Characterization of Test Dust for Product Qualification

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For product qualification and quality assurance utilities, devices, frames or other assemblies have to undergo a protection class test. During this IP protection class test the test item has to be evaluated for its ingress protection against water and/or dust particles. Within the protection class IP 5X dusts may enter the equipment but must not interfere with the product function. In protection class IP 6X, however, it has to be dust proof. Especially with regard to the durability the feeding properties and effects of the particles to the interior of the utility have to be examined more closely [1-4]. Within this paper some of the dusts that are currently used for testing were examined regarding their particle size distribution, average particle size, particle density, bulk and tap density, specific surface area and morphology.

***Keywords**: test dust, particle properties, product qualification arizona dust*

1. Introduction

During an IP protection class test the test item that consists of mechanically moveable or electronic components has to be evaluated for its ingress protection against water or dust particles. Within the protection class IP 5X dusts may enter the equipment but must not interfere with the product function. However in the protection class IP 6X it has to be dust proof. Especially with regard to the durability the feeding properties and effects of the particles to the interior of the utility have to be examined more closely [1-4]. Within this paper some of the dusts that are currently used for testing were examined regarding their particle size distribution, average particle size, particle density, bulk and tap density, specific surface area and morphology.

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2. Materials

The above mentioned attributes of the following particle collectives respectively test dust were determined:

- Portland cement and fly ash (50 wt.%/50 wt.%) in accordance with DIN 40 050 – 9 [1]
- Portland cement
- Arizona dust “fine” in accordance with SAE J 726 [2] and ISO 12 103-1 [3]
- Arizona dust “coarse” in accordance with SAE J 726 [2]
- Test dust in accordance with JIS Z 8901 Class 8

3 Product Properties and Measuring Methods

3.1 Particle Density

Solid density and particle density respectively is a very important product property that describes the mass per volume

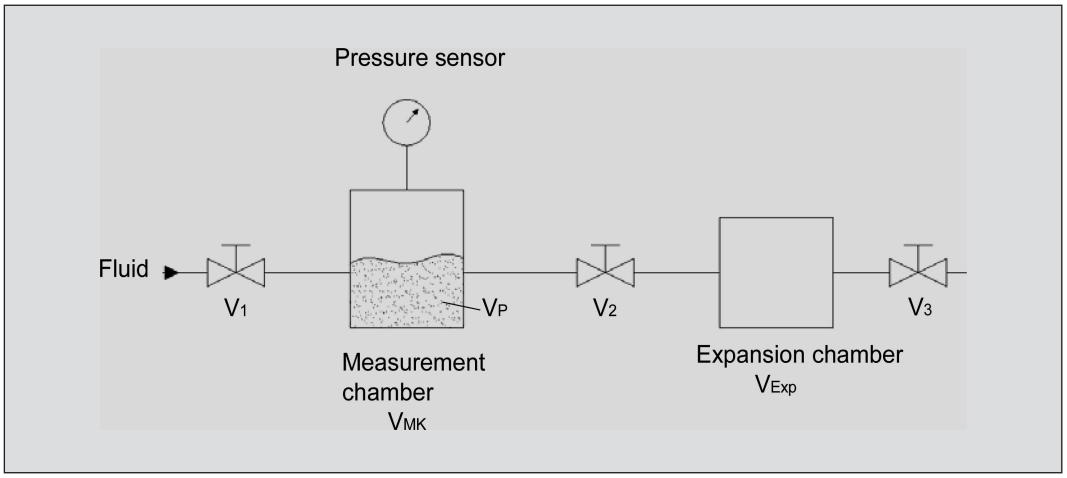


Fig. 1: Schematic view of a pycnometer

ratio. The solid density can be determined with a pycnometer [5]. Therefore, the volume of the individual sample with known mass is determined with pressure measurement within a calibrated measurement chamber (Fig. 1).

With known sample mass the according volume is equal (1) and the density ρ_P can be determined.

$$V_P = V_{MK} - \frac{V_{Exp}}{\frac{\Delta p_1}{\Delta p_2} - 1} \tag{1}$$

3.2 Bulk and Tap Density

Besides solid density the bulk and tap densities are other very important product properties. In contrast to particle density the bulk and tap density contains information about the particle collective and the behaviour of the respective powder.

It applies, for example, that spherical particles due to their higher movability and lower affinity for arching take a higher place in the order of the system and have therefore a larger bulk density than irregular shaped particles. Therefore the bulk density is primarily depending on size, orientation of particles within the bulk, surface roughness, particle shape and the width of particle size distribution of the particle collective and contains further product properties of the testing material implicitly.

The bulk density is additively composed from particle density ρ_P and density of cavity filling fluid ρ_F according to volume share. The tap density is determined by exposing the particle system to vibration for a given time period to make it settle as dense as possible [6, 7].

3.3 Specific Surface Area

The specific surface area of a discrete-disperse system can be calculated from measured particle size distribution or measured directly by photometric, sorption or permeation methods. With these measurement techniques different specific particle size distributions are determined. While permeation methods are used to measure the outer surface area of a powder which corresponds approximately to the geometric surface (calculated from particle size distribution), the sorption methods are used to determine the inner surface area of a collective as well which is particularly eminent for porous particles.

A fluid passes through the bulk particle and, thereby, exposes it to a certain resistance. This flow resistance is the higher the finer the powder is, i.e. the higher its specific surface the higher is it's flow resistance.

The specific surface can be determined with Eq. (2) which is based on the CARMAN-KOZENY equation:

$$S_V^2 = \frac{\Delta p \cdot \varepsilon^3}{L \cdot k_1 \cdot \eta_F \cdot u \cdot (1-\varepsilon)^2} \tag{2}$$

where k_1 is the Carman-Kozeny constant,
η_F is the dynamic viscosity of the fluid,
Δp is the pressure difference over the length of the packing L,
ε is the porosity and
u is the fluid thoughput velocity.

The gas adsorption technique to determine the specific surface area is based on the adsorption of molecules from the adjacent gas or liquid phase onto the solid surface. The quantity of molecules adsorbed to the lower layer is only depending on the geometric shape of the surface. If the necessary amount of gas n_M to cover the monolayer and the required space A_0 of a molecule is known the mass specific surface area S_m can be determined with this Eq. (3):

$$S_m = \frac{n_M}{m} \cdot N_A \cdot A_0 \tag{3}$$

where N_A is the Avogadro number:
$N_A = 6.023 \cdot 10^{23}$ molecules/mole

For the calculation of the specific surface area it is necessary to qualify the adsorption process with the corresponding isotherm. An important model to describe the process is the multi layer adsorption model (BET - Isotherm) developed by BRUNAUER, EMMETT and TELLER (1938). See also [8-12].

The volume related specific surface area S_V can be calculated with the acquired mass related specific surface area S_m as follows:

$$S_V = \rho_P \cdot S_m \tag{4}$$

3.4 Particle Size and Particle Size Distribution

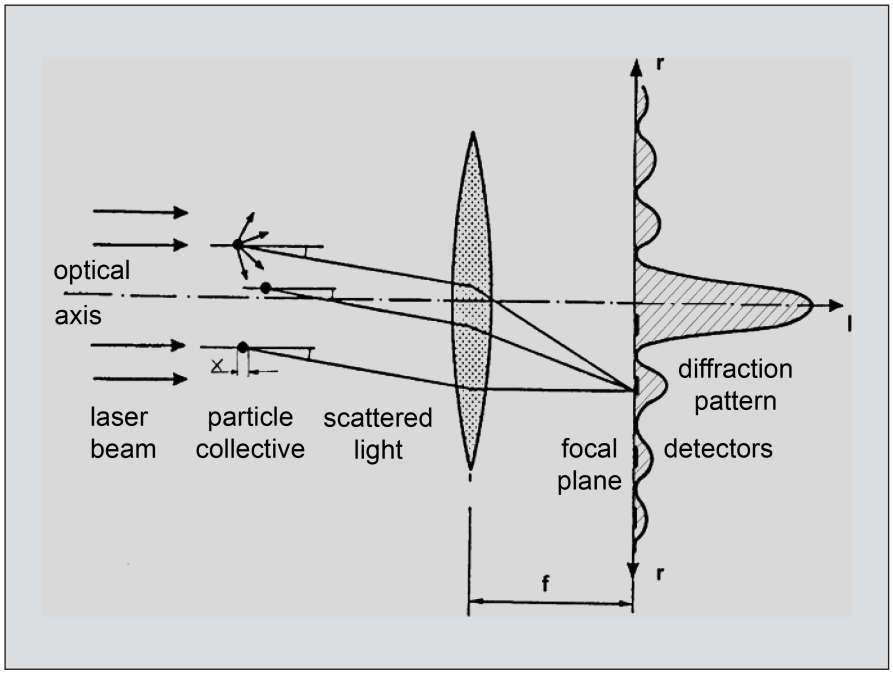
To measure the particle size and particle size distribution within this work the laser diffraction process was used. Laser light diffraction spectrometry is a method that determines particle size distribution based on the scattered light distribution, which originates from particle-light interactions, i.e., from the scattering that occurs as light waves expands through a particulate medium [13,14]. Fig. 2 schematically shows the laser diffraction process.

The volume sum function $Q_3(x)$ and volume density distribution $q_3(x)$ were the result of the particle size analysis. The cumulative distribution $Q_3(x_i)$ represents the concentration of particles equal to or smaller than a given particle size x_i.

$$Q_r(x_i) = \frac{\text{Amount of particles } x \le x_i}{\text{Total amount ofg particles}} \tag{5}$$

The density distribution $q_r(x_i)$ represents the amount of particles of a given particle size x_i, relative to the entire particle size distribution. Amounts that fall within a given particle size interval are referenced to the interval size:

Fig. 2: Basic design of the laser light diffraction spectrometer



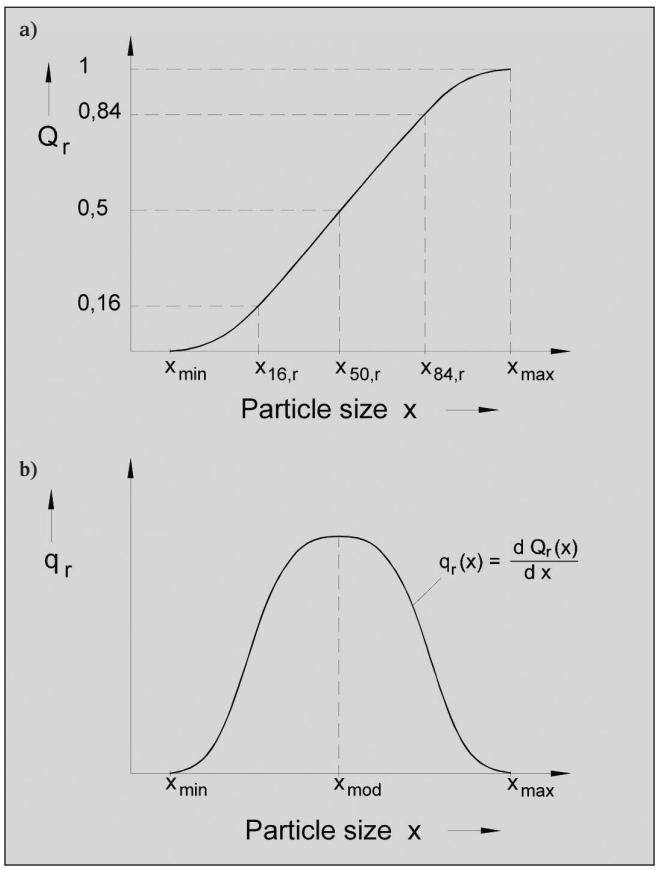


Fig. 3: Particle Site distributions; a) Cumulative distribution $Q_r(x)$; b) density distribution $q_r(x)$

$$q_r(\bar{x}_i) = \frac{\text{Amount between } x_i \text{ and } x_{i+1}}{\text{Interval size } x_{i+1} - x_i} \tag{6}$$

For characterisation of the particle collectives several parameters can be ascertained from the measured particle size distribution. The median $x_{50,r}$ represents the particle size valid for $Q_r(x_{50,r}) = 0{,}5$. This means that 50% of the total quantity of the collective are bellow this measure. The mode of the distribution is the particle size at which the density distribution $q_r(x)$ has a maximum. For spherical particles the volume based specific surface is:

$$S_V = \frac{6}{x} \tag{7}$$

4 Results and Discussion

4.1 Portland Cement and Fly Ash

DIN 40 050 part 9 top 7.3.1 (IP protection class test for road vehicles) requires the test dust compound to consist of 50 wt. % unburnt Portland cement

and 50 wt.% fly ash. In addition this norm demands the test dust to consist of 33 weight proportions $x \leq 32$ µm and 67 weight proportions 32 µm < x ≤ 250 µm.

