Supporting Information for

Experimental approaches to data generation for REACH compliance of multi-walled carbon nanotubes: substance identification

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1 TEST MATERIAL CHARACTERISATION

Table S 1: K-Nanos MWCNTs summary of substance characteristics

Parameter	Method	K- Nanos 100	K- Nanos 210	K- Nanos 300
Purity [(w/w)%]		94.00~97.00	91.95~98.00	95.00~98.60
Impurity, elements	ICP-OES	Al, Co, Fe, Mg, Mo, V		
Single tube length [µm] based on measured data *(bundle n=1, single tube n=9)*	SEM	Mean: 41.68 Min.: 39.37 Max.: 48.04 D10: 39.37 D50: 40.75 D90: 48.04	Mean: 60.54 Min.: 54.22 Max.: 68.55 D10: 54.22 D50: 59.67 D90: 68.55	Mean: 95.94 Min.: 89.70 Max.: 107.74 D10: 89.70 D50: 92.97 D90: 107.74
Single tube length [µm] based on extrapolation [1]	-	Mean: 46.22 Min.: 16.61 Max.: 89.89 D10: 25.44 D50: 46.59 D90: 78.43	Mean: 51.59 Min.: 25.22 Max.: 137.44 D10: 27.34 D50: 45.91 D90: 96.48	Mean: 62.76 Min.: 30.31 Max.: 134.81 D10: 37.50 D50: 55.42 D90: 119.68
Single tube diameter, minimum Feret diameter [nm] *(n=50)*	TEM	Mean: 12.2 Min.: 7.68 Max.: 17.46 D10: 8.93 D50: 11.87 D90: 15.79	Mean: 12.13 Min.: 7.28 Max.: 19.68 D10: 8.05 D50: 12.08 D90: 16.30	Mean: 14.58 Min.: 7.36 Max.: 24.12 D10: 9.63 D50: 14.69 D90: 19.69
Aspect ratio [-]	Single tube length / single tube diameter	Mean: 3789 Min.: 2162 Max.: 5149	Mean: 4253 Min.: 3465 Max.: 6983	Mean: 4304 Min.: 4118 Max.: 5589
Bundle length [µm] *(n=30)*	SEM	Mean: 42.00 Min.: 15.97 Max.: 70.86 D10: 24.47 D50: 43.30 D90: 61.82	Mean: 48.07 Min.: 26.24 Max.: 113.09 D10: 28.44 D50: 43.40 D90: 79.39	Mean: 54.85 Min.: 28.33 Max.: 104.92 D10: 35.06 D50: 49.98 D90: 93.14
Bundle diameter [µm] *(n=40)*	SEM	Mean: 3.21 Min.: 0.45 Max.: 12.80 D10: 0.99 D50: 2.45 D90: 6.39	Mean: 2.97 Min.: 0.87 Max.: 15.44 D10: 1.168 D50: 2.207 D90: 5.327	Mean: 2.46 Min.: 0.70 Max.: 8.30 D10: 1.16 D50: 1.77 D90: 5.24

[1] Single tube length was extrapolated using the respective ratio reported in Table 4 to the available data set of measured full bundle length after 55 minutes (n=30). Note, D10, D50, D90: 10th, 50th, and 90th percentile of the particle size distribution. For method abbreviations see Table S 1 continued.

Table S 1: K-Nanos MWCNTs summary of substance characteristics (continued)

Parameter	Method	K- Nanos 100	K- Nanos 210	K- Nanos 300
Number of walls *(n=30)*	TEM	6–24	5–17	5–25
Crystallinity [%]	Raman	97.00~98.40	97.80~98.60	97.00~98.20
Surface area [m²/g] *(n=26)*	BET, batch-to-batch variation	Mean: 201 Min.: 152 Max.: 287	Mean: 248 Min.: 202 Max.: 298	Mean: 228 Min.: 152 Max.: 348
Zeta potential [mV] (in Ethanol)	Zetasizer	−0.459	−0.122	−0.176
Surface treatment		none (pristine)	none (pristine)	none (pristine)
Relative density [-]	OECD TG 109, gas pycnometer	1.761 ±0.030	-	-
Bulk density [g/cm³]	Constant volume method	0.009–0.030	0.007–0.030	0.005–0.030

Abbreviation: BET – Brunauer, Emmett and Teller method, ICP-OES – Inductively coupled plasma optical emission spectroscopy, SEM – Scanning electron microscopy, TEM – Transmission electron microscopy.

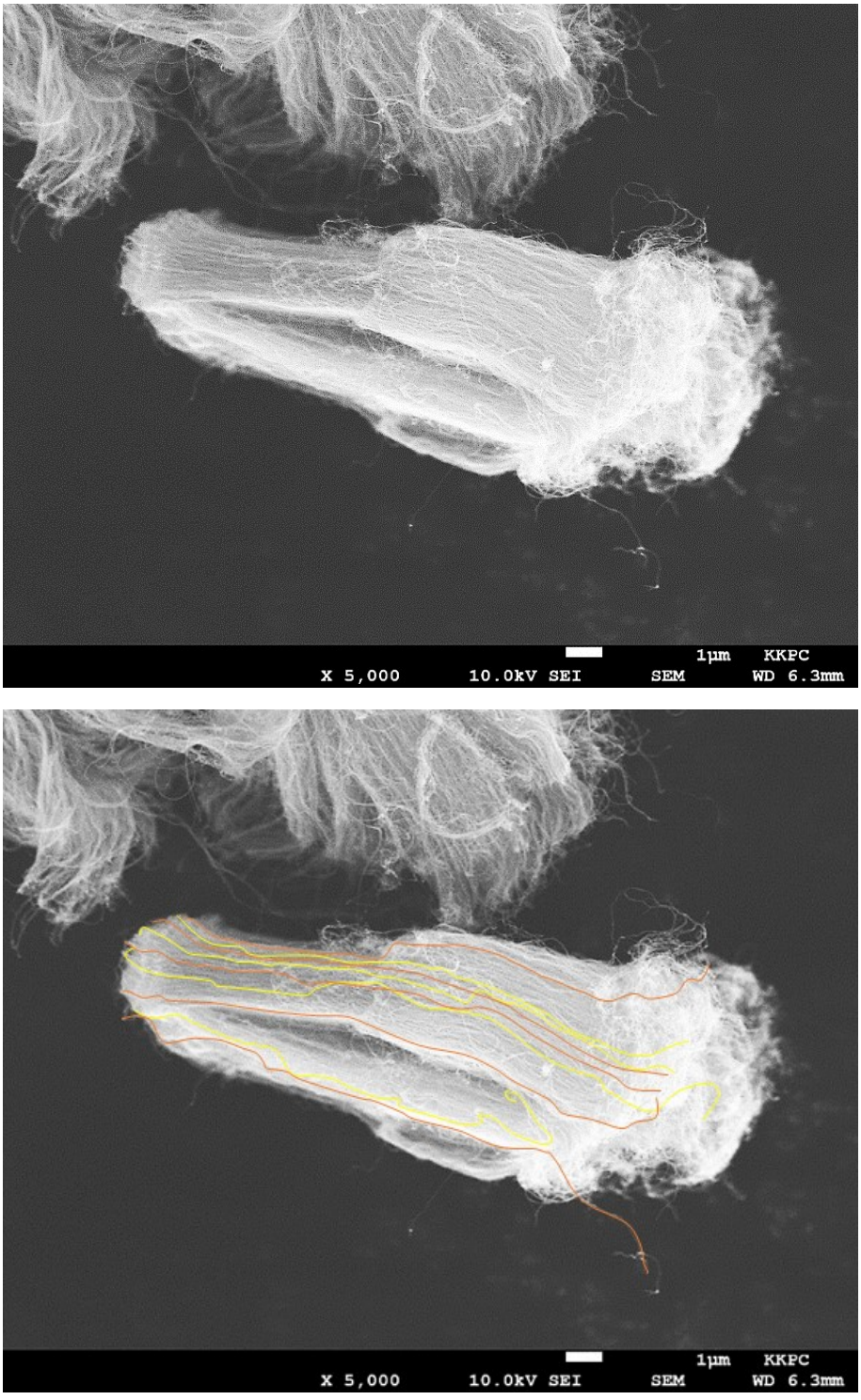

Figure S 1: K-Nanos 100 after 30 min growth time, with and without individual tube tracking using Java ImageJ (v1.54i); bundle length: 22 µm.

Image taken by KKPC, 2024. Image is property of KKPC, with permission.

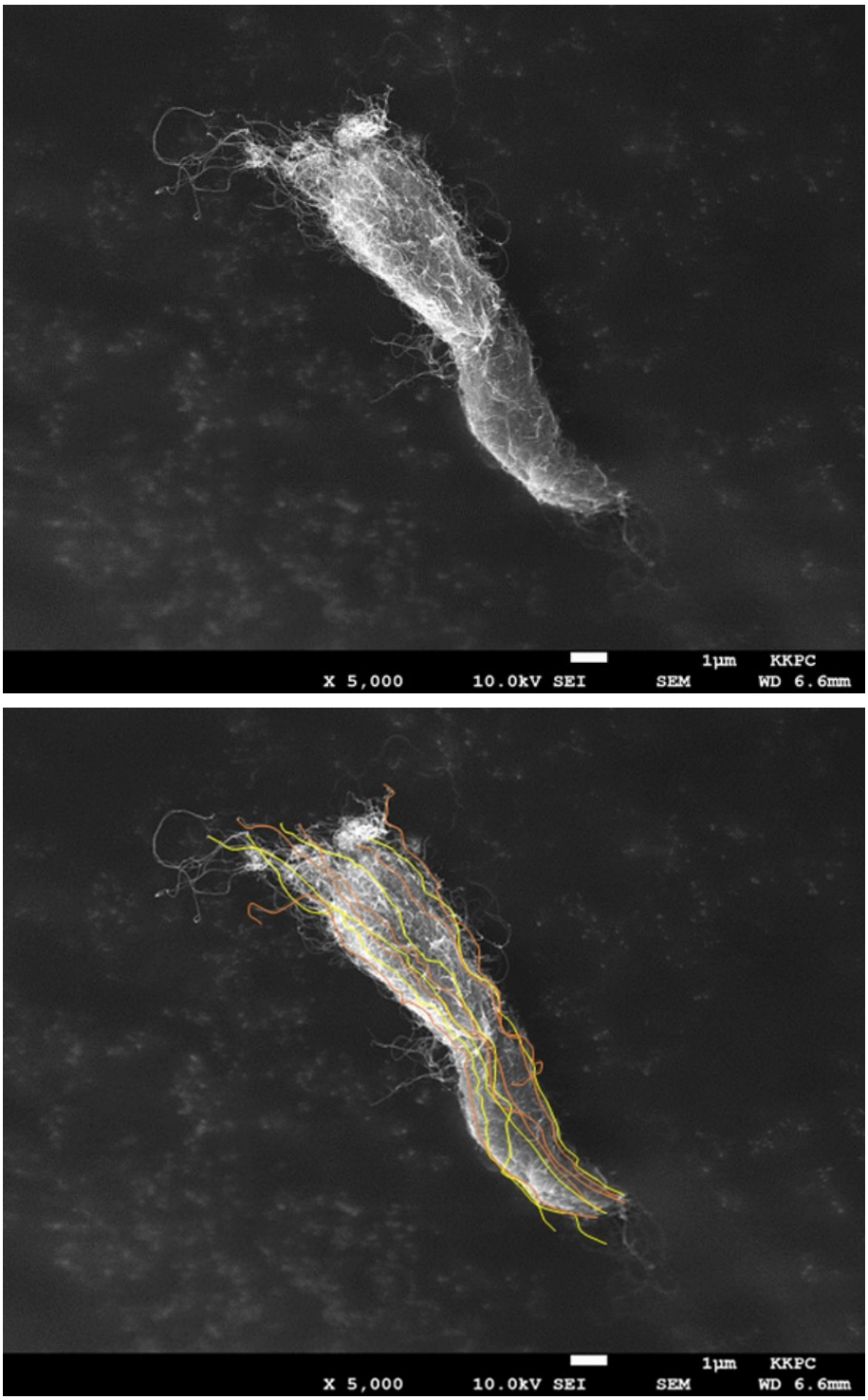

Figure S 2: K-Nanos 210 after 15 min growth time, with and without individual tube tracking using Java ImageJ (v1.54i); bundle length: 17.3 µm.

Image taken by KKPC, 2024. Image is property of KKPC, with permission.

