Discussion about the use of the Volume Specific Surface Area (VSSA) as criteria to identify nanomaterials according to the EU definition.
Part two: Experimental approach

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Supporting information

Summary

In the supporting information, details are presented about the industrial materials (pigments and MWCNT) employed for the main article but which are not described in the literature. TEM data of pigments were determined by Currenta and provided by U. Hempelmann from Lanxess Deutschland GmbH. The data on the MWCNT NC 7000 were provided by Nanocyl

1. TEM images and particle shapes

The pigments were dispersed in about 10 ml of distilled water by means of ultrasound for 1min with the power of 100W. Thus, agglomerates are destroyed forming single or aggregated particles. The particles are placed on a TEM grid and tested by high resolution scanning transmission electron microscopy (HR STEM Company: FEI, QS- No.: 02464; SOP 00013 Vers.2; Reference Material: UER3 922.00).

The NC 7000 was dispersed in ethanol. The particles are placed on a TEM grid and tested by high resolution transmission electron microscopy at Liège University (Belgium)

FIGURE S1 (a, b, c, d, e, f, g, h, i, j, k, l)

2. Parameters of the particle size distributions

The particle size distribution of the pigments was determined by Image Analysis in accordance with the Council Regulation (EC JRC73260, nanomaterial). The test method is in all essential part identical with the OECD Guideline, Test no. 110, method B.

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For the determination of the particle size distribution, 35 pictures have been taken by STEM at a magnification between 100.000 and 500.000 times. The particle size distribution of the primary particles of inorganic pigments is obtained by evaluation of a series of stored images. The evaluation is performed by "analySIS Pro" (Software from Olympus).

From the area of each particle, the diameter of a circle of equal area is calculated, and stored as particle diameter. The diameter range, usually 0.01 to 1.0 µm, is divided into 64 size classes equally spaced on a logarithmic diameter scale. A histogram is generated showing the number of particles in each size class. The total number is normalized to 100 percent.

Approximately 1000 particles are identified and evaluated. For the agglomerates present, the primary particles building up the agglomerate are evaluated, and not the agglomerate itself. Complex agglomerates are ignored.

For pigments, two distributions were determined: one corresponding to the smallest sizes of the particles and the other corresponding to the largest sizes of the particles.

The particle size distributions obtained by TEM are of the lognormal type as illustrated on *Figure S2* for the Fe2O3 Red 2 sample, the size being given on a logarithmic scale.

FIGURE S2

The median and the quartiles 25 and 75 were reported in the *Table S1* hereafter as well as the standard deviation of the corresponding lognormal distribution, σy, calculated by solving the following set of equations:

$$\text{mean} = [\ln(Q25) + \ln(Q75)] / 2$$

$$\sigma_y = [\ln(Q75) - \text{mean}] / 0.67$$

$$\mu_y + \tfrac{1}{2} (\sigma_y)^2 = \ln(\mu_D)$$

The number percentage of particles below 100 nm is also given. This can be used to assess the nano- or non-nano-character of the samples according to the EC definition.

The aspect ratio for pigments is calculated by dividing the median of distribution of the largest sizes of the particles by the median (µD) of the distribution of the smallest sizes of the particles.

TABLE S1

Table S1: Characteristics of the particle size distributions for the series of industrial samples

Figure S1: TEM images of the industrial pigments (measured particles are colored depending on their mean diameter) and multiwall carbon nanotube (MWCNT)

Figure S2: Example of a lognormal particle size distribution obtained by electron microscopy on the Fe2O3 Red 2 sample

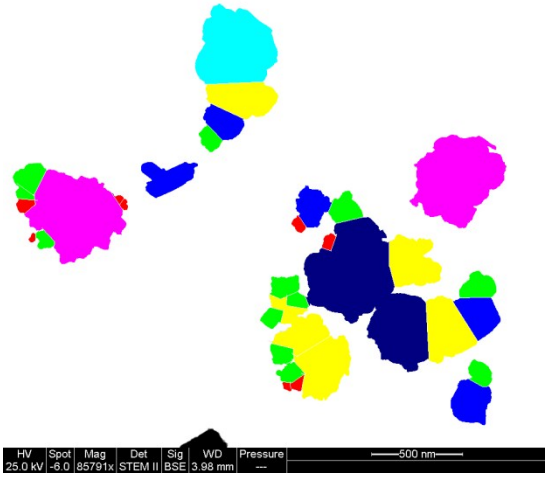
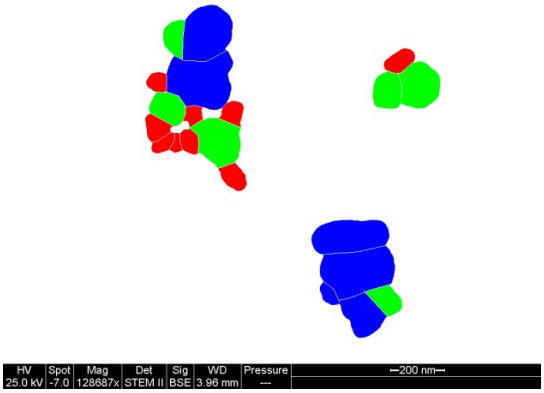


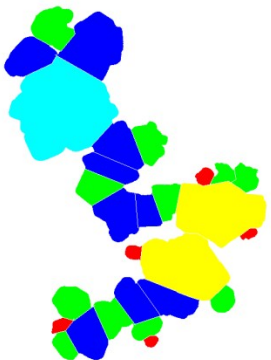
TABLE S1

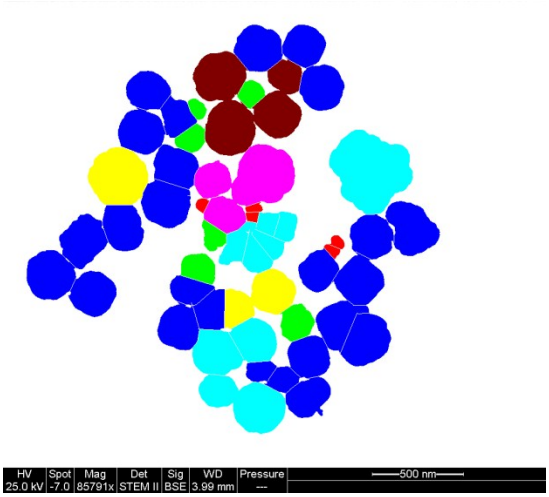
Samples	µD nm	Q25 nm	Q75 nm	σy	Aspect ratio	% particles <100 nm
Cr2O3 pigment	107	74	150	0.527	1.6	44.1
Fe3O4 Black 1	123	72	210	0.799	1.5	40.9
Fe3O4 Black 2	195	120	230	0.485	1.2	13.3
Fe3O4 Black 3	106	60	150	0.684	1.4	46.9
Fe2O3 Red 1	104	65	160	0.672	1.4	47.2
Fe2O3 Red 2	82	57	106	0.463	1.5	66.1
Fe2O3 Red 3	84	60	104	0.410	1.5	65
FeOOH Yel 1	80	57	105	0.456	2.5	67.4
FeOOH Yel 2	105	70	160	0.617	2.1	46.1
FeOOH Yel 3	103	75	160	0.565	2.0	47.8
NC 7000 CNT	9	6.45	11.8	0.453	>100	100

FIGURE S1

a-Chromium oxide

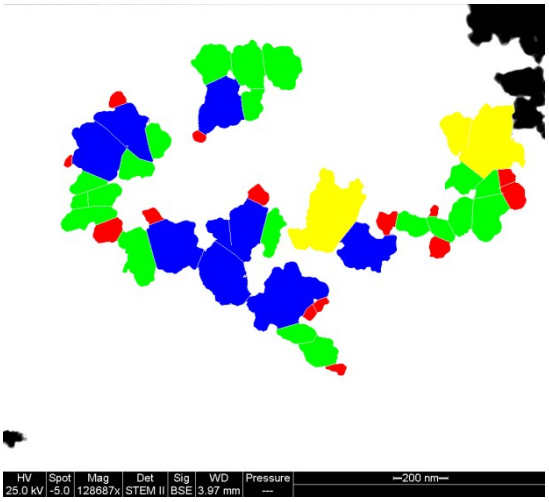






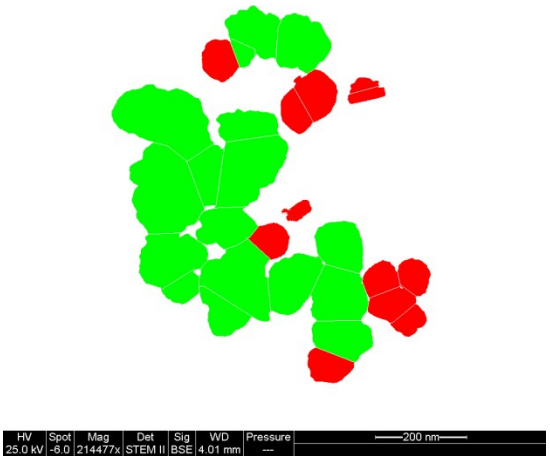
c-Iron oxide Black 2

d-Iron oxide Black 3

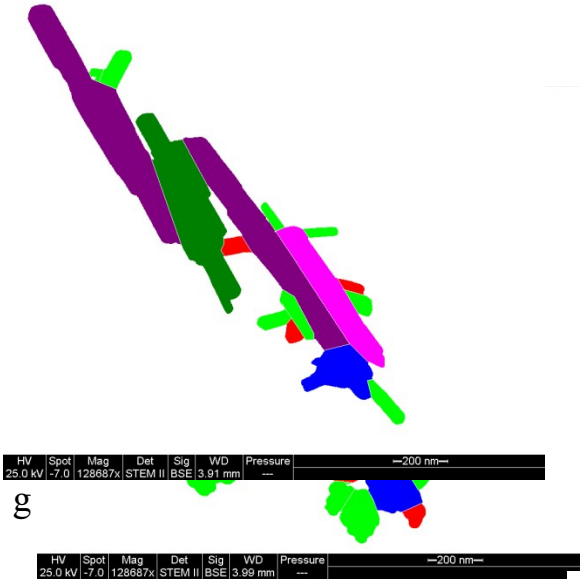


e-Iron oxide Red 1

f-Iron oxide Red 2

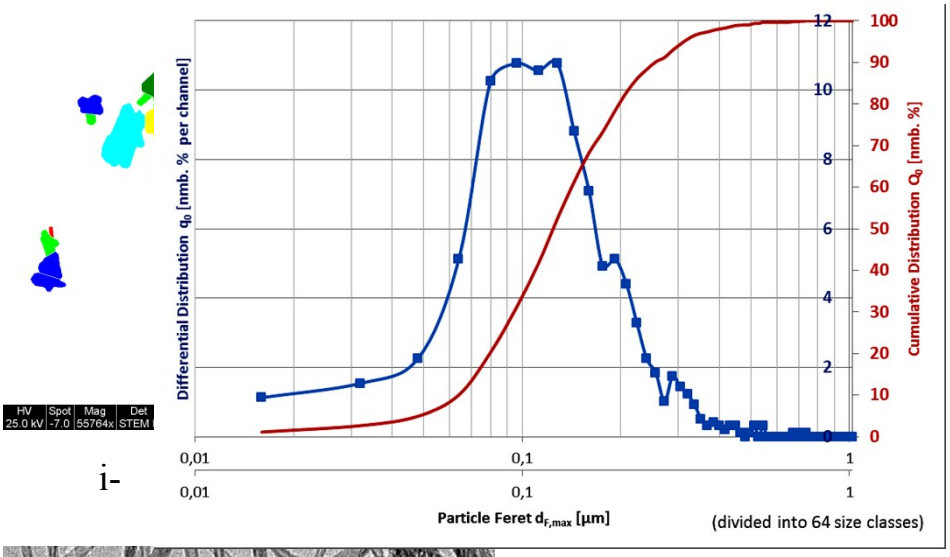


-Iron oxide Red 3

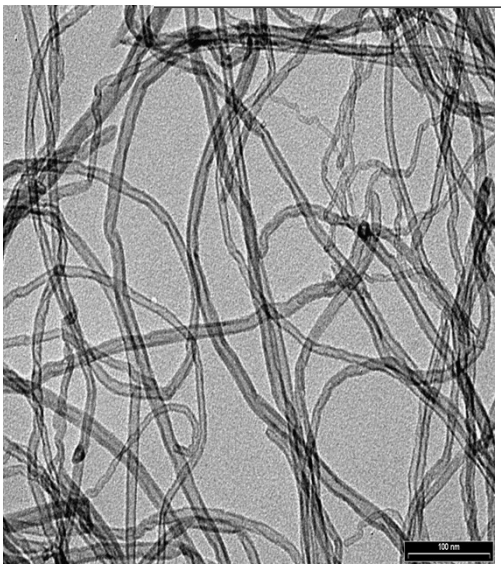


g

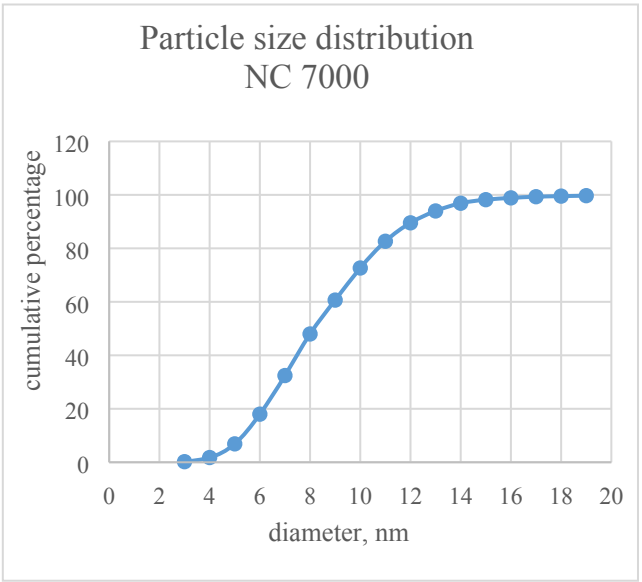
h-Iron oxide Yellow 1



i- Iron oxide



Yellow 2 j-Iron oxide Yellow 3



k-TEM of MWCN NC 7000

l- Diameter distribution NC 7000

Figure S2