Electronic Supplementary Information (ESI)

for

From the Inside-Out: Leached of Metal Impurities in Multiwall Carbon Nanotubes for Purification or Electrocatalysis

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1. Supplementary data

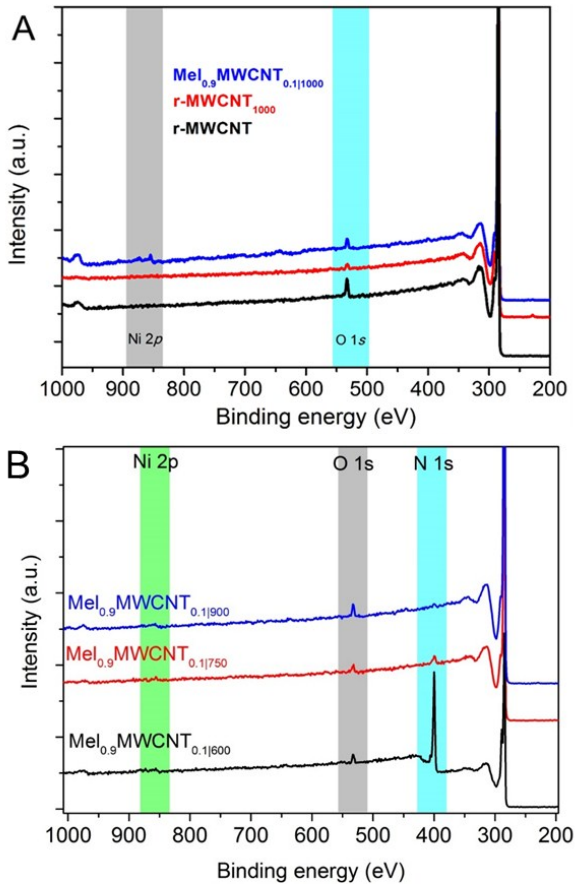

Figure S1. XPS Survey scan of (A) r-MWCNT, r-$MWCNT_{1000}$ and $Mel_{0.9}MWCNT_{0.1|1000}$, and (B) $Mel_{0.9}MWCNT_{0.1|T}$ pyrolyzed at various temperatures.