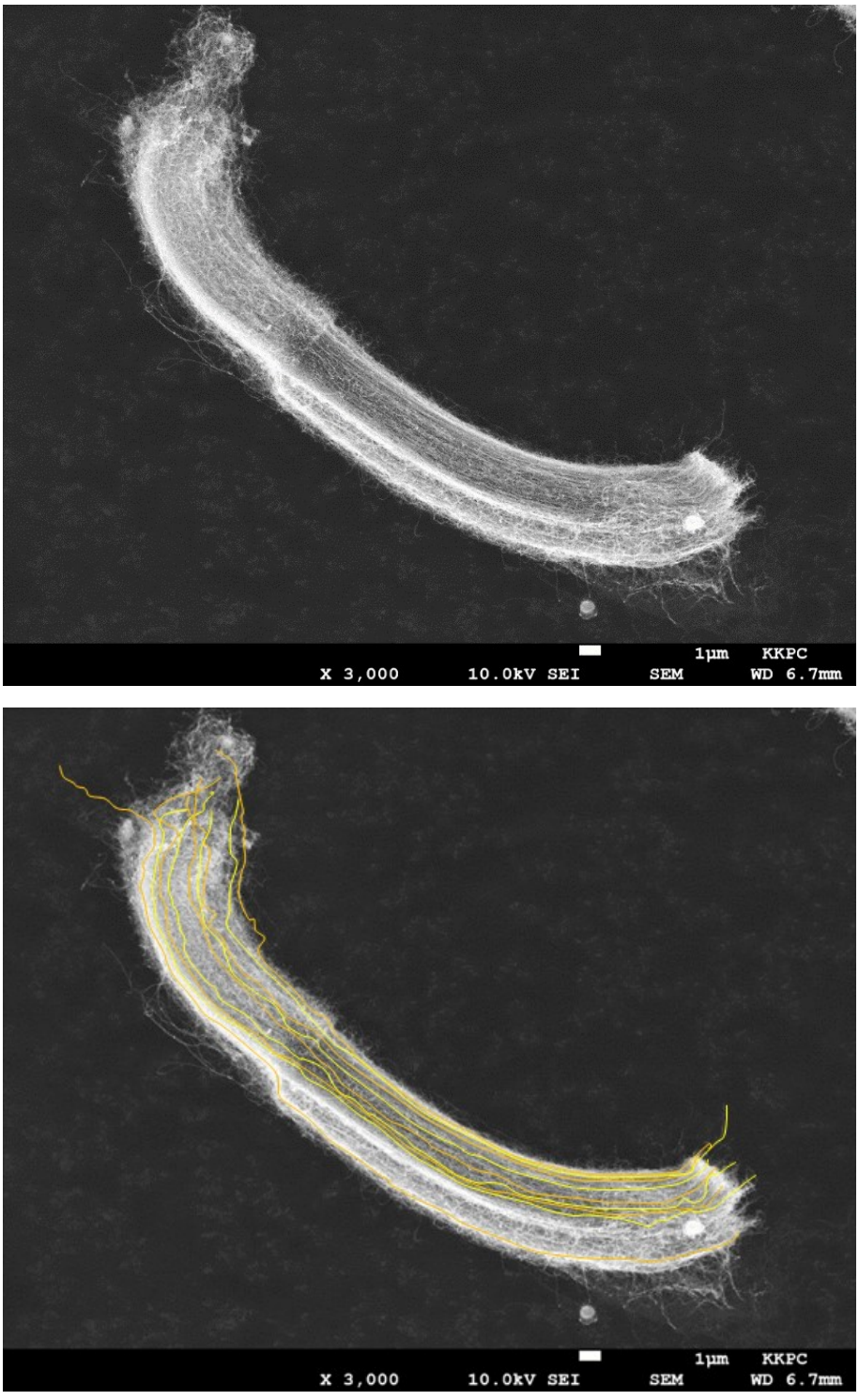

Figure S 3: K-Nanos 300 after 30 min growth time, with and without individual tube tracking using Java ImageJ (v1.54i); bundle length: 47 µm.

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Table S 2: K-Nanos 100 MWCNTs Summary statistics of measured single tube lengths at 1, 15, and 30 min. Single tube length after 55 min full growth time, extrapolated based on linear regression of measured data points.

Parameter	Unit	Single tube length			
		1 min	15 min	30 min	55 min
Mean	µm	2.42	15.36	23.02	41.68
SD	-	0.52	2.24	1.49	NA
CoV	%	22	15	6	NA
Min.	µm	1.77	12.01	21.87	39.37
Max.	µm	3.18	18.67	26.60	48.04
D10	µm	1.77	12.01	21.87	39.37
D50	µm	2.14	15.87	22.22	40.75
D90	µm	3.18	18.67	26.60	48.04

Table S 3: K-Nanos 210 MWCNTs Summary statistics of measured single tube lengths at 1, 15, and 30 min. Single tube length after 55 min full growth time, extrapolated based on linear regression of measured data points.

Parameter	Unit	Single tube length			
		1 min	15 min	30 min	55 min
Mean	µm	1.46	19.83	32.65	60.54
SD	-	0.66	1.66	2.49	NA
CoV	%	45	8	8	NA
Min.	µm	0.62	16.81	29.00	54.22
Max.	µm	2.49	21.71	37.60	68.55
D10	µm	0.62	16.81	29.00	54.22
D50	µm	1.19	20.74	31.95	59.67
D90	µm	2.49	21.71	37.60	68.55

Table S 4: K-Nanos 300 MWCNTs Summary statistics of measured single tube lengths at 1, 15, and 30 min. Single tube length after 55 min full growth time, extrapolated based on linear regression of measured data points.

Parameter	Unit	Single tube length			
		1 min	15 min	30 min	55 min
Mean	µm	1.82	19.81	53.49	95.94
SD	-	0.36	1.44	3.28	NA
CoV	%	20	7	6	NA
Min.	µm	1.22	18.62	49.71	89.70
Max.	µm	2.27	23.40	60.01	107.74
D10	µm	1.22	18.62	49.71	89.70
D50	µm	1.90	19.14	51.93	92.97
D90	µm	2.27	23.40	60.01	107.74

Table S 5: K-Nanos MWCNTs extrapolated bundle length and single tube lengths summary statistics after 55 min based on measured data.

	K-Nanos 100	K-Nanos 210	K-Nanos 300
Bundle length [1] [µm]	37.9	56.4	83.9
Parameter	Single tube length [µm] [2]		
Mean	41.68	60.54	95.94
Min.	39.37	54.22	89.70
Max.	48.04	68.55	107.74
D10	39.37	54.22	89.70
D50	40.75	59.67	92.97
D90	48.04	68.55	107.74
	Ratio		
Mean	1.10	1.07	1.14
Min.	1.04	0.96	1.07
Max.	1.27	1.22	1.28
D10	1.04	0.96	1.07
D50	1.08	1.06	1.11
D90	1.27	1.22	1.28

D10: 10th percentile; D50: median; D90: 90th percentile; [1] based on linear regression (data set n=1); [2] based on linear regression (data set n=9).

Table S 6: K-Nanos MWCNTs linear regression of single tube length: mean average, minimum, maximum, percentiles D10, D50 (median), and D90 as a function of growth time.

