Electronic Supplementary Material (ESI) for Nanoscale.

SUPPORTING INFORMATION

Biodegradable multi-walled carbon nanotubes trigger anti-tumoral effects

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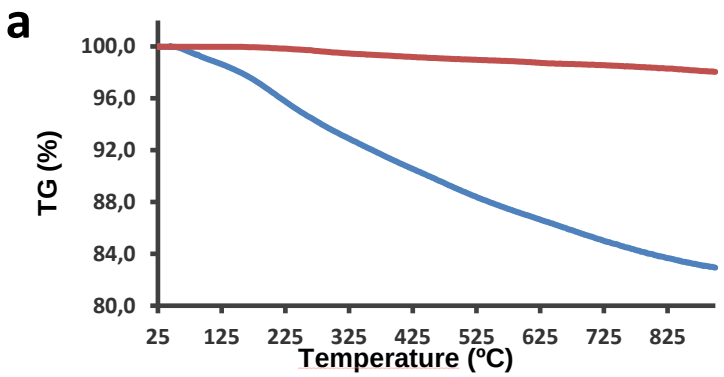
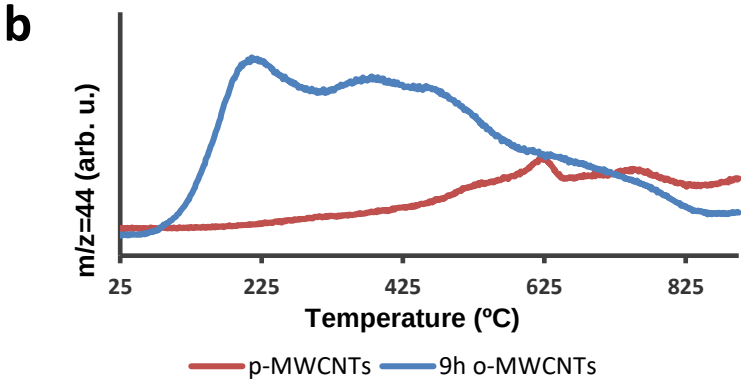

a
100,0
96,0
92,0
88,0
84,0
80,0
TG (%)
25
125
225
325
425
525
625
725
825
Temperature (ºC)