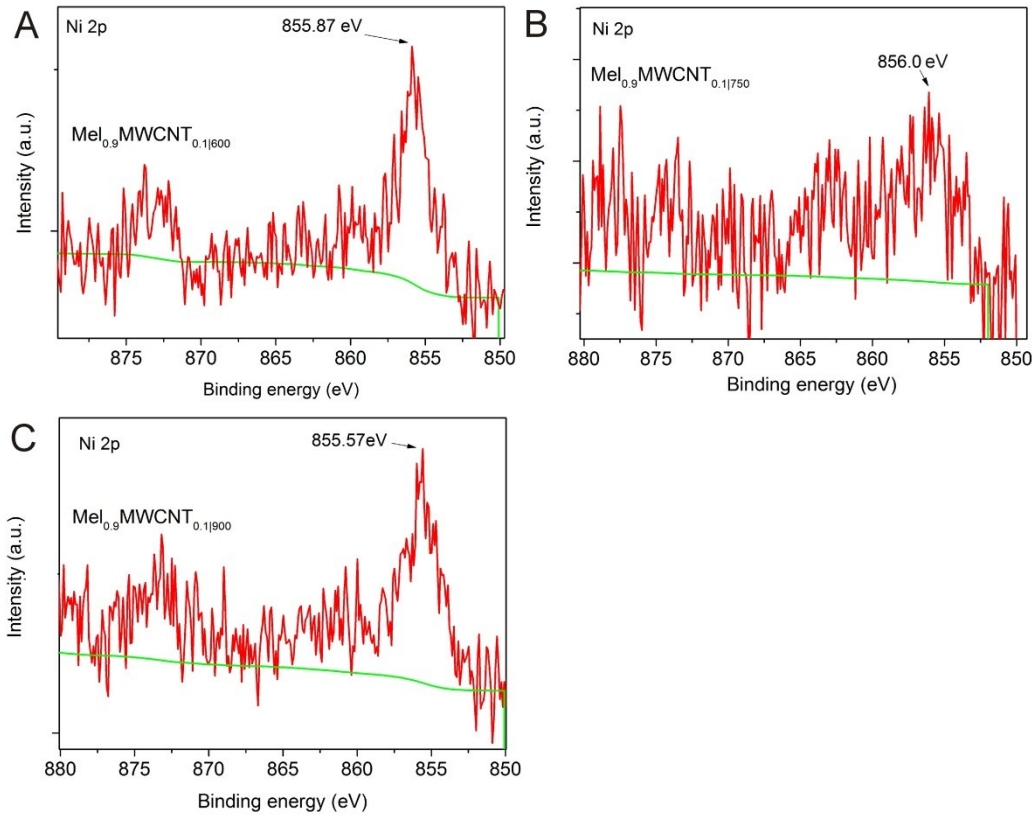

Figure S2. High resolution Ni 2*p* XPS scan for $Mel_{0.9}MWCNT_{0.1|T}$ pyrolyzed at various temperatures (A) 600°C, (B) 750°C and (C) 900°C.

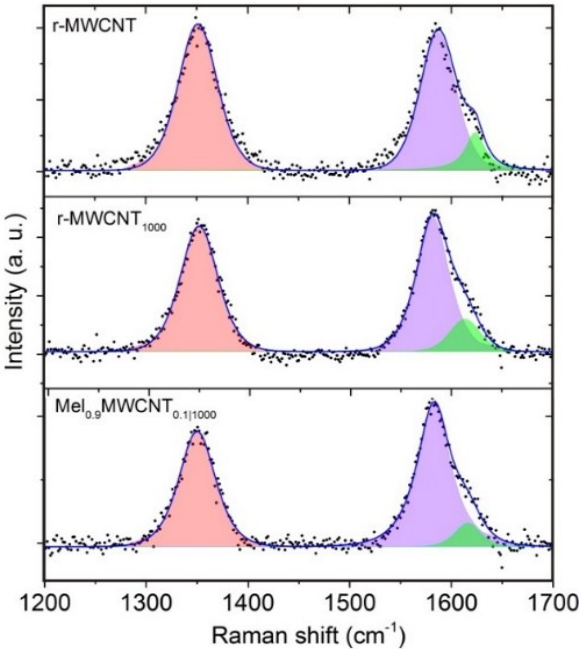

Figure S3. Raman spectrum of $Mel_{0.9}MWCNT_{0.1|1000}$, r-$MWCNT_{1000}$ and r-MWCNT, the red and green shaded peaks correspond to defect sites, the purple shaded peaks correspond to sp^2-hybridized C-C bonds.

Table S1. Detection of metal impurities amount (Ni, Fe and Co) by ICP-MS.

Sample	Purification procedure	Metal content (ppm)			
		Ni	Fe	Co	Total
raw MWCNT	As received	9907	102	71	10080
r-MWCNT	5 M HCl, 120°C	9049	104	66	9219
r-$MWCNT_{1000}$	1000°C, 5 M HCl, 120°C	5204	19	44	5267
$Mel_{0.9}MWCNT_{0.1\|1000,}$[1]	1000°C, 5 M HCl, 120°C	901	18	7	926
$Mel_{0.9}MWCNT_{0.1\|1000,}$[2]	1000°C, 5 M HCl, 120°C	703	14	7	724
$Mel_{0.9}MWCNT_{0.1\|1000,}$[3]	1000°C, 5 M HCl, 120°C	294	13	<1	308
$Mel_{0.9}MWCNT_{0.1\|1000,}$[4]	1000°C, 5 M HCl, 120°C	107	13	<1	121

N.B: $Mel_{0.9}MWCNT_{0.1|1000,}^{y}$, *y* corresponds to the number of melamine and HCl purification cycles. The uncertainty for ICP-MS analysis is ± 1.00%.

Table S2. The amount of mass yield percentage of pyrolysed $Mel_{(1-x)}MWCNT_{x|T}$ and its associated N and Ni contents by XPS detection.

Sample	Mass Yield (%)	N 1*s* (at.%)	Ni 2*p*(at.%)
$Mel_{0.9}MWCNT_{0.1\|1000}$	96.58	N.D.	0.10
$Mel_{0.9}MWCNT_{0.1\|900}$	96.15	0.47	0.21
$Mel_{0.9}MWCNT_{0.1\|750}$	98.11	1.66	0.12
$Mel_{0.9}MWCNT_{0.1\|600}$	200.87	25.74	0.15