	Linear regression		
	K-Nanos 100	K-Nanos 210	K-Nanos 300
Mean	$Y = 0.708x + 2.7411$ $R^2 = 0.9723$	$Y = 1.0729x + 1.5317$ $R^2 = 0.9851$	$Y = 1.7873x – 2.3645$ $R^2 = 0.9766$
Min.	$Y = 0.693x + 1.2568$ $R^2 = 0.9991$	$Y = 0.9766x + 0.5032$ $R^2 = 0.9898$	$Y = 1.6768x – 2.5278$ $R^2 = 0.98$
Max.	$Y = 0.804x + 3.8212$ $R^2 = 0.9589$	$Y = 1.2088x + 2.0612$ $R^2 = 0.9945$	$Y = 1.9963x – 2.0541$ $R^2 = 0.9822$
D10	$Y = 0.693x + 1.2568$ $R^2 = 0.9991$	$Y = 0.9766x + 0.5032$ $R^2 = 0.9898$	$Y = 1.6768x – 2.5278$ $R^2 = 0.98$
D50	$Y = 0.6892x + 2.844$ $R^2 = 0.9485$	$Y = 1.0569x + 1.7537$ $R^2 = 0.9696$	$Y = 1.7306x – 2.2128$ $R^2 = 0.9753$
D90	$Y = 0.804x + 3.8212$ $R^2 = 0.9589$	$Y = 1.2088x + 2.0612$ $R^2 = 0.9945$	$Y = 1.9963x – 2.0541$ $R^2 = 0.9822$

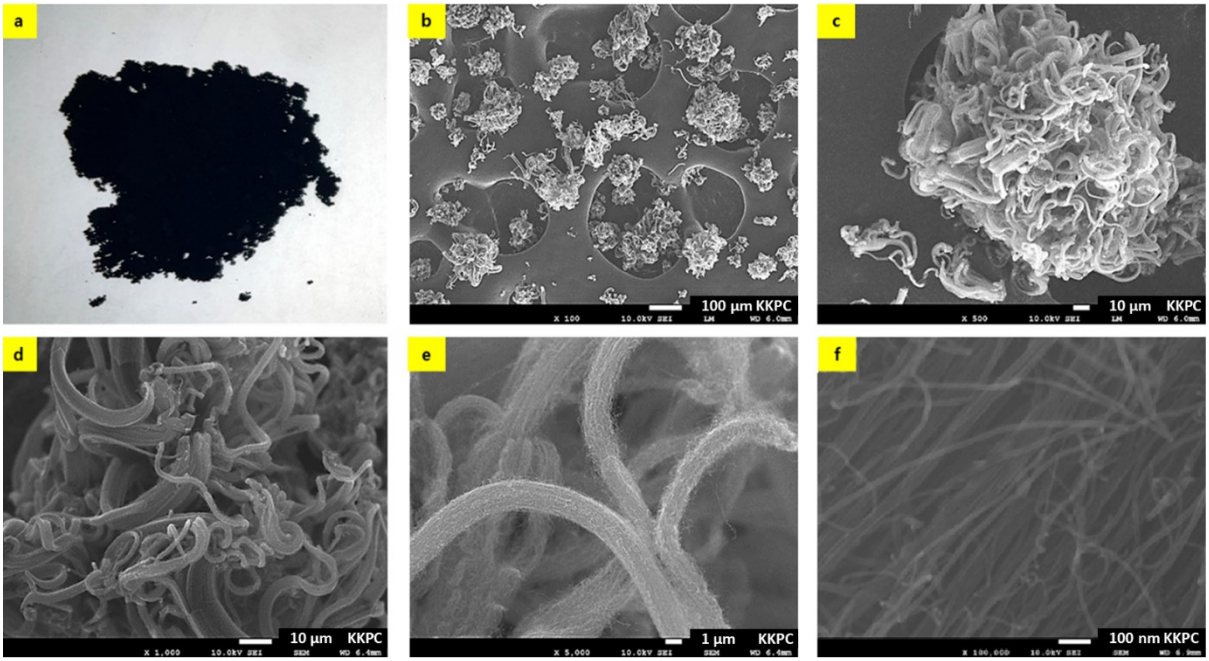

Figure S 4: SEM images of MWCNT bundles and single carbon nanotubes.

a) Photographs of pristine MWCNTs, b) to d) SEM images of MWCNT agglomerates in form of bundles, e) to f) high magnification SEM images of single bundles consisting of 10^4–10^5 single carbon nanotubes that are aligned in parallel rope-like strands.

The images show that it is impossible to visualise the full length of a single carbon nanotube in one image due to limitation related to the magnification of image size, allowing only a fraction of bundle to be captured.

Image taken by KKPC, 2023. Image is property of KKPC, with permission.