b
m/z=44 (arb. u.)
25
225
425
625
825
Temperature (ºC)
p-MWCNTs
9h o-MWCNTs

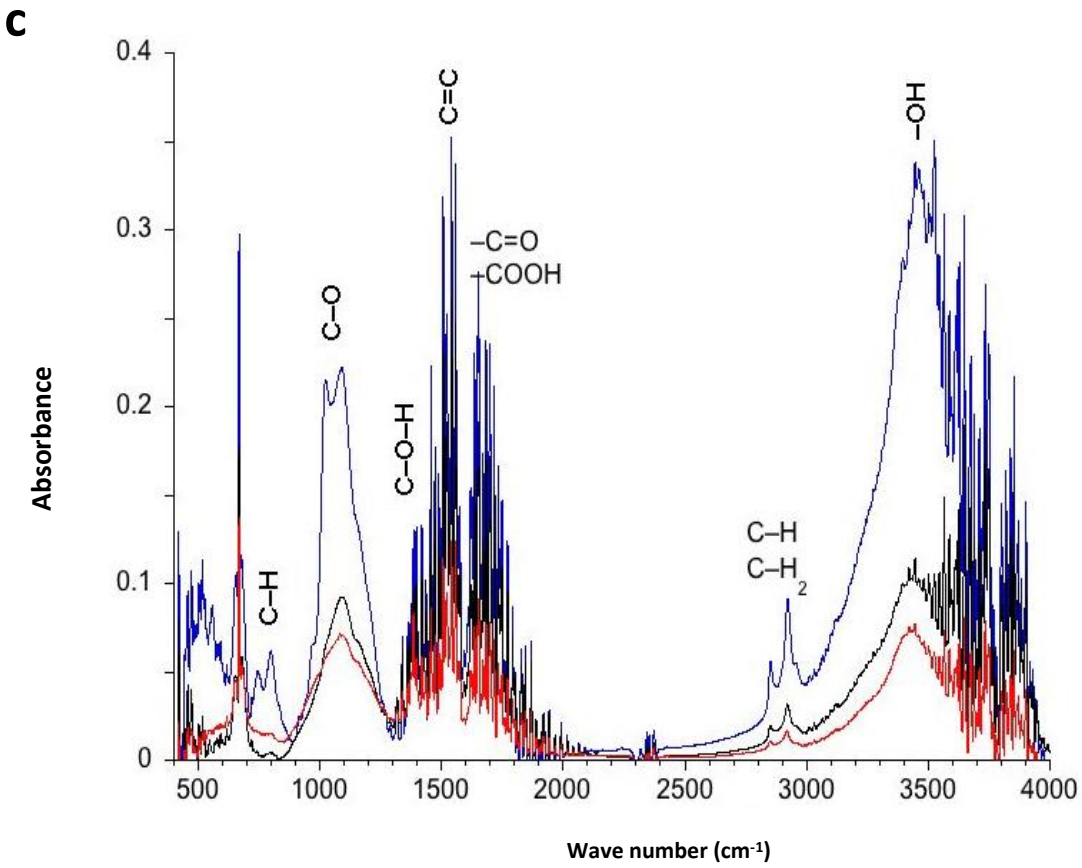

c
Absorbance
0.4
0.3
0.2
0.1
0
C=C
–C=O
+COOH
C–O
C–O–H
C–H
–OH
C–H
C–H2
500
1000
1500
2000
2500
3000
3500
4000
Wave number (cm-1)

Fig. S1. (a) Thermogravimetric analysis (TG) and **(b)** mass spectrometry signal corresponding to CO_2 (m/z=44) of the p-MWCNTs (red) and 9h o-MWCNTs (blue) samples under N_2 atmosphere. **(c)** FTIR absorption obtained for MWCNTs in a KBr pellet. The spectra take into account the MWCNTs concentration and pellet thickness. No smoothing treatment or signal subtraction of CO2 or H2O has been applied to the data. The IR absorption spectra were acquired on a Jasco LE4200 FTIR spectrophotometer by accumulating 264 scans at 4 cm^{-1} resolution in the range 400-4000 cm^{-1} . Samples were prepared by mixing the weighted MWCNTs with KBr. Infrared spectra were recorded on a Jasco LE4200 spectrophotometer. The concentration and thickness of the pellet was obtained into account in quantitative analysis. Neither correction for H_2O and CO_2 absorption nor smooth technique were used in our data. The MWCNTs concentrations were 2400 ug, 2400 µg and 1576 µg per gram for p-MWCNTs, 2h o-MWCNTs and 9h o-MWCNTs respectively gram of KBr, with pellet thicknesses of 350 µm, 280 um and 280 um, respectively.

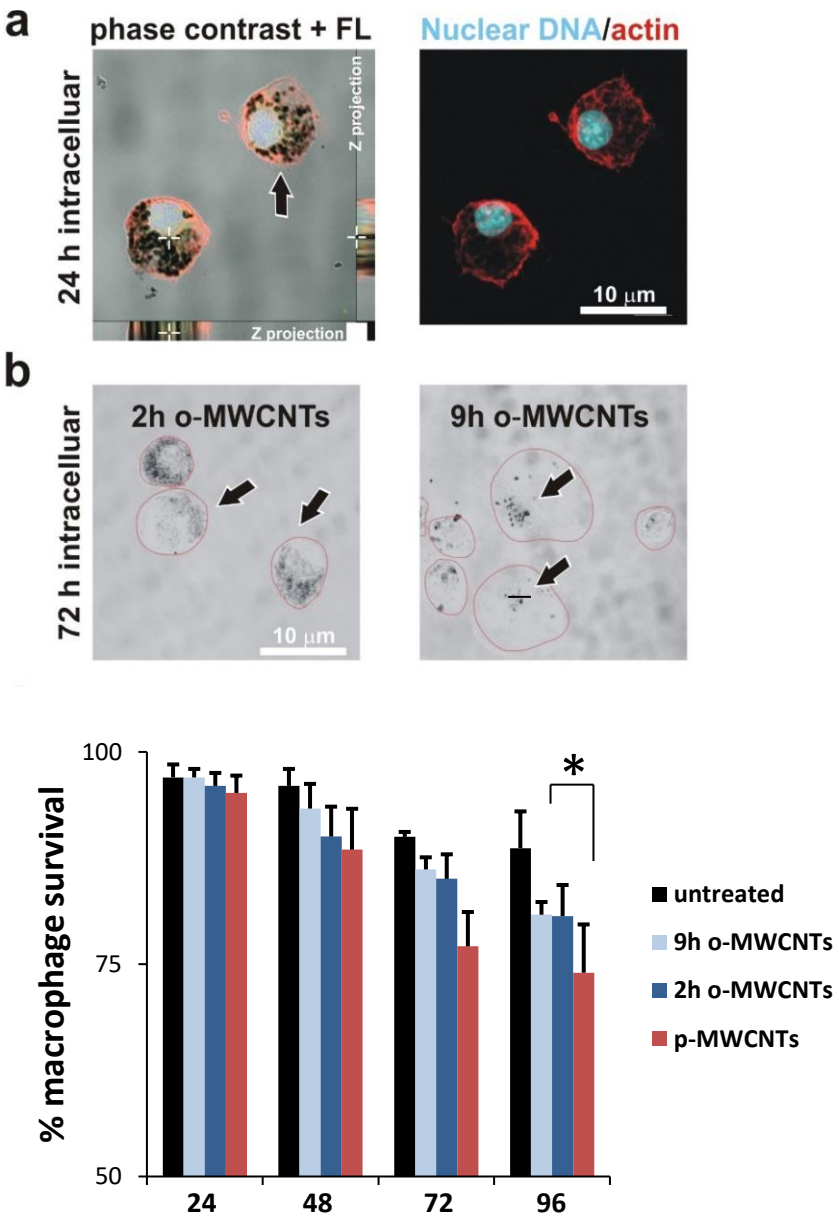


Fig. S2. o-MWCNT biodegradation and biocompatibility in cultured macrophages. (a) Phase contrast/fluorescent single Z projection images of macrophages exposed to 2h o-MWCNTs stained for actin (red channel) and nuclear DNA (blue channel). Crosses in Z-projection lateral images show representative intracellular carbon masses such as those analysed for the confocal Raman spectroscopic analysis. (**b**) Degradation observed in macrophages by phase contrast imaging. The cellular contour is highlighted by a red line. Black arrows indicate intracytoplasmic carbon aggregates inside phagosomes. (**c**) Macrophage survival after exposure to the 2 Vs. 9h o-MWCNTs compared to untreated and p-MWCNTs control cells. Longer oxidation times significantly improve macrophage survival to MWCNT exposure. Histogram represents automatic cell counts (t= 2.05; n= 4; *= $t_{.95}$).

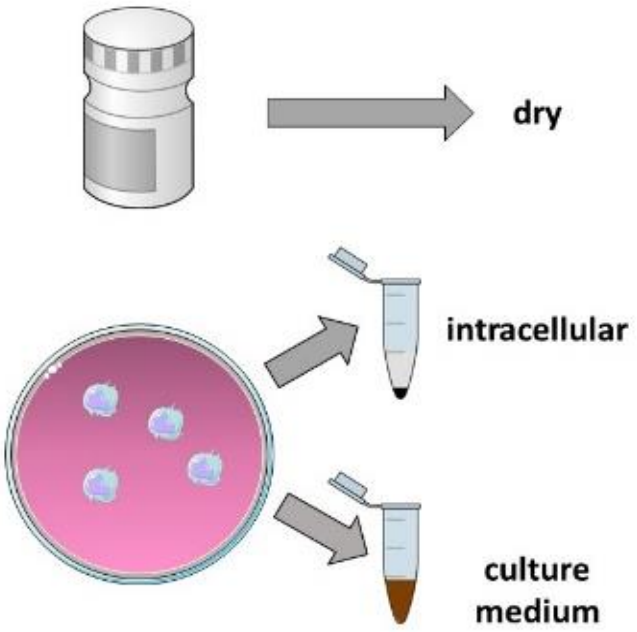


Fig. S3. MWCNTs types and isolation procedures. Dry MWCNTs correspond to the *as-prepared* MWCNTs.

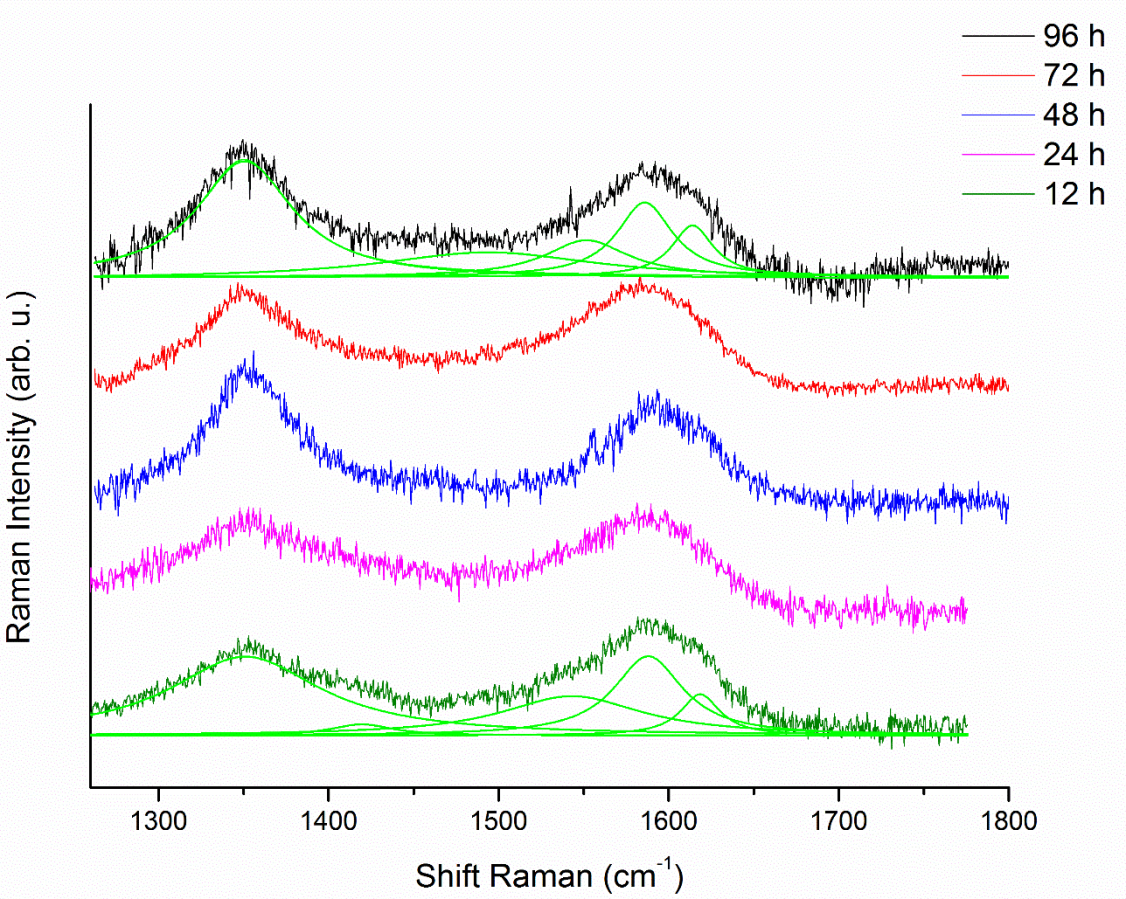


Figure S4. Representative Raman spectrograms used in the semi/quantification of the intracellular degradation of 9h o-MWCNT in macrophages (Figure 2b) including the Lorentzian curves (green) fitting used for peak area determination. Time points indicate the times of intracellular exposure to nanotubes. Changes in the signatures of the distinctive D and G bands in the wavelength range 1200-1800 cm-1 are observable.