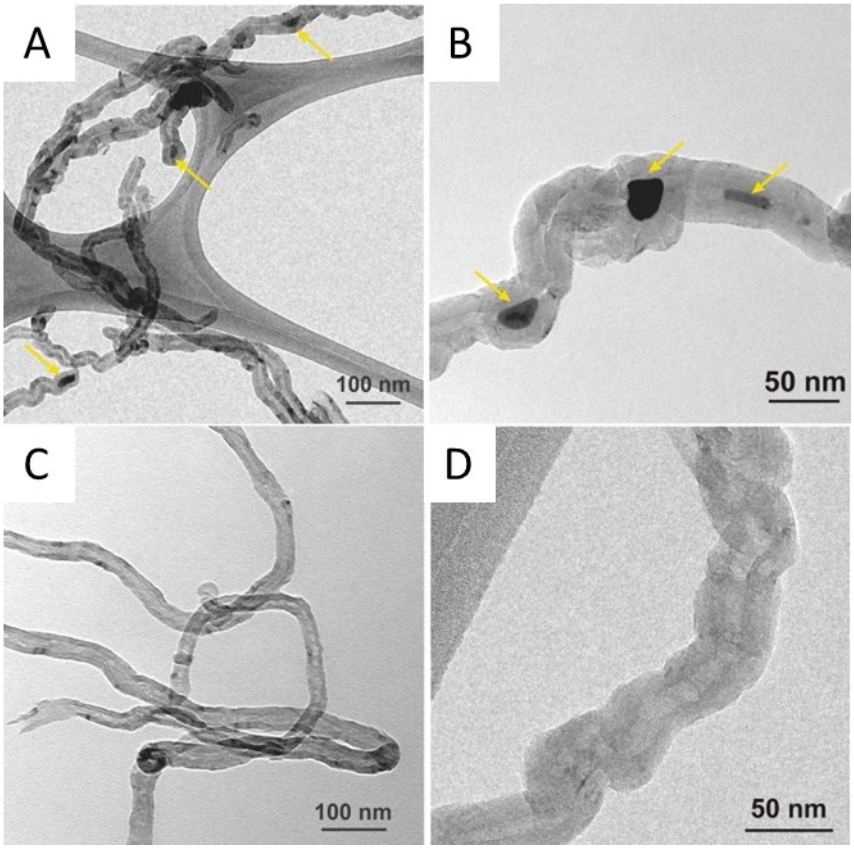

Figure S4. Transmission electron microscopy (TEM) characterization of MWCNTs, (A,B) r-$MWCNT_{1000}$, (C,D) acid purified $Mel_{0.9}MWCNT_{0.1|1000}$. The yellow arrows in the figures point to heavier elements as indicated by the darker contrast in the images.

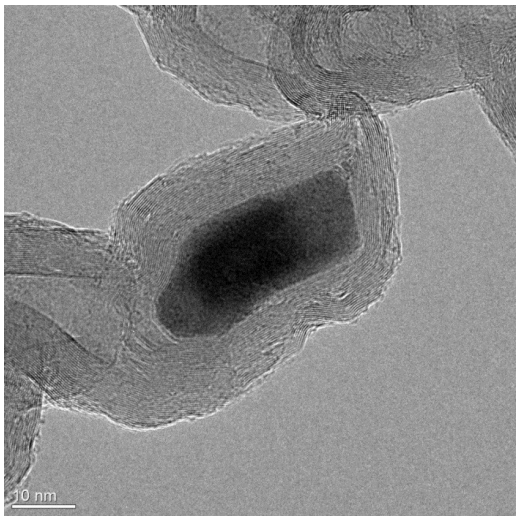

Figure S5. High resolution TEM shows the full encapsulation of Ni particles in MWCNT.

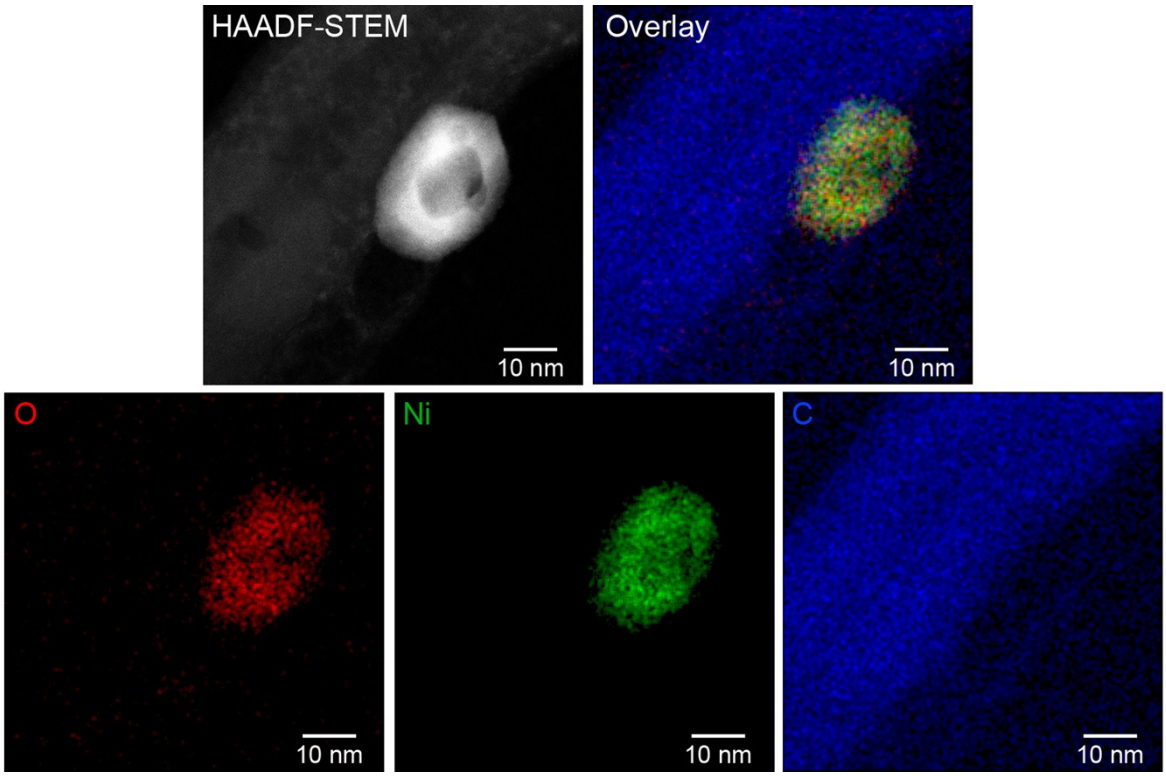

Figure S6. High-angle annular dark-field (HAADF)-STEM image and STEM-EDS elemental mapping of the leached metallic impurities *via* the leak mode, the same CNT as shown in Fig 2B. The false color overlay and maps of O K-edge (red), Ni K-edge (green) and C K-edge (blue) clearly show a nickel oxide nanoparticle on the surface of a MWCNT.

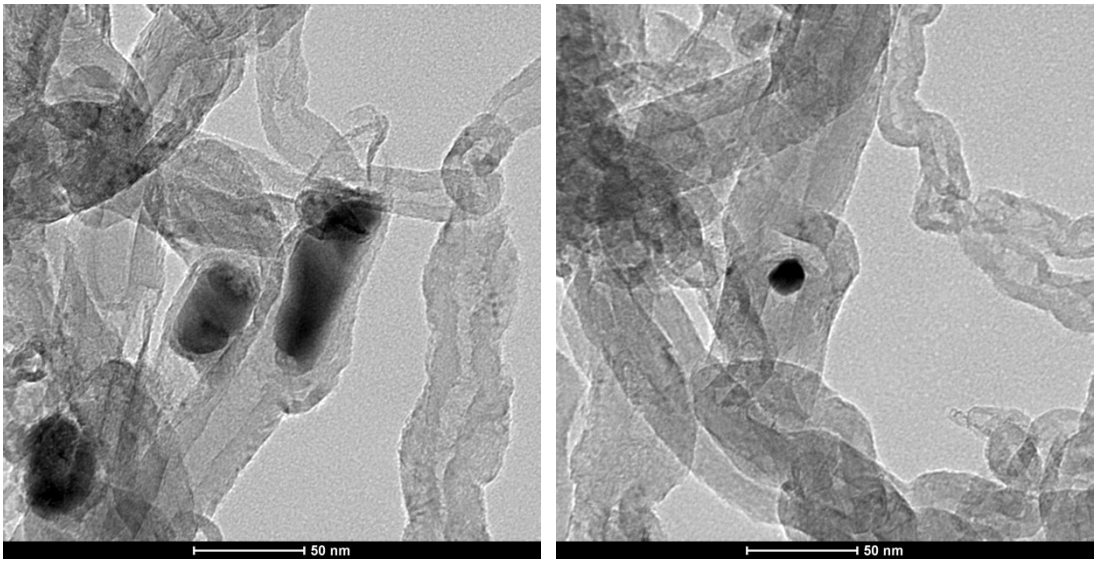

Figure S7. The typical TEM images of the $Urea_{0.9}MWCNT_{0.1|1000}$