Portland cement consists of approx. 58% to 66% calcium oxide, 18% to 26% of silicon dioxide (SiO_2), 4% to 10% Aluminium oxide (Al_2O_3) and 2% to 5% Iron(III) oxide (Fe_2O_3).

Fly ash consists predominantly of Silicon-, Aluminium and Iron oxide. Details on product attributes of test dusts are not presented within that norm.

The product properties of three charges (P1 to P3) of the test dust according to DIN 40050 part 9 were determined the results are shown in the following table and figures.

Table 1: Portland cement and fly ash - characteristics

Sample	solid density g/cm³	Bulk density g/cm³	Tap density g/cm³	$x_{10,3}$ µm	$x_{50,3}$ µm	$x_{90,3}$ µm	S_m, calculated cm²/g	S_m Blaine cm²/g
P1	2,53	-	-	4,3	61,69	167,6	1828	2838
P2	2,54	1,33	1,74	4,3	55,63	167,4	1852	2844
P3	2,47	1,27	1,64	6,1	70,87	180,7	1471	2740

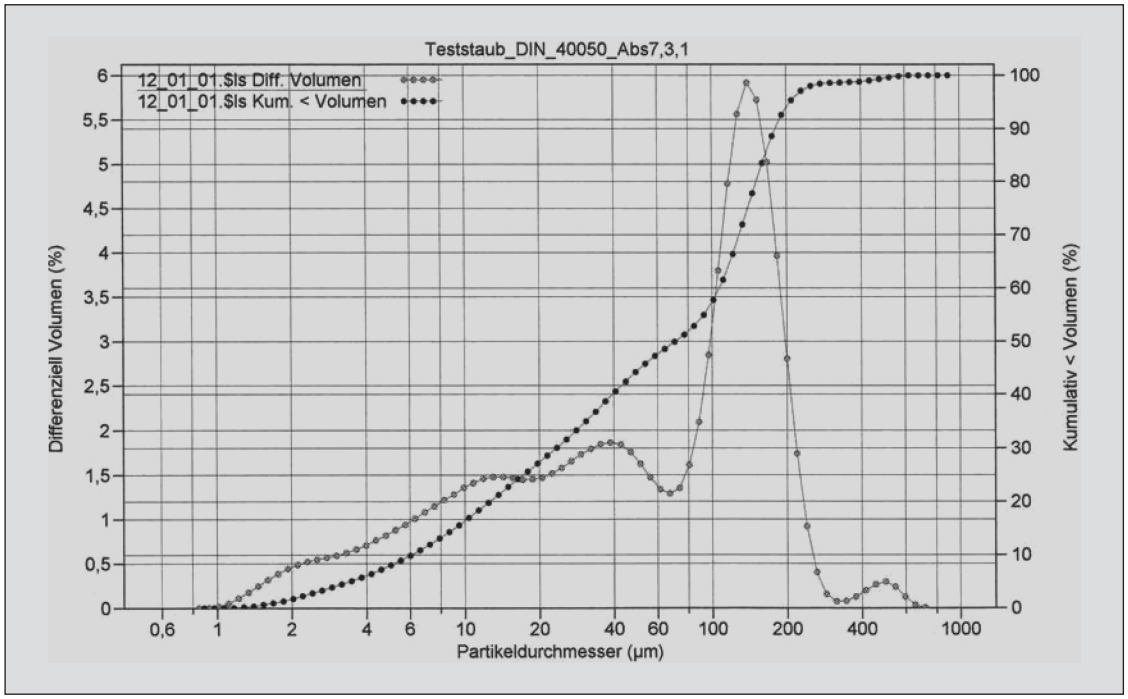
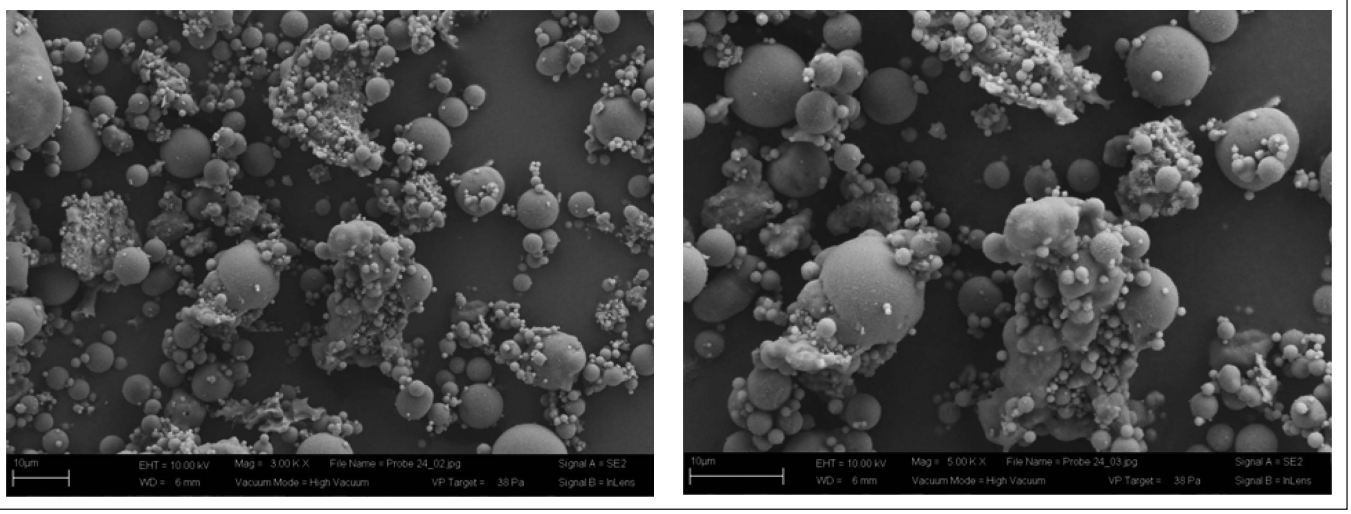


