

Supplementary Information

Improved Functionalization of Multiwalled Carbon Nanotubes in Ultra-Low Acid Volume: Effect of Solid/Liquid Interface

Vinícius G. Castro,^{a,b} Ingrid B. Costa,^a Felipe S. Medeiros,^{a,b} Éder J. Siqueira,^a Alexander H. Kasama,^c Kátia C. S. Figueiredo,^{a,b} Rodrigo L. Lavall^a and Glaura G. Silva *,^a

^aDepartamento de Química, Instituto de Ciências Exatas, Universidade Federal de Minas Gerais, 31270-901 Belo Horizonte-MG, Brazil

^bDepartamento de Engenharia Química, Escola de Engenharia, Universidade Federal de Minas Gerais, 31270-901 Belo Horizonte-MG, Brazil

^cCentro de Pesquisas Leopoldo Américo Miguez de Mello (CENPES), Petrobras, 21941-598 Rio de Janeiro-RJ, Brazil

Table S1. Elemental analysis: ratio of each element for the functionalized MWCNTs

Treatment	C / mass%	O / mass%	H / mass%	N / mass%	S / mass%
50 °C, 90 min, 176 mL, with OCFs	87.1 ± 0.1	6.8 ± 0.1	0.3 ± 0.0	< 0.3	0.4 ± 0.0
70 °C, 20 min, 176 mL, with OCFs	86.0 ± 0.3	7.1 ± 0.1	0.4 ± 0.1	< 0.3	< 0.3
70 °C, 20 min, 88 mL, with OCFs	87.4 ± 0.3	6.6 ± 0.1	0.4 ± 0.2	< 0.3	0.3 ± 0.1
70 °C, 20 min, 44 mL, with OCFs	86.9 ± 0.2	7.5 ± 0.8	0.4 ± 0.1	< 0.3	< 0.3
70 °C, 20 min, 20 mL, with OCFs	89.0 ± 0.1	5.2 ± 0.3	< 0.3	< 0.3	0.4 ± 0.1
50 °C, 90 min, 176 mL, without OCFs	90.3 ± 0.1	4.5 ± 0.0	0.3 ± 0.0	< 0.3	< 0.3
70 °C, 20 min, 176 mL, without OCFs	87.1 ± 0.1	6.8 ± 0.1	< 0.3	< 0.3	< 0.3
70 °C, 20 min, 88 mL, without OCFs	90.8 ± 0.2	4.3 ± 0.1	0.3 ± 0.0	< 0.3	< 0.3
70 °C, 20 min, 44 mL, without OCFs	90.2 ± 0.2	3.8 ± 0.0	0.4 ± 0.1	0.3 ± 0.1	< 0.3
70 °C, 20 min, 20 mL, without OCFs	92.4 ± 0.9	4.0 ± 0.3	< 0.3	< 0.3	< 0.3

OCFs: oxygenated carbonaceous fragments.

