Table 5: Products investigated, PSD at the aerosol generator, bulk and tamped density of powders (HDK only informative here)

Substance	SiO_2	SiO_2	SiO_2	$CaCO_3$
Substance type	pyrogenic	silica gel	pyrogenic	precipitated
Surface treatment	HDMS-treated	untreated	untreated	untreated
Product	AEROSIL® R 812	SYLOID® 244 FP	HDK® N 20	SOCAL® UP-G
$x_{10;3}$ [µm]	5.15	1.10	5.15	0.89
$x_{16;3}$ [µm]	6.25	1.31	6.29	1.20
$x_{50;3}$ [µm]	11.6	2.31	13.6	3.31
$x_{84;3}$ [µm]	20.0	3.62	31.6	7.15
$x_{90;3}$ [µm]	23.3	4.05	40.1	8.41
Bulk density [kg/m³]	40.8	61.0	32.5	226
Tamped density [kg/m³]	64.0	83.7	46.7	447
Eff. particle density [kg/m³]	64.0	242	46.7	1000
x/x_{ae}	4	2	4.6	1

The following measurements were carried out as part of the tests at ITEM Hannover:

➔ Particle size distribution (geometric) using laser diffraction spectroscopy (Sympatec Helos KR), measurements were made regularly over the entire test time about every 15 minutes.

1. AEROSIL® R 812: 19 measurements
2. SYLOID® 244 FP: 19 measurements
3. $CaCO_3$ UP: 19 measurements

➔ PSD (aerodynamic) by means of cascade impactor, impactor ITEM 1... 2 x for all three fabric systems, impactor TUD one measurement each for AEROSIL® R 812 and for $CaCO_3$ UP

➔ Mass concentration gravimetric

1. AEROSIL® R 812: ITEM 3 x, TUD 5 x
2. SYLOID® 244 FP: ITEM 4 x, TUD 4 x
3. $CaCO_3$ UP: ITEM 3 x, TUD 5 x

4 Conclusions

Within the scope of the measurement campaign at the ITEM Hannover from 23 to 25 June 2021, it was to be investigated whether an OECD-compliant aerosol atmosphere with a solids concentration of 500 to 600 mg/m³ can be generated over a period of 4 h using the substance systems SYLOID® 244 FP, AEROSIL® R 812 and $CaCO_3$ UP. On suggestion of TU Dresden (Dr. Benno Wessely and Prof. Michael Stintz) and in the agreement with the client (SASforREACH, represented by the study monitor Dr. Nils Krüger and the sponsor Dr. Jürgen Nolde), the geometric particle size distribution was monitored using a Sympatec HELOS-KR laser diffraction spectrometer and flanked by cascade impactor measurements and gravimetric concentration measurements.

As a result of the investigations, it can be stated that the generation of an OECD-compliant aerosol atmosphere is possible for all the materials investigated over a period of 4 h.

Only in the case of **AEROSIL® R 812** were there brief instances of slight exceedances (Fig.22). However, these were only detectable with the laser diffraction analysis, since the geometric particle size of the fragile agglomerates could be determined here without additional mechanical stress. The results of the impactor measurements contained in Figs. 26 and 27 show a median value that is lower by a factor of about 25 (!), even after conversion into geometric diameters. The reason is essentially based on the systematic, shear-induced destruction of the flakes when flowing through the impactor.

In the case of **SYLOID® 244 FP** (Figs. 18 and 19) with relatively compact primary particles and a relatively low agglomeration tendency, this effect is lower, but the ratio of the geometric median values (laser diffraction/impactor) is still about 3.

For the **$CaCO_3$ UP**, only a very low agglomeration tendency was observed. Since shear-sensitive agglomerates were present only to a small extent, the impactor measurements show good agreement with the values of the laser diffraction measurements.

Concerning the particle size measurement, it can be stated that the use of cascade impactors in the presence of shear sensitive agglomerates can give a completely wrong picture of the particle size distribution of the aerosol atmosphere. This is also true for aerodynamic particle counters (APS). Relevant agglomerate structures are systematically destroyed during the measurement. Furthermore, the measuring range of these instruments is too small to detect critical coarse particles at all, which is also true for conventional particle counters. A qualified characterization of the aerosol atmosphere of e.g. pyrogenic materials would require the development and application of a measuring system which works absolutely low shear forces and can detect particle sizes from about 0.5 µm to >100 µm with high reliability even in low concentrations in a stable manner over the test period..