Fig. 4: Particle size distribution (volume) Portland cement and fly ash (P3)

Fig. 5: Portland cement and fly ash - REM-photos



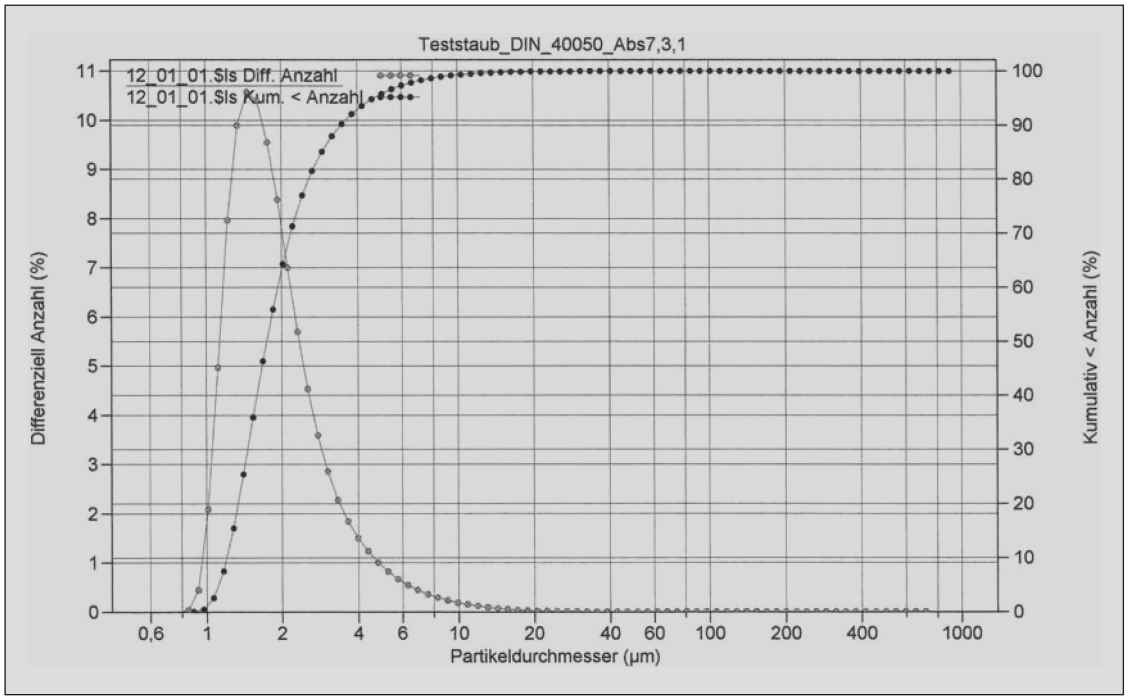


Fig. 6: Particle size distribution (number) Portland cement and fly ash (P3)

Fig. 4 exemplarily shows the volume sum - and volume density distributions of sample P3. At $x \approx 150$ µm the distribution has a modal value. Furthermore it is apparent that it is a tri-modal distribution with the second modal value at $x \approx 40$ µm and the third at $x \approx 14$ µm. This characteristic is shown by all examined test dust samples according to DIN 40050 part 9. The multi-modal characteristic of this sample can also be identified on the images taken by the scanning electron microscope (Fig. 5). The images show that the particle collective has an almost spherical shaped component and that a significant quantity of fine particles exists.

4.2 Arizona Dust

SAE J 726 Rev. June 93 demands synthetically manufactured dust for testing purposes. Its chemical composition is presented in Table 2.

Furthermore SAE J 726 specifies a volume based particle size distribution within the size range between 5,5 µm ≤ x ≤ 125 µm.

4.2.1 Arizona Dust *coarse*

For the particle collectives P4 and P5 the following product properties were determined within the scope of this work.

Fig. 7 shows the volume sum – and volume density distribution of sample P4. It is also apparent that it is a bi-modal distributed product with a median particle size $x_{50,3}$ = 40,2 µm and a large fraction of coarse particles within the area of 90 µm as well as a large fraction of fine particles within the size of 2 µm ≤ x ≤ 15 µm.

At this point we'd like to point out that the coarse and fine particles included in the product have different impacts on the equipment under test if applied for dust test in the environmental simulation. The maximum particle size of Arizona dust *coarse* is at $x_{max} \approx 280$ µm and thereby clearly above the demanded particle size of x = 125 µm.

Table 2: Chemical composition of Arizona dust

Chemical composition	Percentage of weight
SiO_2	67 - 69
Fe_2O_3	3 - 5
Al_2O_3	15 - 17
CaO	2 - 4
MgO	0,5 - 1,5
Total Alkalis	3 - 5
Ignition Loss	2 - 3

Table 3: Arizona dust coarse - characteristics

Sample	solid density	Bulk density	Tap density	$x_{10,3}$	$x_{50,3}$	$x_{90,3}$	S_m, calculated	S_m Blaine
	g/cm³	g/cm³	g/cm³	µm	µm	µm	cm²/g	cm²/g
P4	2,86	-	1,64	3,33	40,22	129,65	1994	3087
P5	2,88	1.02	1,73	3,33	40,22	129,75	2001	3369