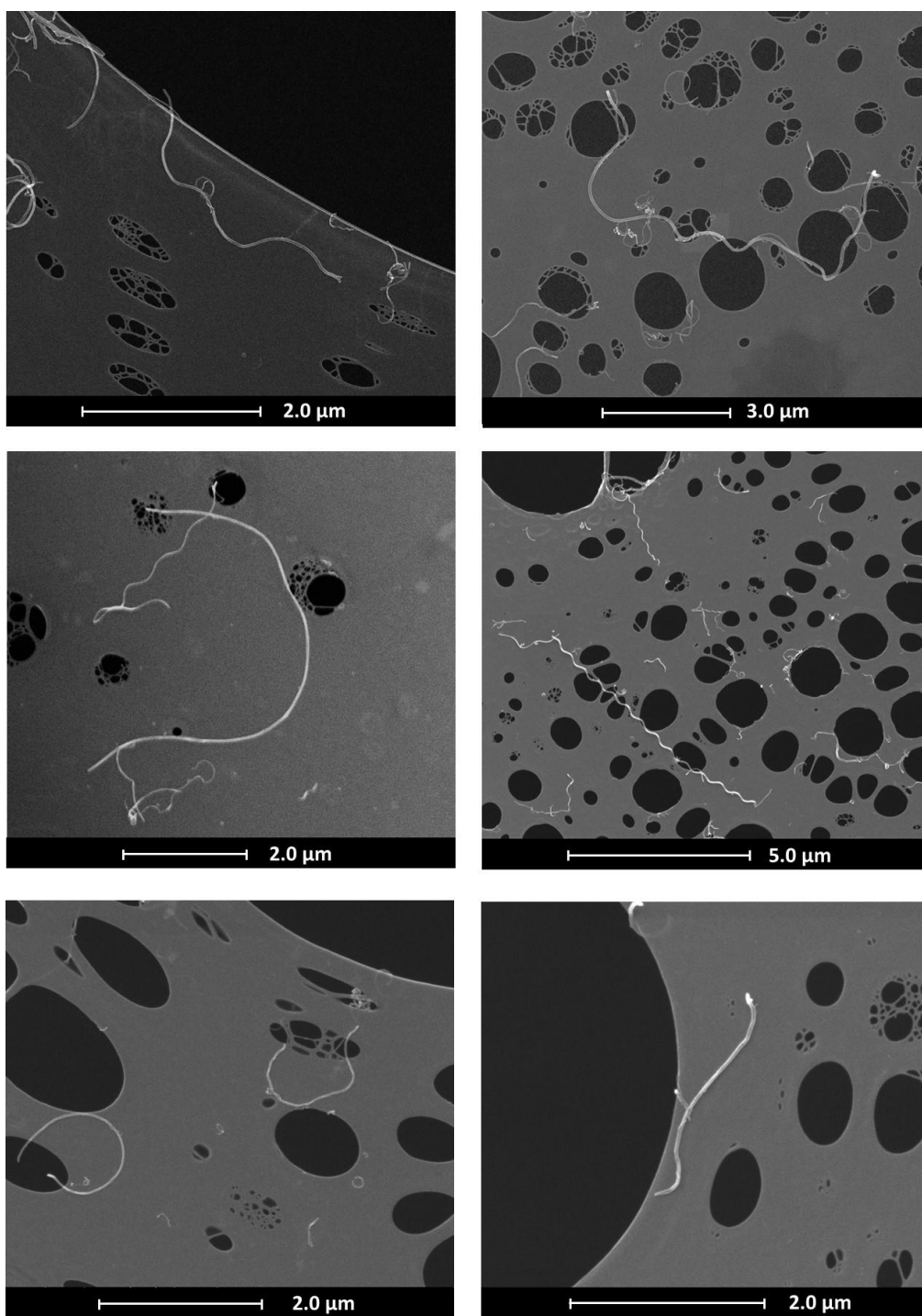


Figure S1. SEM images of MWCNTs after functionalization at 70 °C, 20 min and 44 mL.

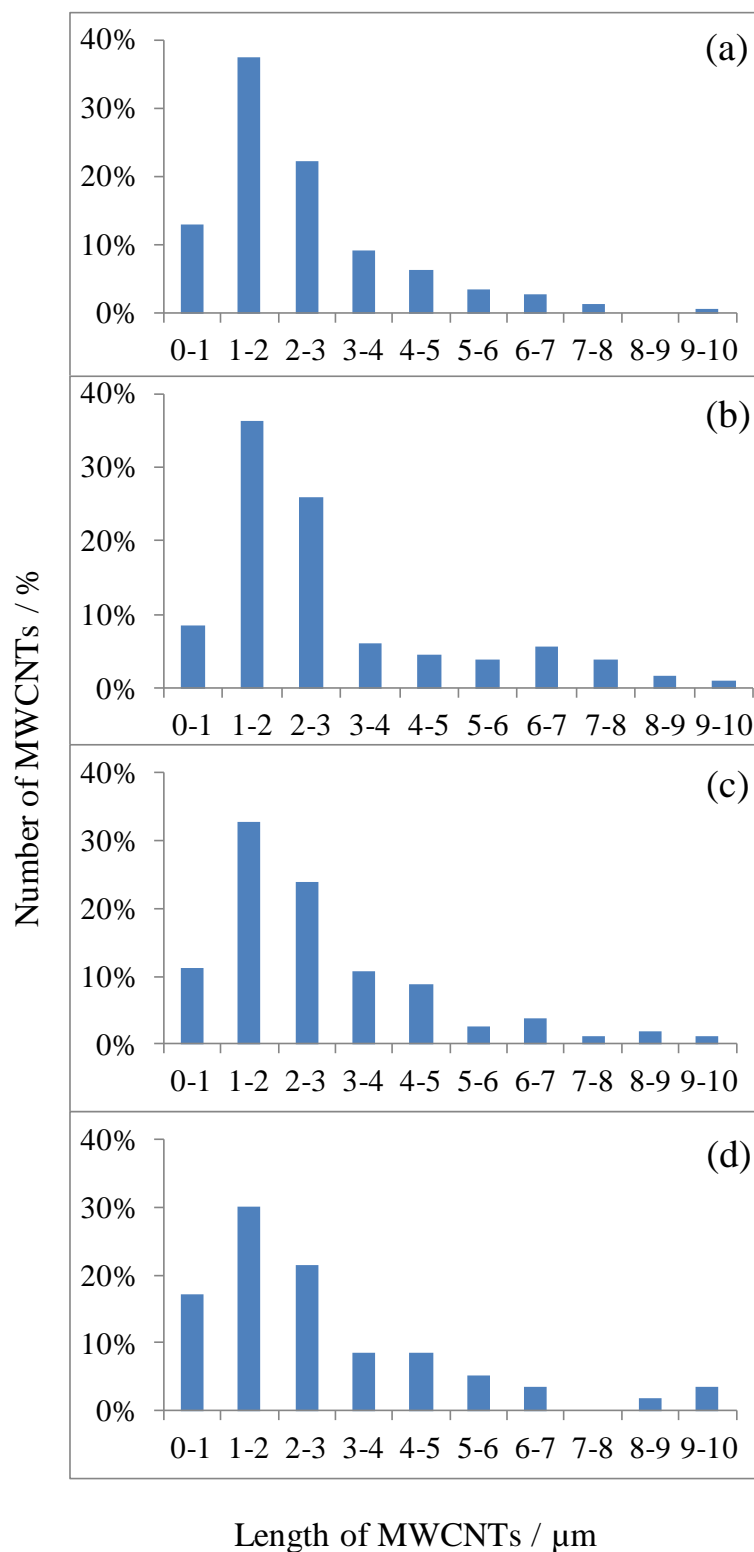


Figure S2. Length distribution of MWCNTs after four treatments on the same conditions: 70 °C, 20 min and 44 mL. (a) 139 measured MWCNTs, arithmetic length mean of 2.6 μm , weighted length mean of 4.4 μm ; (b) 170 measured MWCNTs, arithmetic length mean of 3.0 μm , weighted length mean of 4.7 μm ; (c) 148 measured MWCNTs, arithmetic length mean of 2.8 μm , weighted length mean of 4.4 μm ; (d) 111 measured MWCNTs, arithmetic length mean of 2.6 μm , weighted length mean of 4.4 μm .

The average MWCNT length was determined from both arithmetic and weighted means. Equation S1 shows the calculation used for arithmetic mean, which considers the same contribution of all measured MWCNTs, regardless of its length.

$$\text{Arithmetic mean} = \frac{\sum n_i L_i}{\sum n_i} \quad (\text{S1})$$

where n_i is the number of MWCNTs in each length interval i and L_i is the length of MWCNTs in each interval i .

Equation S2 was used for the determination of weighted mean, which considers a greater contribution of longer MWCNTs. This equation guarantees a better representation for the average value of length for the bulk sample, since longer MWCNTs involve a greater mass and volumetric contribution in comparison to shorter ones.

$$\text{Weighted mean} = \frac{\sum n_i L_i^2}{\sum n_i L_i} \quad (\text{S2})$$

Therefore, the arithmetic mean is an important result due to its more common use, but the weighted mean is more relevant for a quantitative analysis of MWCNT length and for its applications.

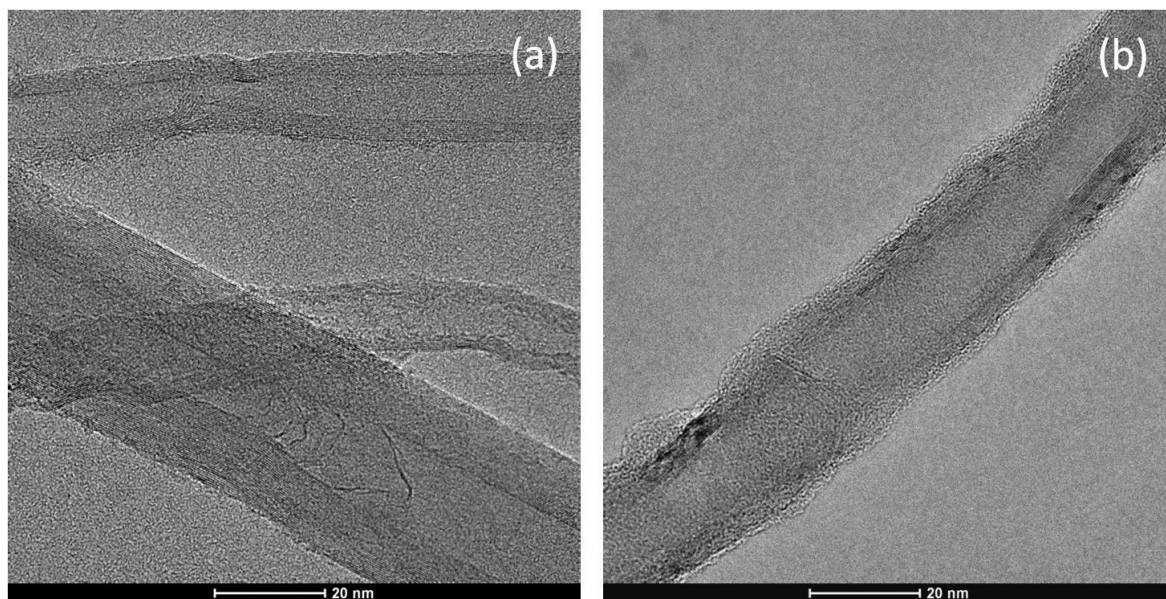


Figure S3. Extra TEM images of (a) pristine MWCNTs and (b) functionalized MWCNTs at 70 °C, 20 min and 44 mL of acid volume.

