

## Supplementary Material:

# Structural Characterization of Graphene Oxide: Surface Functional Groups and Fractionated Oxidative Debris

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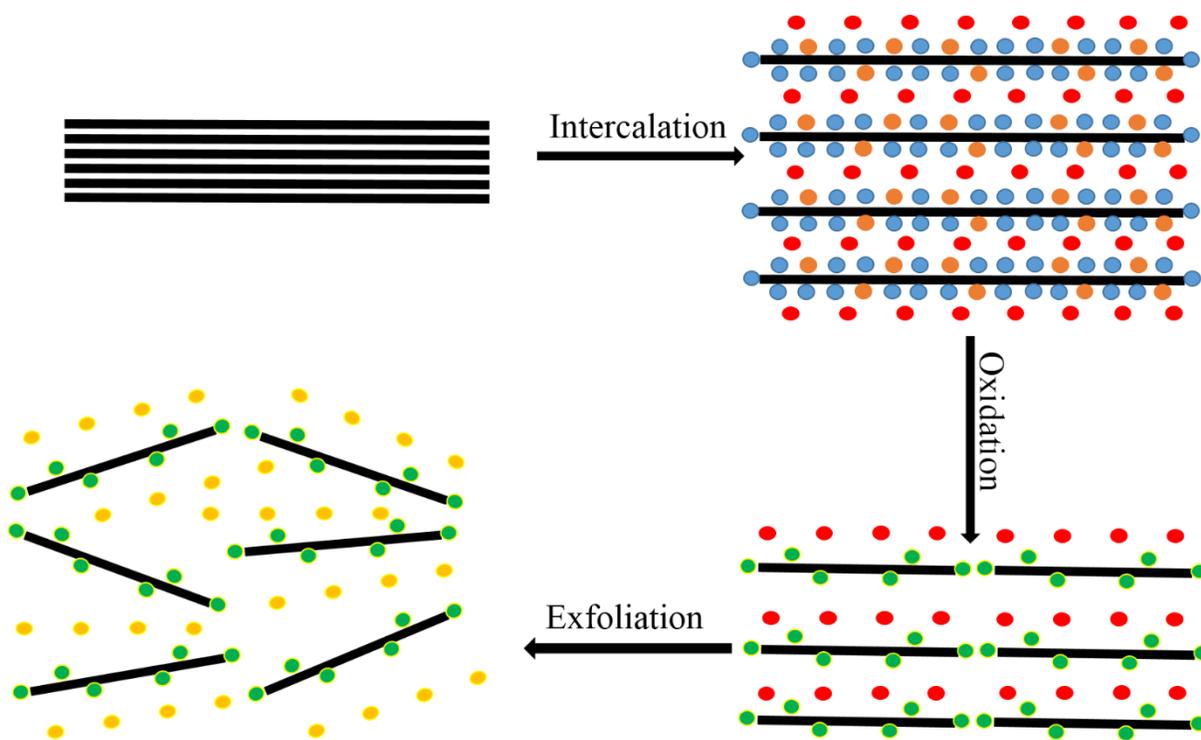
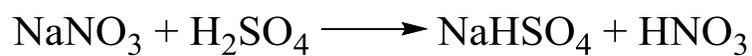
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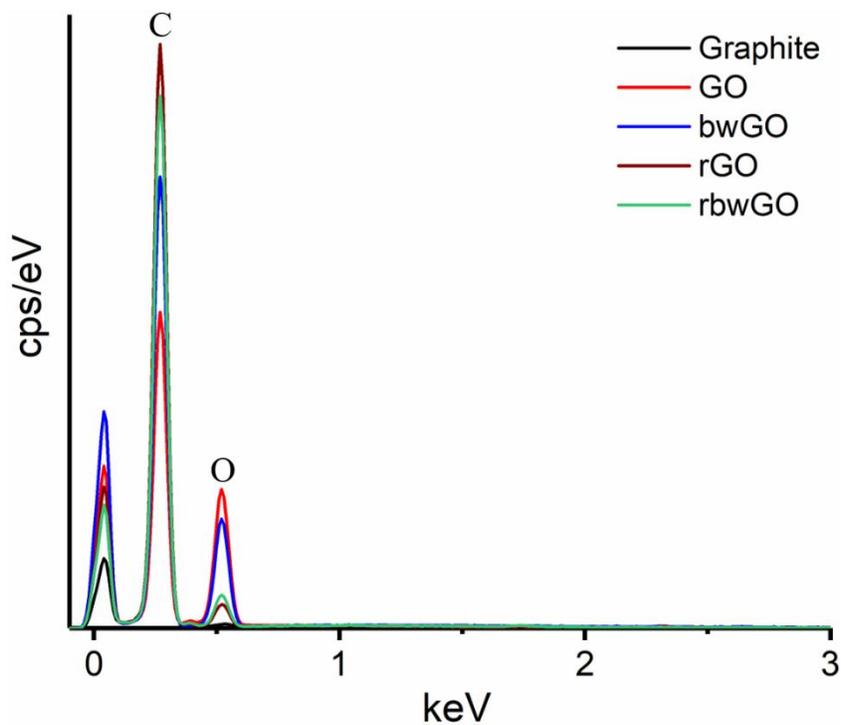
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**Figure S1.** Intercalation, oxidation and exfoliation of graphene oxide layers: ● - HSO<sub>4</sub><sup>-</sup>; ● - NO<sub>3</sub><sup>-</sup>; ● - Na<sup>+</sup>; ● - oxygen functionalities; ● - oxidative debris.



