal distribution of the results. However, the gray bars do not fully correspond to this and indicate that in fact the results of repeated evaluations a_z follow a shifted normal distribution. This is a consequence of the exponential form of the relation (8) for the calculation of a_z. In this case, it can be stated that the numerical Monte Carlo simulation approximates reality better than the analytical calculation.

GUM Workbench software [26] was used for all analytical calculations of parameter estimates, associated uncertainties, and numerical Monte Carlo simulations.

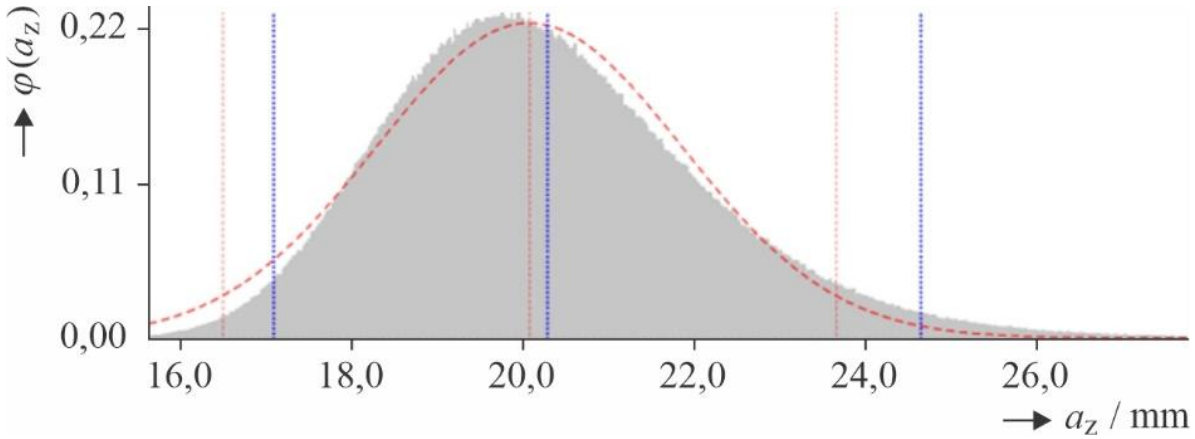


Fig. 2 Illustration of the obtained results for the apparent specific surface area

7 Analysis of results

The resulting relative expanded uncertainty $U_r(a_z)$ of the apparent specific surface a_z reaches a relatively high value mainly due to the sensitivity of the computational model (8) to the input parameters and their uncertainties. The exponent of the power model α and the lower limit of the particle size x_{min} play a significant role, while their uncertainties are significantly transferred to the result (see Table 4). In particular, the parameter x_{min} shows a high relative uncertainty, which is a consequence of its small absolute value and the limits of the power model in the area of small particles. Although the relative uncertainty of the parameter x_{min} is high, its contribution to the combined uncertainty is limited.

Improvement of the resulting uncertainty $u(a_z)$ can be achieved mainly by reducing the uncertainty of the dominant input quantities. Based on the balance of uncertainties, a more accurate determination of the exponent α and a better characterization of the smallest particles in the system using the parameter x_{min} are key.

In this context, the question arises whether the parameters x_{max} and x_{min} should not be set as a fixed number, without the burden of measurement uncertainty (namely when the values $D(x_{min})$, $D(x_{max})$ were taken as constants). In the case of these parameters, it is a matter of setting the limits of the particle size that will be included in the calculation of a_z. Let us now assume that $x_{max} = 1.7$ mm and $x_{min} = 0.01$, both parameters being fixed numbers, without the associated measurement uncertainty. To calculate the estimate a_z, we use the same relationships and the same values and uncertainties of the remaining parameters from Table 2. We get the result $a_z = (20.7 \pm 3)$ mm^{-1}, which reduces the relative expanded uncertainty to the value $U_r(a_z) = 14.5\%$.

CONCLUSION

The results obtained show that the relative expanded uncertainty of SSA determination using the power-law model reaches a value of approximately 15 to 18%, which is higher than for permeability Blaine method (approximately 0.7% for CRM, or approximately 2 to 5% in practice) and usually more than the BET adsorption method (approximately 5 to 10%, or up to 20%). On the other hand, it is comparable to methods based on granulometry and density (approximately 5 to 20%), which confirms its character as an indirect calculation method. The increased uncertainty is mainly due to the sensitivity to the uncertainty in the determination of the parameter α and the uncertainty in the determination of the lower limit of the particle size.

The method of calculating SSA using the power model is therefore particularly suitable for cases where non-porous, non-aggregated particles of approximately spherical shape with a relatively narrow size distribution are involved. It will be applied primarily for rapid estimation of SSA for technical powders in the micrometer range, where specialized instrumentation is not available. On the contrary, for reference measurements with a required small measurement uncertainty, or for materials with a developed porous structure, it is more appropriate to use adsorption or permeability methods.

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