1. Acid-base titration of oxSWCNTs to determine the number of total acidic sites

**Material:**

SWCNTs-1: synthesized by laser ablation, donation from IFW Dresden.

SWCNTs-2: synthesized by CVD using iron (ferrocene as a catalyst), purchased from NanoLab, Inc., purity >95%, length 1–5 µm, Lot No. 90907).

**Method:**

First, an equivalent of 10 mg oxSWCNTs dispersed in water was ultrasonicated in a water bath for 15 min and treated with nitrogen for 10 min to degas CO₂. Next, 4 mL NaOH 10 mM was added and the mixture stirred for 2 h at room temperature. Subsequently, the mixture was filtered through 0.2 µm polycarbonate filters (Whatman Ltd.) and washed several times with deionised water. The filtrate was then titrated with HCL 10 mM until reaching the neutral point, as monitored by a pH meter. The volume required to reach the neutral point was subtracted from the initial volume of NaOH used to obtain the volume of NaOH which has reacted with acidic sites of the oxSWCNTs. For example, if 2.5 mL NaOH were required to reach the neutral point, 1.5 mL NaOH 10 mM have reacted with the oxSWCNTs, which corresponds to an amount 15 µmol of NaOH, and therefore, 15 µmol acidic sites per 10 mg oxSWCNTs (or 1.5 µmol acidic sites per mg oxSWCNTs). The measurement was repeated three times for each sample and the average calculated.

Under the assumption that the oxidised nanotubes are solely composed of carbon, the mole percentage of total acidic sites can be calculated: 1 mg oxSWNTs consists of 83.33 µmol carbon atoms (obtained through division by the molecular weight of carbon). The mole
percentage of total acidic sites in this example would therefore be 1.5 µmol/83.33 µmol or 1.8%

Results & Discussion:
Figure S1 shows an exemplary titration curve of oxSWNTs-1 after incubation with a known amount of NaOH and back titration of the unreacted NaOH with HCl. The neutral point in this case was reached after addition of 2.45 mL HCl. Table S1 shows the results for both samples:

Table S1 Results of the acid-base titrations

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<thead>
<tr>
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<th>oxSWCNTs-1</th>
<th>oxSWCNTs-2</th>
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<tr>
<td>Amount of total acidic sites per mg oxSWCNTs</td>
<td>1.533 µmol</td>
<td>1.4 µmol</td>
</tr>
<tr>
<td>Mole percentage of total acidic sites</td>
<td>1.84%</td>
<td>1.8%</td>
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Figure S1 a) Titration curve of oxSWCNTs-1, b) Derivative of titration curve to determine the volume used to reach the neutral point.
The values obtained for the two samples (one synthesised by laser ablation and the other by CVD) were quite similar and in excellent agreement with the literature. For example, Hu et al. tested three different commercial oxSWCNT samples and obtained molar percentages of total acidic sites between 1.0% and 2.7% using acid-base titration [1], whereas Blanchard et al. have obtained molar percentages between 1.45% - 1.93% for oxSWCNT samples oxidized by a method similar to the one used in this work using X-ray photoelectron spectroscopy (XPS) [2].

2. Comparison of MTT and WST cytotoxicity assays to assess their interactions with carbon nanotubes

In order to compare the performance of the MTT and WST viability assay for carbon nanotube samples, we compared the results of three different samples; doxorubicin, PEGylated oxSWCNTs (oxSWCNT_{PEG}), and doxorubicin-loaded oxSWCNT_{PEG} (dox-oxSWCNT_{PEG}). Figure S2 shows that the MTT assay only led to false positive results for oxSWCNT_{PEG}, but not for dox-oxSWCNT_{PEG}, which indicated that the interaction of MTT with the CNT surface is reduced if the surface is already occupied with other molecules, e.g. doxorubicin. In order to avoid this problem entirely, we used the WST assay for all further experiments. Note that the results for doxorubicin only were also slightly different with the WST assay.
Figure S2 Dose-response curves obtained by the MTT assay (black columns) and WST assay (grey columns) for doxorubicin (a), oxSWCNT_{PEG} (b), and dox-oxSWCNT_{PEG} (c). The results differ significantly for oxSWCNT_{PEG}, but not for dox-oxSWCNT_{PEG}.

References:
