

## Electronic Supporting Information for: Positive Zeta Potential of Nanodiamonds

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## Electronic Supporting Information (ESI)

This file contains: a) extended data and figures (Fig. S1, Fig. S2, Fig. S3 and Fig. S4)

### a) Extended Data

#### Particle Size Distribution

Figure S1 shows the size distribution of the particles in the colloid obtained from the air treated diamond powder. The colloid was prepared as explained in the Experimental Section. Two different methods for the determination of the particles' size were used. In the first one, the particles were characterized by dynamic light scattering (DLS) after 10 minutes centrifugation at 20000g. Several accelerative forces (from 5000g to 30000g) were also applied to the samples with identical results. As can be seen in figure S1b, the sample's size distribution median is 51 nm.

In DLS, the particle size is determined from intensity fluctuations in the Rayleigh scattering off a volume of the particles. As the intensity of Rayleigh scattering is proportional to  $d^6$ , large particles or aggregates can mask the measurement over smaller particles, so the use of alternative methods for the particles' size characterization is important, mostly for colloids in which polydisperse sizes' distribution are present.<sup>1</sup> To confirm the particles' size distribution, a second characterization method based on nanoparticle tracking analysis (NTA) was used. NTA gives a more precise measure as individual particles can be tracked. The particle size distribution measured by NTA is plotted in figure S1a. A slightly bimodal distribution can be observed, with two clear particles' size distributions at 44 nm and 59 nm. The concentrations of particles in the colloid is in the order of  $10^7$  particles/ml. The colloid was previously diluted until the required concentration was achieved (usually between  $10^7$ - $10^9$  particles/ml).<sup>2,3</sup> In both methods, the obtained size is the hydrodynamic radio, usually bigger

than the size measured in the dried particles. Notwithstanding the particles' size value is in accordance with the value given by the commercial powder supplier.

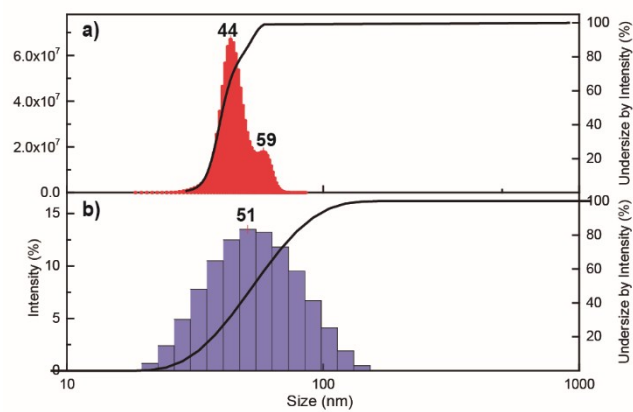
#### Fourier Transform Infrared Spectroscopy (FTIR)

In order to eliminate loosely bounded hydrocarbon originated from air exposure, all the samples were annealed at 550°C inside the FTIR chamber. To avoid unexpected IR signal, all spectra were recorded after cooling down the samples to room temperature. Un-annealed and 200°C temperature annealed samples' spectra were also measured to compare hydrocarbon contamination. Fig. S2 shows the FTIR spectra of the different treated diamond powders.

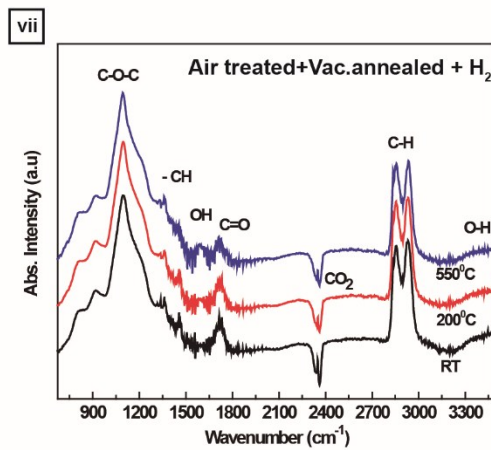
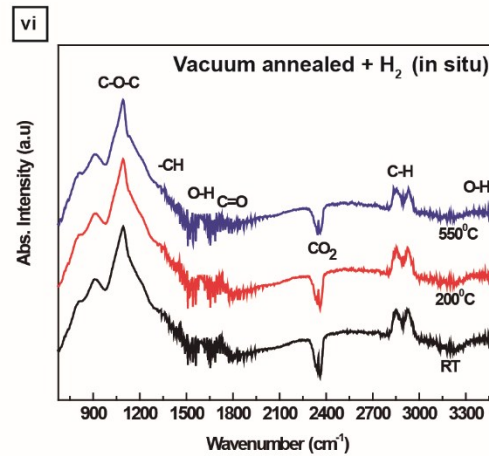
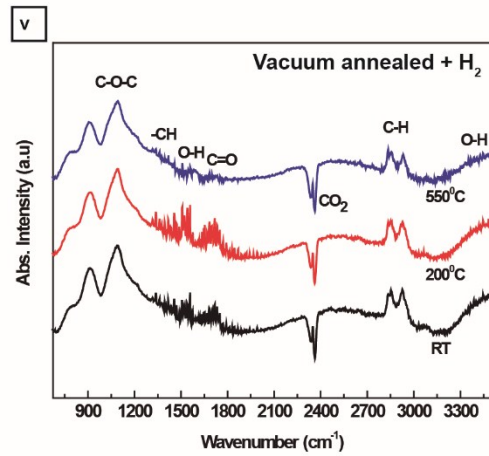
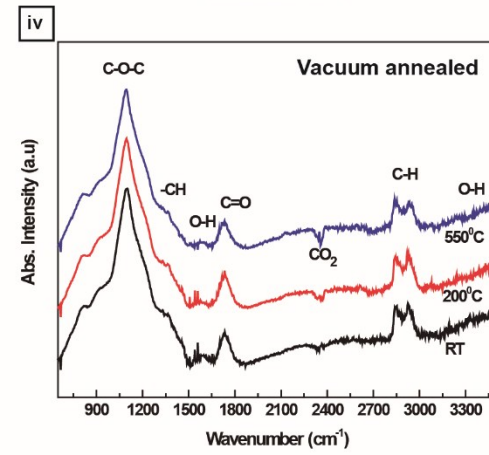
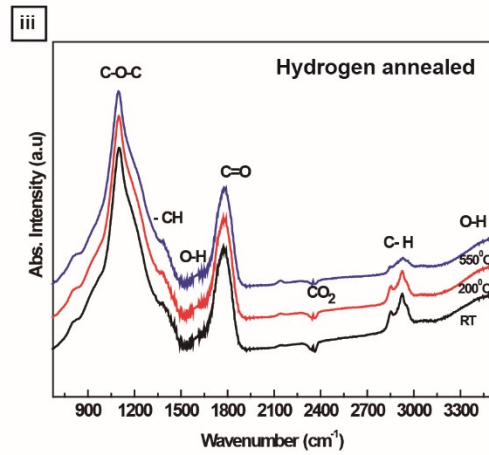
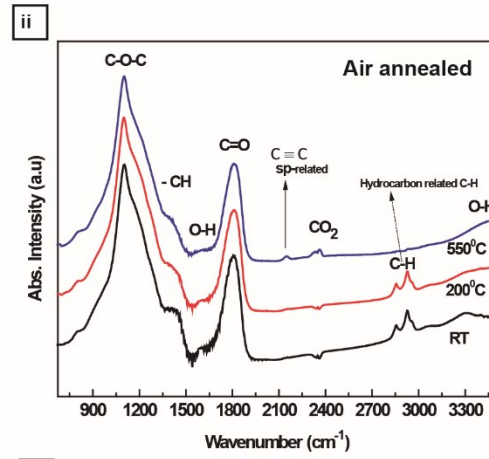
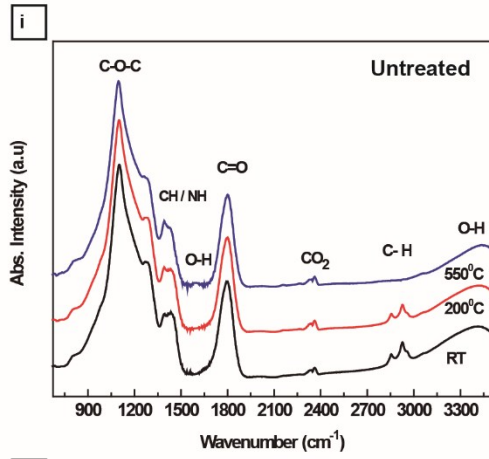
#### Zeta Potential and Particle Size Distribution without the High-Power Ultrasound Probe

Zeta potential measurements and size distribution measurements (measured with DLS) were also performed in hydrogen annealed detonation diamond colloids, prepared without using a high-power ultrasound probe. These measurements shown in Fig. S3, prove that positive zeta potential values are not coming from the probe contaminants. Note that Fig. S3b, show a slightly bigger particles' size distribution without the probe.

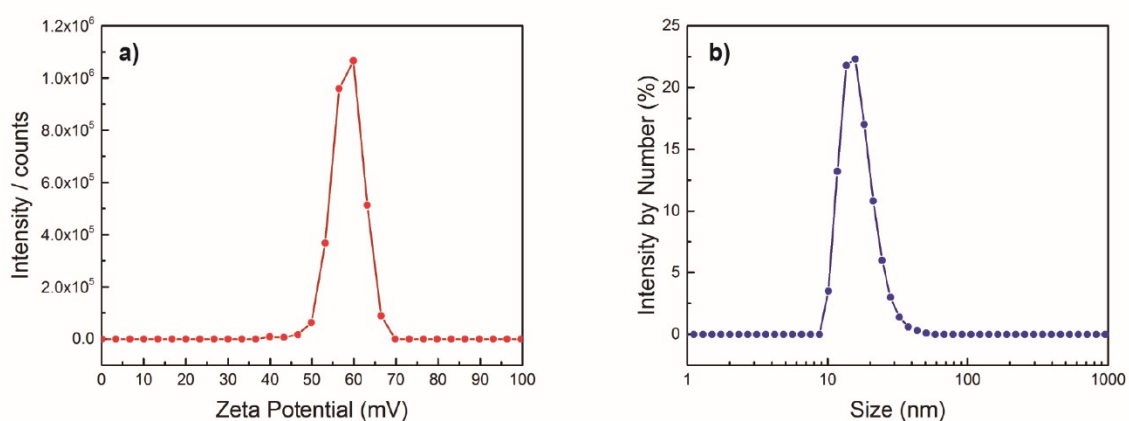
## Supporting Figures



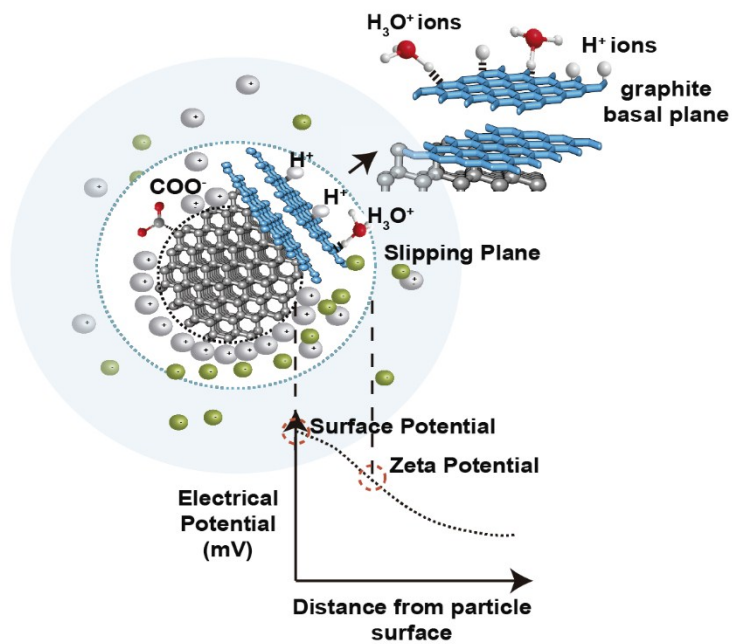
**Figure S1. Particles' size distribution of the air treated colloid. a,** Nanoparticle tracking analysis (NTA) size distribution. **b,** Dynamic light scattering (DLS) size distribution.



**Figure S2. FTIR spectra measurements of surface treated and untreated nanodiamond particles at different temperatures, room temperature, 200°C and 550°C. (i),** Untreated 50 nm nanodiamond particles. **(ii),** Air annealed at 480°C. **(iii),** Molecular hydrogen treated at 500°C. **(iv),** Vacuum annealed at 1000°C. **(v),** Sample (iv) treated with molecular hydrogen at 500°C. **(vi),** Sample (iv) treated *in situ* with molecular hydrogen at 500°C. **(vii),** Sample (ii) annealed in vacuum at 1000°C and the treated *in situ* with molecular hydrogen at 500°C.



**Figure S3.** Zeta Potential and Particle size distribution for hydrogenated detonation diamond colloid prepared without using a high-power ultrasound probe. **a)** Zeta potential measure at pH 7.6 and **b)** Size Distribution for 5nm particles.



**Figure S4.** Schematic figure explanation for the positive zeta potential measured in the samples subjected to a previous vacuum annealing treatment.

## REFERENCES

- 1 D. Mahl, J. Diendorf, W. Meyer-Zaika and M. Epple, *Colloids Surfaces A Physicochem. Eng. Asp.*, 2011, **377**, 386–392.
- 2 R. D. Boyd, S. K. Pichaimuthu and A. Cuenat, *Colloids Surfaces A Physicochem. Eng. Asp.*, 2011, **387**, 35–42.
- 3 V. Filipe, A. Hawe and W. Jiskoot, *Pharm. Res.*, 2010, **27**, 796–810.