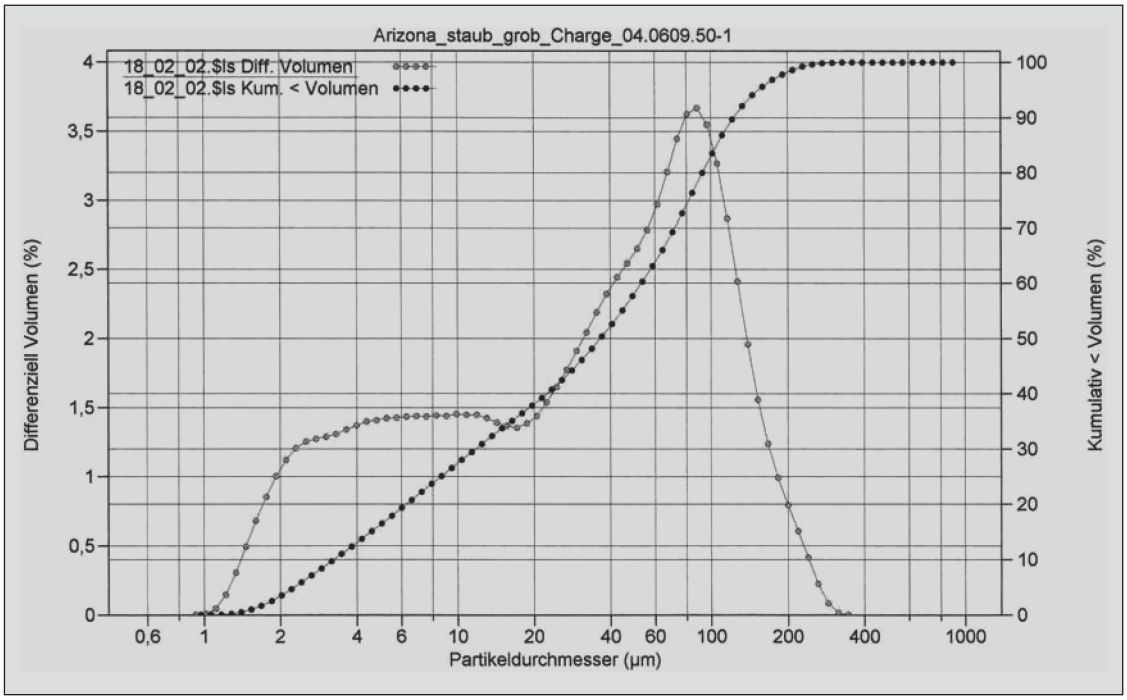


Fig. 7: Particle size distribution (volume) Arizona dust coarse

4.2.2 Arizona Dust fine

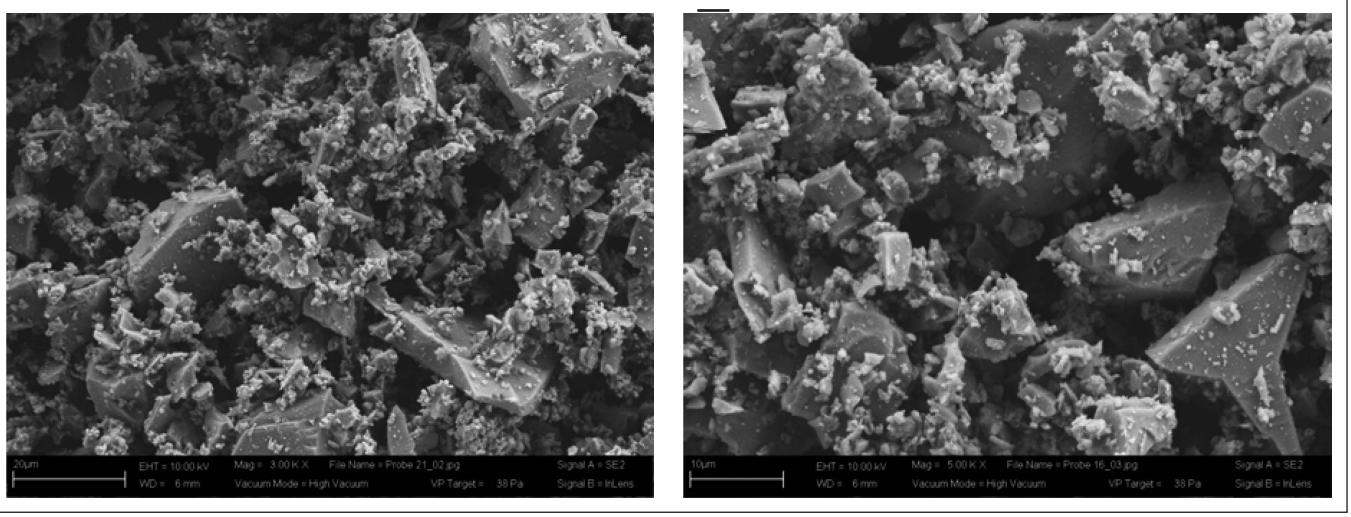
Another particulate product is intended within SAE J 726 Rev. Juni 93 Arizona dust "fine". For the charges P6 to P10 the product properties were determined. These are displayed in Table 4. It gets especially apparent that the product properties of all the examined samples which were all taken from different containers do not show significant discrepancies.