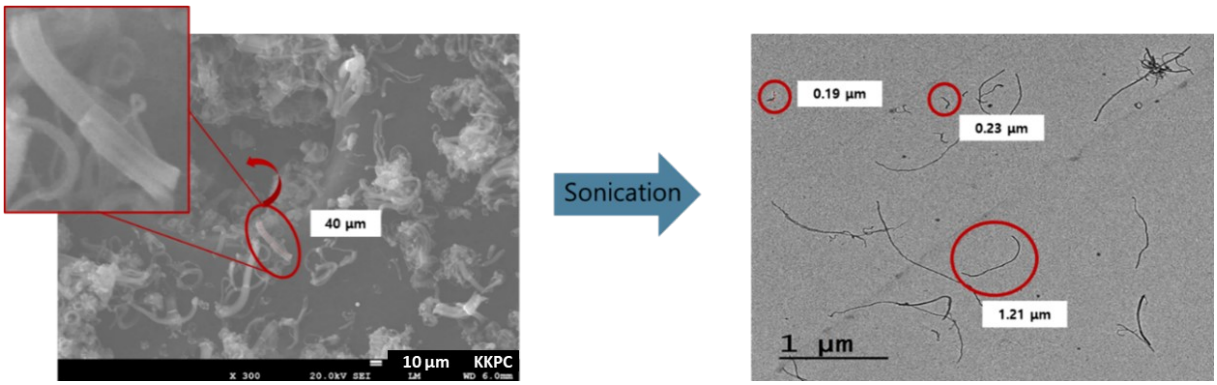

Image taken by KKPC, 2023. Image is property of KKPC, with permission.

Figure S 5: SEM images of K-Nanos MWCNTs before and after sonication.

After sonication in NMP solution for 3 min in 15-seconds intervals at 500 W and 20 kHz, MWCNT bundles were dispersed in individual constituent particles, i.e., single carbon nanotubes. However, the separated constituent particles were significantly shorter than integrated particles of the untreated MWCNT bundle agglomerates. Separated individual MWCNT nanotubes were shortened in length from initially ca. 40 µm-bundle length to < 2 µm .

Image taken by KKPC, 2023. Image is property of KKPC, with permission.

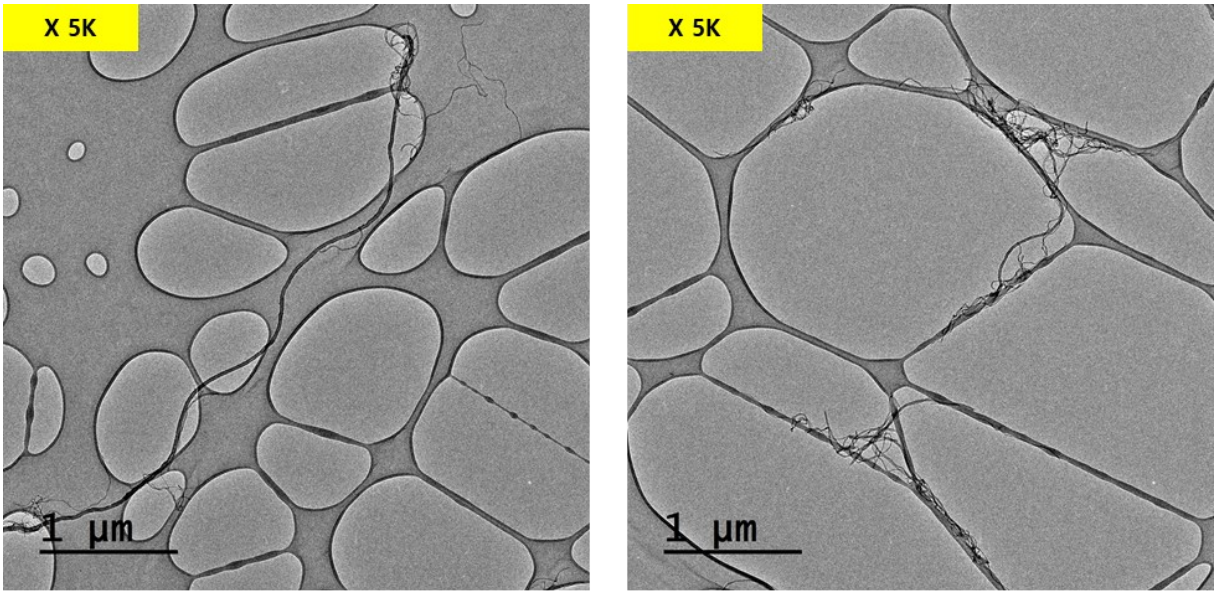

Figure S 6: TEM images of dispersed KKPC MWCNTs by sonication.

Low-magnification TEM images captured after ultrasonication show separated individual MWCNTs preparation (left and right image). The images allow for a closer examination of the sonicated K-Nanos, confirming that the bundle structures were destroyed and the resulting individual tubes exhibit residues from the original material. Ultrasonication was applied for sample preparation for TEM analysis in NMP solution for 3 min in 15-seconds intervals at 500 W and 20 kHz.

In Figure S 6, the large net-like features correspond to the lacy carbon grid. In contrast, the thin, narrow, thread-like structures observed between the larger grid openings are K-Nanos MWCNTs. When examined more closely, these filamentous structures show interruptions and visible tube ends, indicating that the nanotubes are partially broken, due to the ultrasonication process.

Image taken by KKPC, 2023. Image is property of KKPC, with permission.