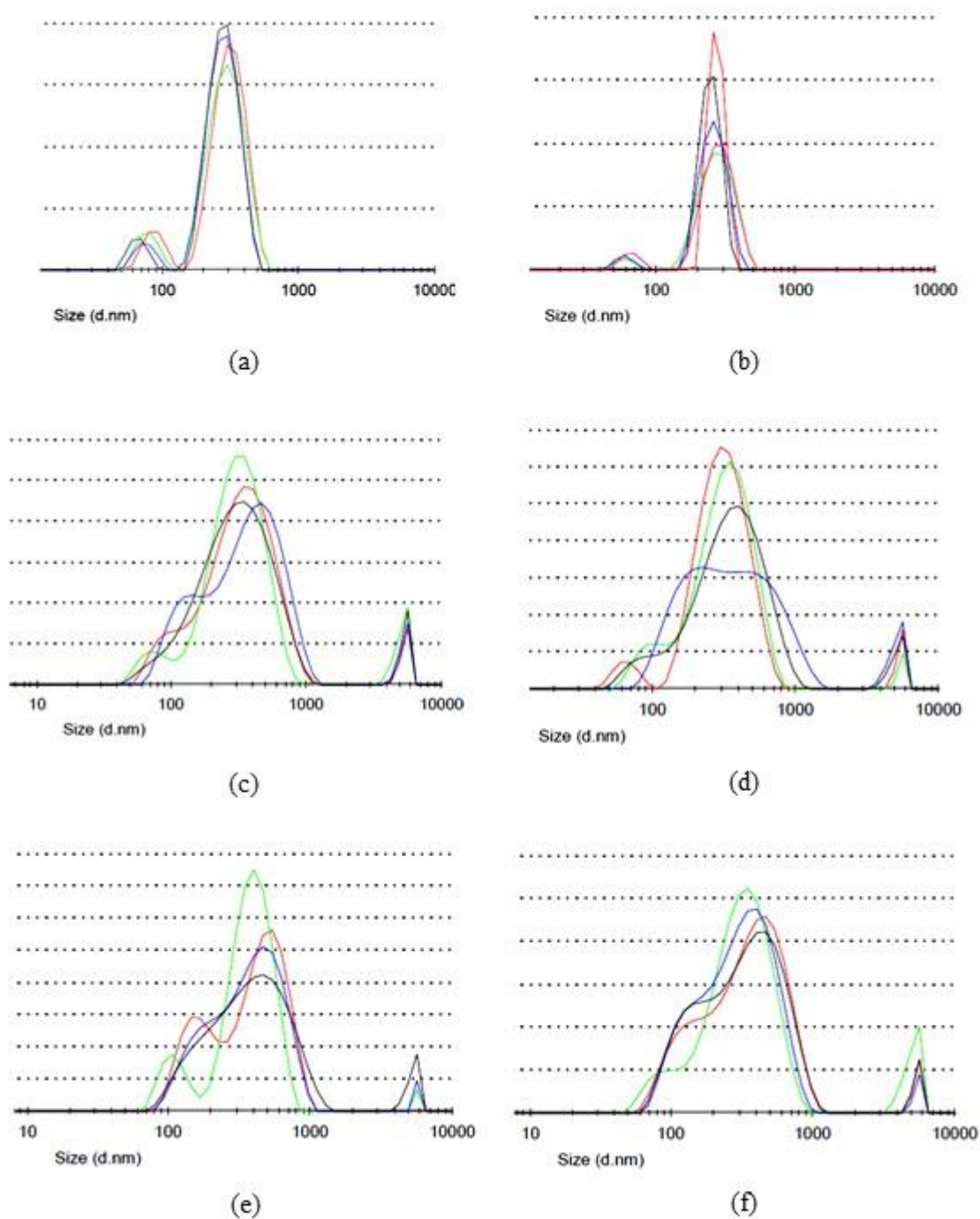


Figure S4. Size distribution (hydrodynamic diameter) obtained from DLS measurements for MWCNTs treated at 70 °C, 20 min and 44 mL of acid volume (a) with and (b) without OCFs; 70 °C, 20 min and 176 mL of acid volume (c) with and (d) without OCFs; 70 °C, 20 min and 20 mL of acid volume (e) with and (f) without OCFs.