**Figure S2.** EDX results for graphene oxide samples.

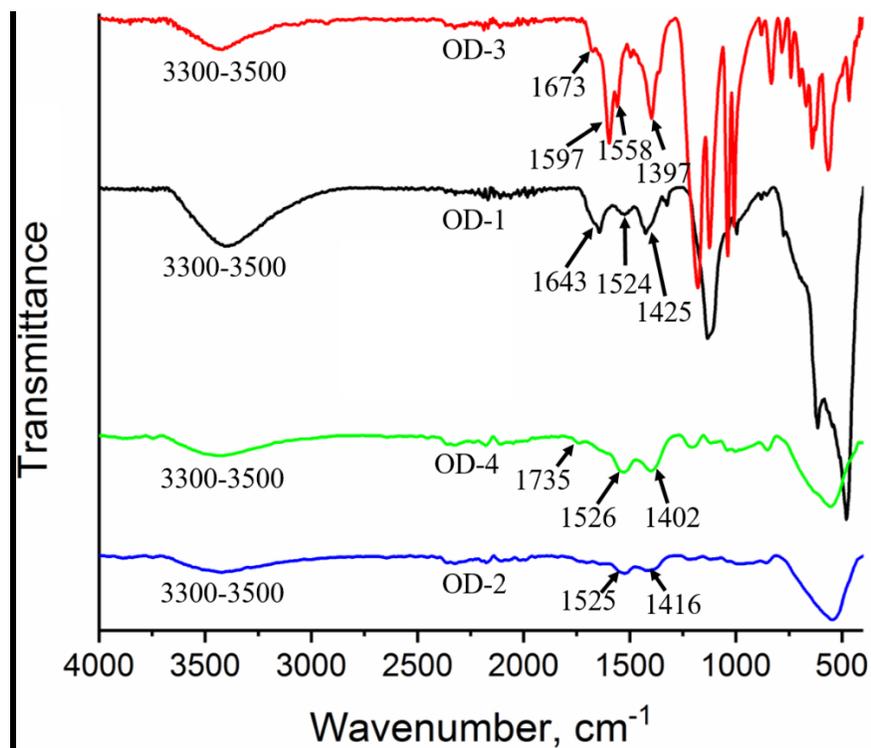


Figure S3. FTIR spectra of oxidative debris.

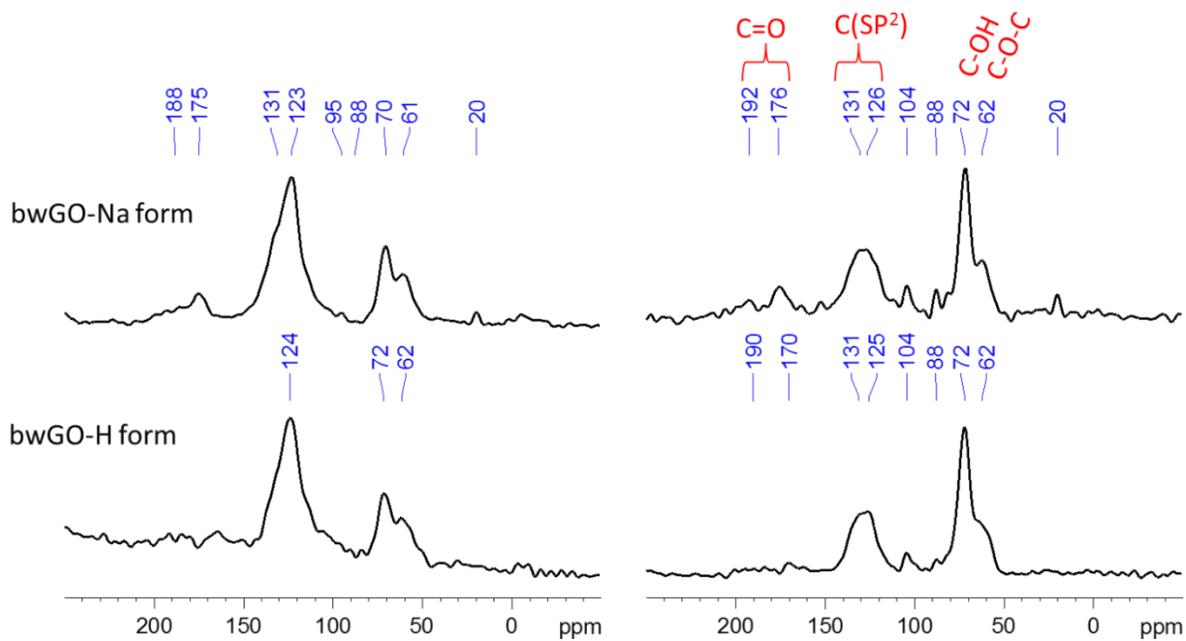
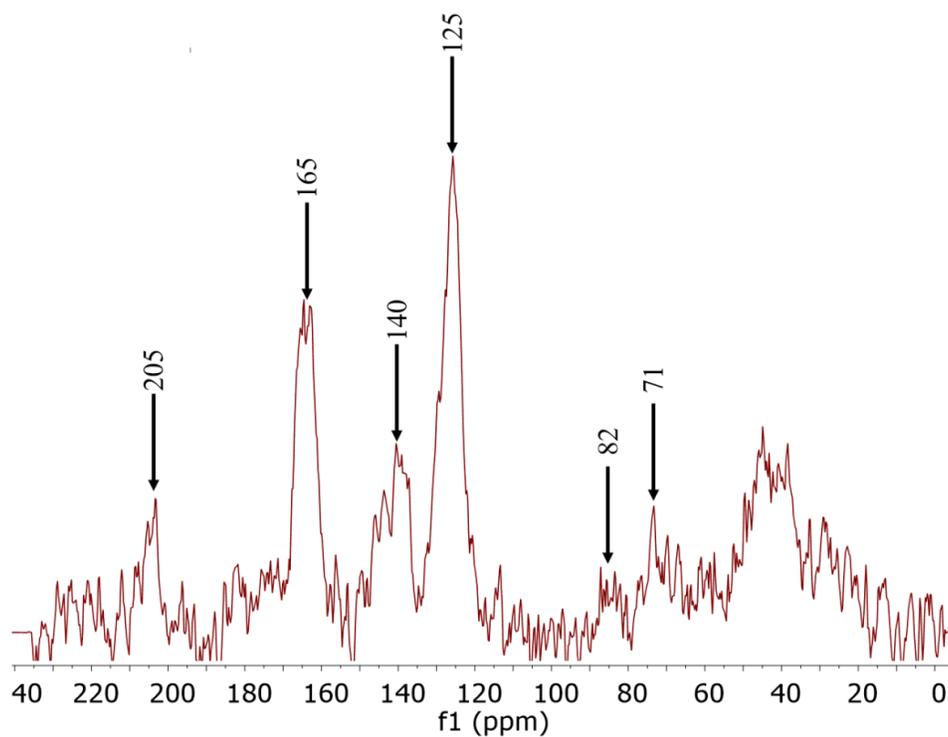
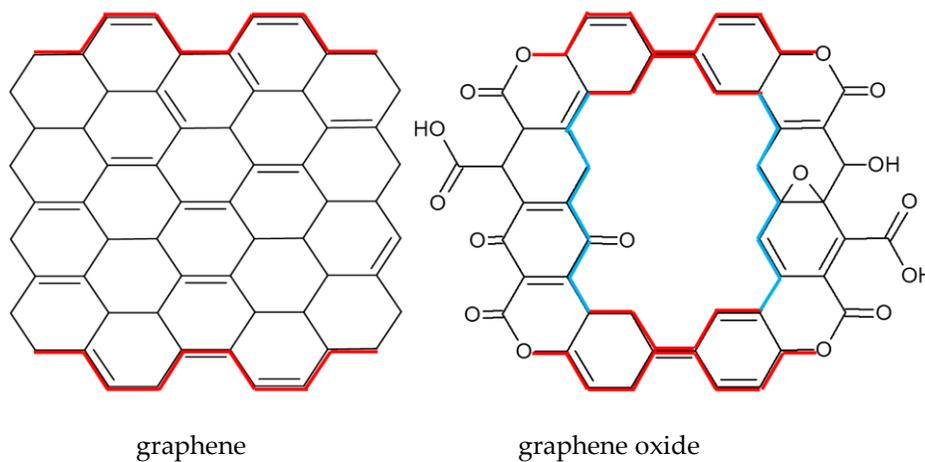


Figure S4. Direct excitation  $^{13}\text{C}$  MAS NMR (left) and  $^{13}\text{C}\{^1\text{H}\}$  CP MAS NMR (right) spectra of base-washed graphene oxide (sodium and hydrogen forms).



**Figure S5.**  $^{13}\text{C}\{^1\text{H}\}$  CP MAS NMR spectrum of OD-1.



**Figure S6.** Lattice model for the edges and round hole. Red and blue lines mark armchair and zigzag edges, respectively.

**Table S1.** Elemental composition of graphene oxide samples.

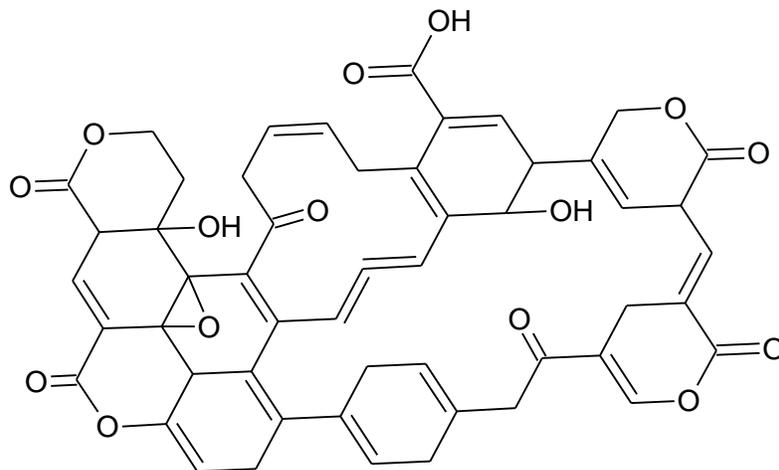
Samples	Elements by % weight			C/O ratio	C/H ratio
	C	H	O		
Graphite	99.6	0.4	-	-	0.4
GO	47.1	4.4	47.6	1.32	0.89
rGO	85.5	0.84	9.6	11.9	8.48
bwGO	55.0	2.4	39.2	1.87	1.91
rbwGO	78.8	1.27	14.8	7.1	5.17