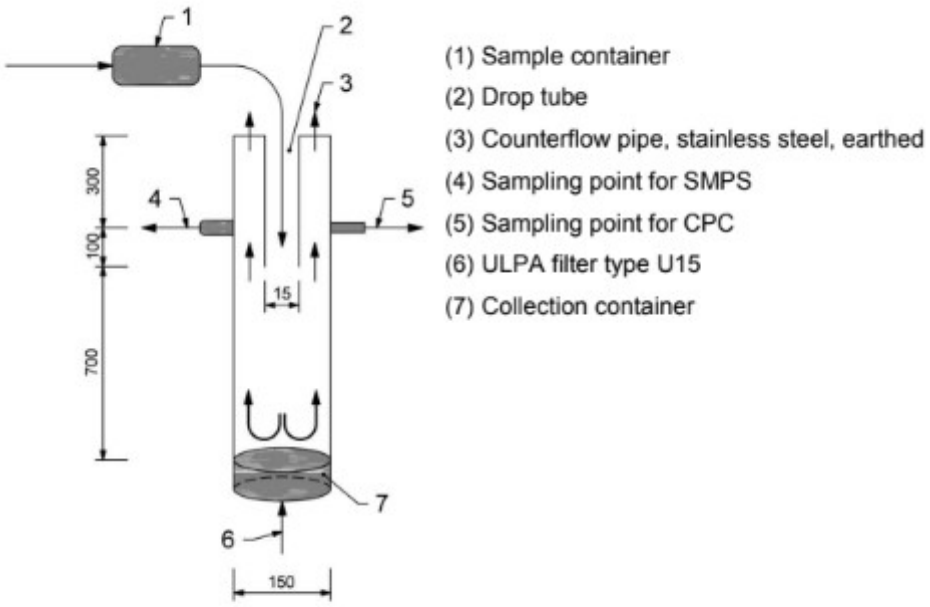

Figure S 7: Drop tube apparatus *(IGF Bochum, with permission).*

SMPS – Scanning Mobility Particle Sizer, CPC – Condensation Particle Counter, ULPA filter – Ultra Low Particulate Air filter.

SMPS and APS measure particle size, CPC serves as detector to quantify the number concentration.

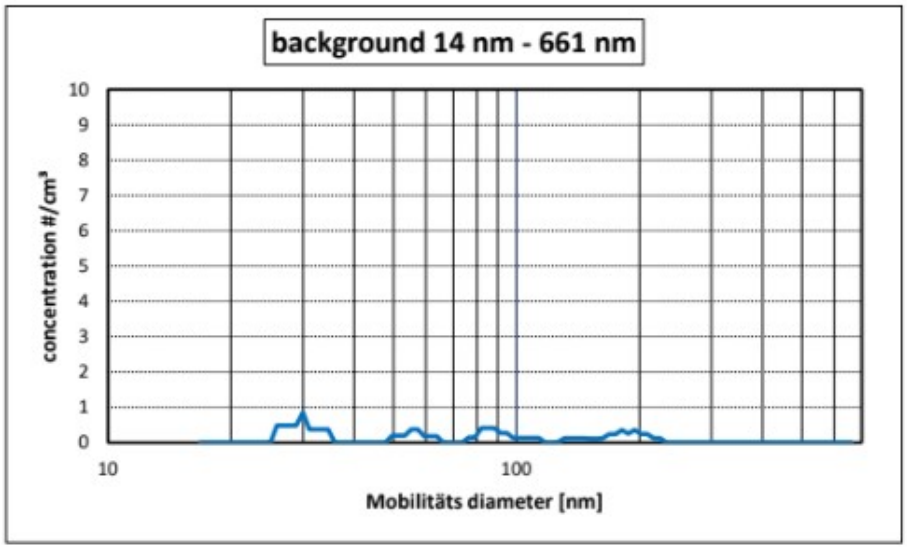

Figure S 8: SMPS[1] purified air, background.

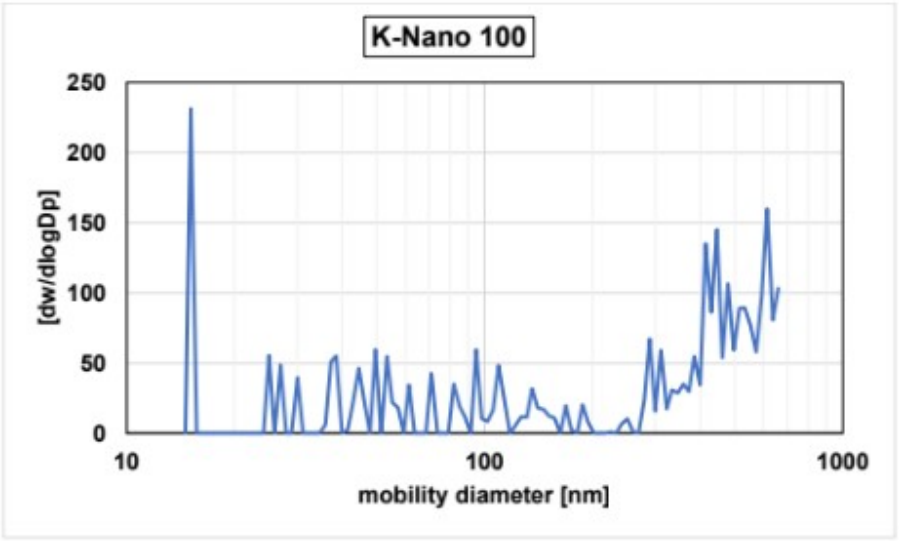

Figure S 9: SMPS for K-Nanos 100.