Arizona dust "fine" shows a relatively wide distribution of size but does not posses a distinct multi modal sys-

Table 4: Arizona dust fine - characteristics

Sample	solid density g/cm^3	Bulk density g/cm^3	Tap density g/cm^3	$x_{10,3}$ µm	$x_{50,3}$ µm	$x_{90,3}$ µm	S_m, calculated cm^2/g	S_m Blaine cm^2/g	S_m Gasad cm^2/g
P6	2,95	-	-	2,44	10,8	39,12	3226	6262	6268
P7	2,95	0,74	1,31	2,64	11,08	39,36	3077	6227	-
P8	2,95	0,73	1,30	2,36	9,51	38,83	3523	6779	6835
P9	2,86	0,77	1,33	2,44	10,80	39,12	3366	6161	-
P10	2,97	0,74	1,34	2,40	10,83	39,44	3292	6498	6753

Fig. 8: Arizona dust fine; sample P9 (left) and P10 (right) - REM-photos



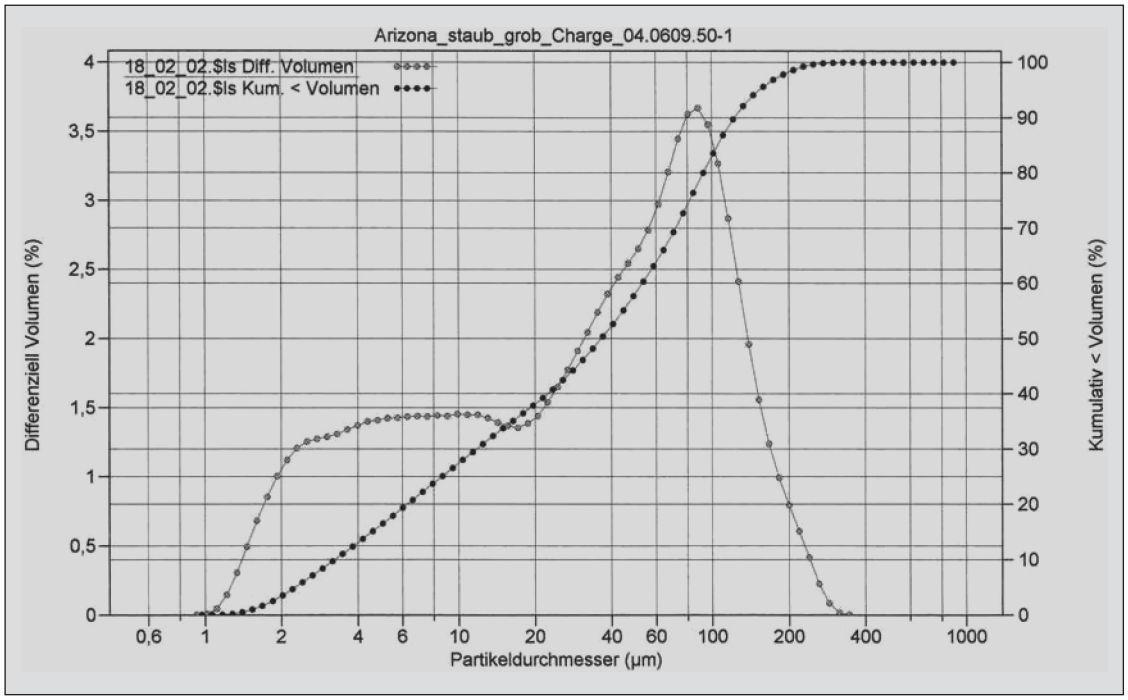


Fig. 9: Particle size distribution (volume) Arizona dust fine

tem. The particles are within the size of 1,5 µm ≤ x ≤ 100 µm. The median size for sample P9 is $x_{50,3}$ = 10,8 µm. Fig. 8 shows example images taken by a scanning electron microscope of samples P9 and P10. These images point out the width of distribution. It is apparent that a large percentage of particles with x = 20 µm is existent within the product which is confirmed by Fig. 9.

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