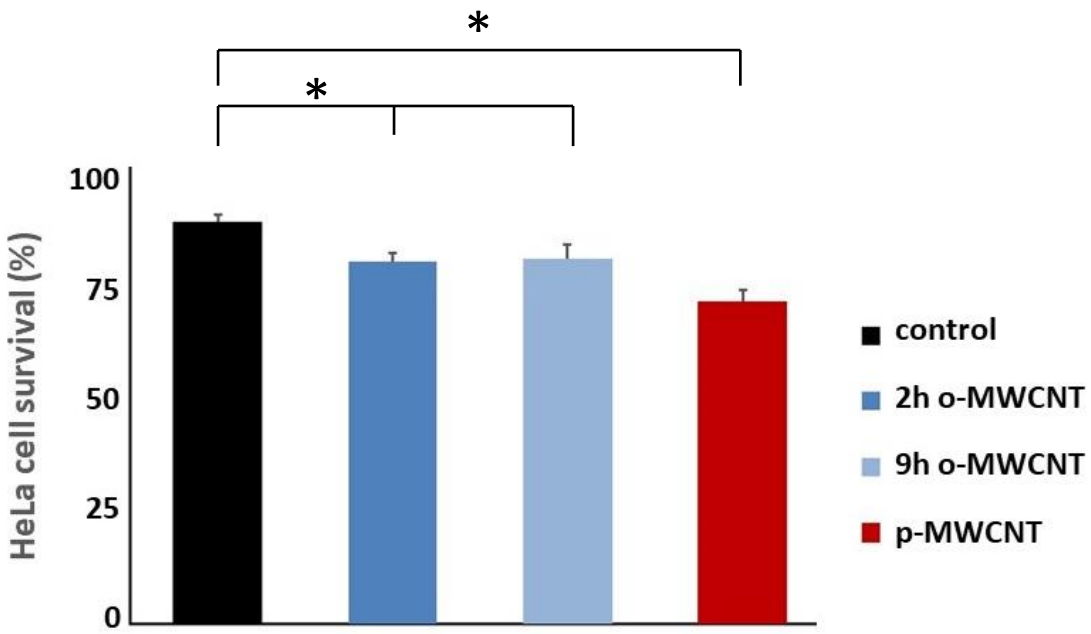


Fig. S5. Hela cell survival after exposure to 50 µg/mL nanotubes during 72 h. p-MWCNTs are more cytotoxic than 2h or 9h o-MWCNTs. (*t*= 1.6; *n*= 150; *= $t_{.95}$)

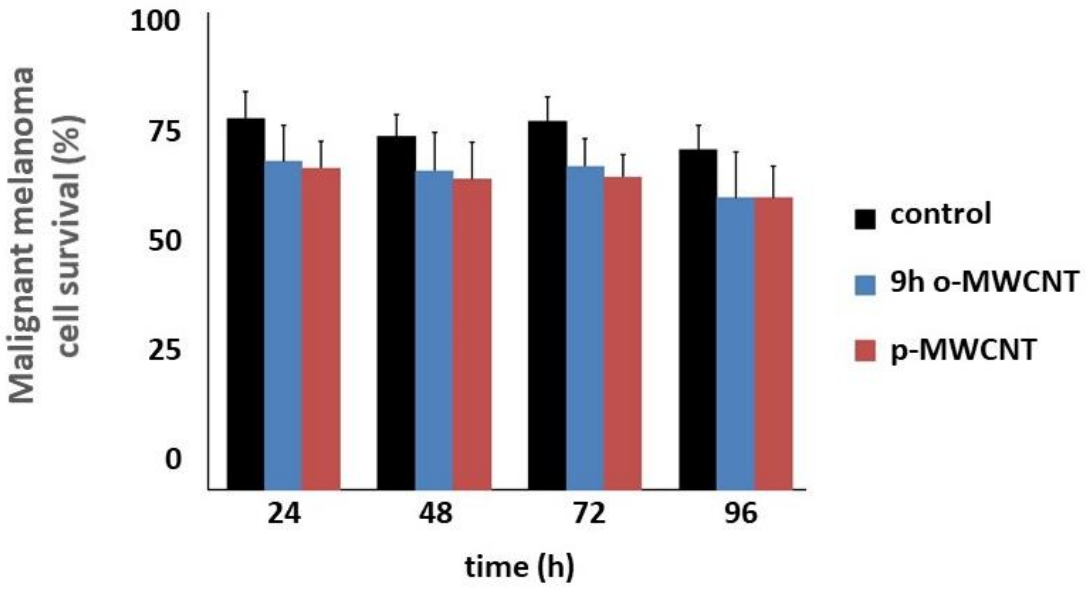


Fig. S6. Malignant melanoma cell survival after exposure to 100 µg/mL nanotubes during 24, 48, 72 and 96 h. At these doses, the cytotoxicity of p-MWCNTs is similar to that of 9h o-MWCNTs (no significant statistical differences are detected).