Table S 7: Numerical results SMPS for K-Nanos 100, n=3.

Range [nm]	Mean [nm]	Concentration [particles/cm³]
14 - 100	43.5	15.0
100 - 661	436.1	9.8
14 - 661	310.9	24.8

[1] SMPS – Scanning Mobility Particle Sizer

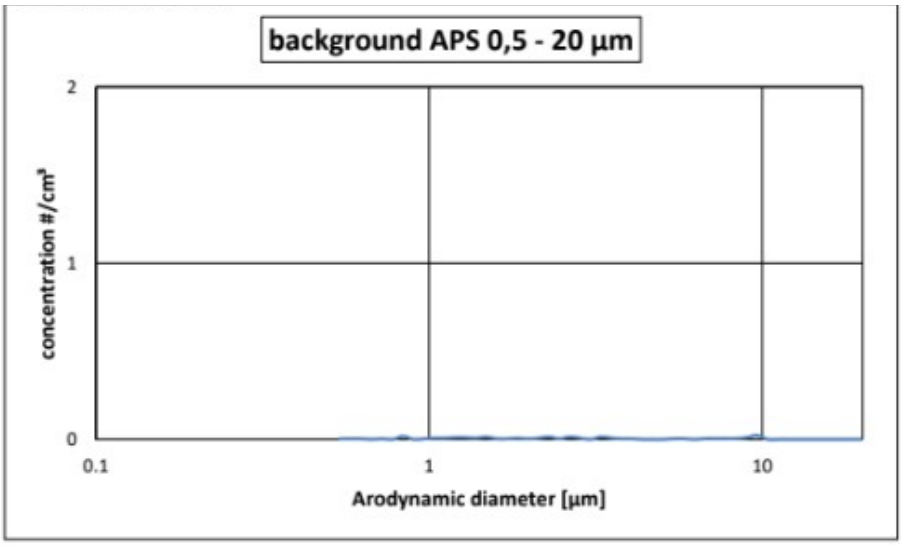

Figure S 10: APS[2] purified air, background.

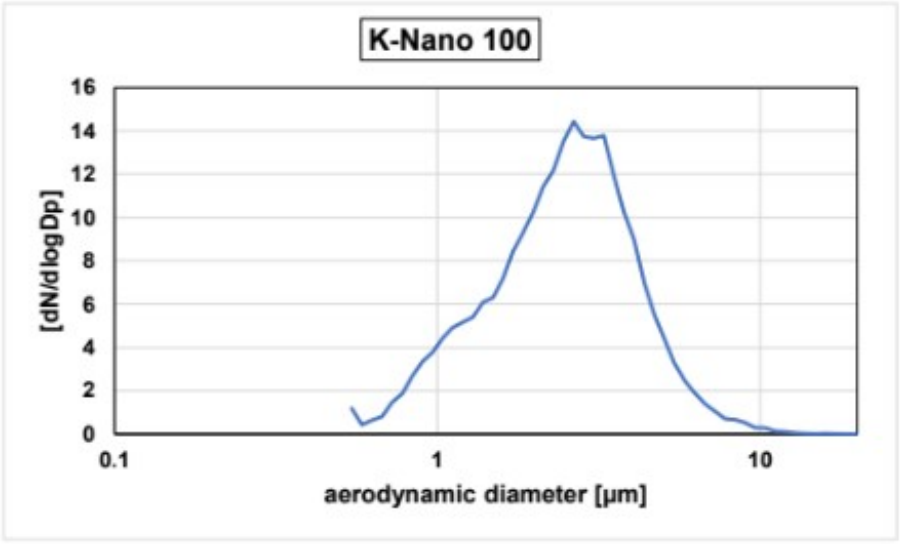

Figure S 11: APS for K-Nanos 100.

Table S 8: Numerical results APS for K-Nanos 100, n=3.

Range [µm]	Mean [µm]	Concentration [particles/cm³]
0.5 - 20	2.3	2.61

[2] Aerodynamic Particle Sizer

Figure S 12: Vibrating feeder with product.

Image taken by IGF Bochum (2022), commissioned by KKPC.

Figure S 13: Product in container after going through drop tube.

Image taken by IGF Bochum (2022), commissioned by KKPC.

ABBREVIATION

BET – Brunauer, Emmett and Teller Method

APS – Aerodynamic Particle Sizer

CoV – Coefficient of Variation

ICP-OES – Inductively Coupled Plasma Optical Emission Spectroscopy

MWCNTs – Multi-Walled Carbon Nanotubes

NMP – N-Methyl-2-pyrrolidone (solvent)

SD – Standard Deviation

SEM – Scanning Electron Microscopy

SMPS – Scanning Mobility Particle Sizer

TEM – Transmission Electron Microscopy