**Table S2.** Raman results for graphene oxide samples.

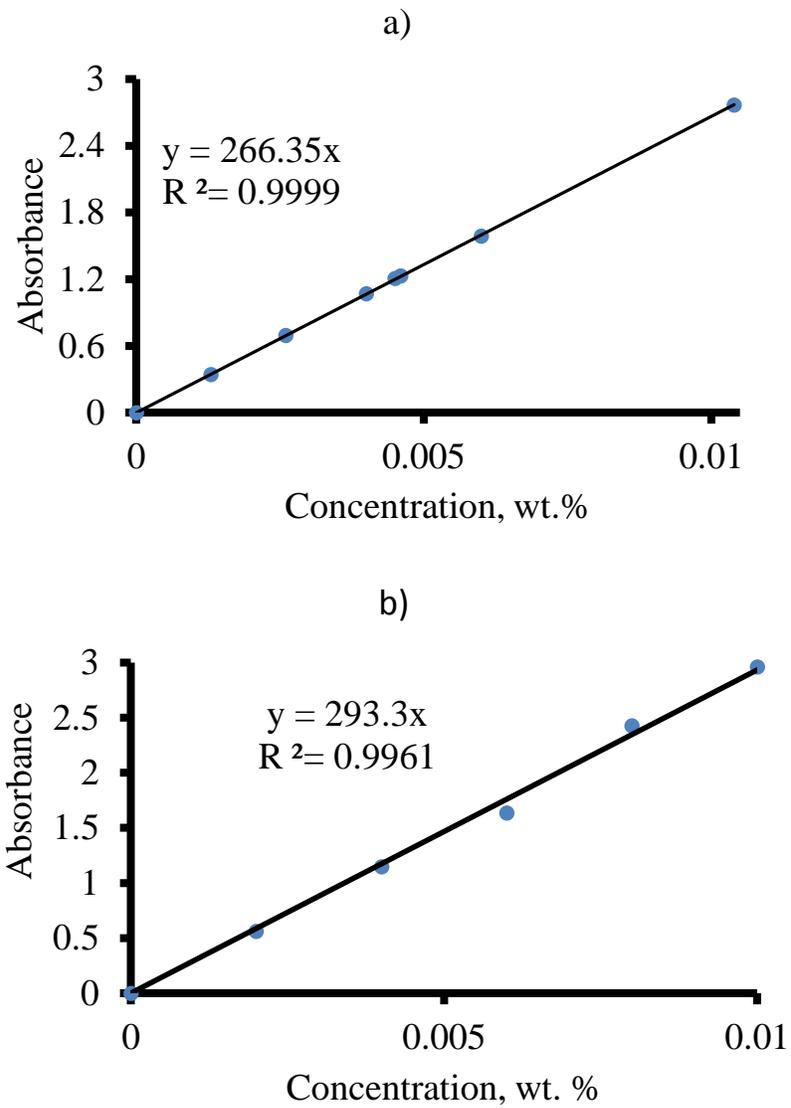
Samples	Raman peaks, cm <sup>-1</sup>					ID/IG	I2D/IG
	D-band	G-band	2D-band	D+D'-band	2D'-band		
Graphite	1359	1578	2713	-	3240	~0.08	~5.6
GO	1356	1592	2683	2920	3204	~0.92	~0.05
rGO	1351	1584	2685	2939	-	~1.47	~0.19
bwGO	1342	1583	2708	2920	3183	~0.97	~0.11
rbwGO	1343	1575	2684	2916	3165	~1.2	~0.09

**Table S3.** XRD analysis results.

Samples	2 $\theta$ max. (002)	FWHM(La)	La (nm)	d (nm)	N	2 $\theta$ max. (100)	FWHM(Lc)	Lc (nm)
Graphite	26.6	0.66	12.9	0.34	38	44.6	0.66	13.6
GO	10.7	6.72	1.24	0.83	2	42.5	7.14	1.25
rGO	-	-	-	-	-	-	-	-
bwGO	13.8	11.37	0.74	0.65	1	42.9	13.19	0.68
rbwGO	-	-	-	-	-	-	-	-



**Figure S7.** Possible structure of oxidative debris.



**Figure S8.** Correlation between absorbance and concentration of GO (a) and bwGO (b) solutions obtained at 600 nm on an UVmini – 1240 spectrophotometer.