Electronic Supplementary Material (ESI) for Nanoscale.

Efficient surface functionalization of detonation nanodiamond using ozone under ambient conditions

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Supporting information

Raman spectra:

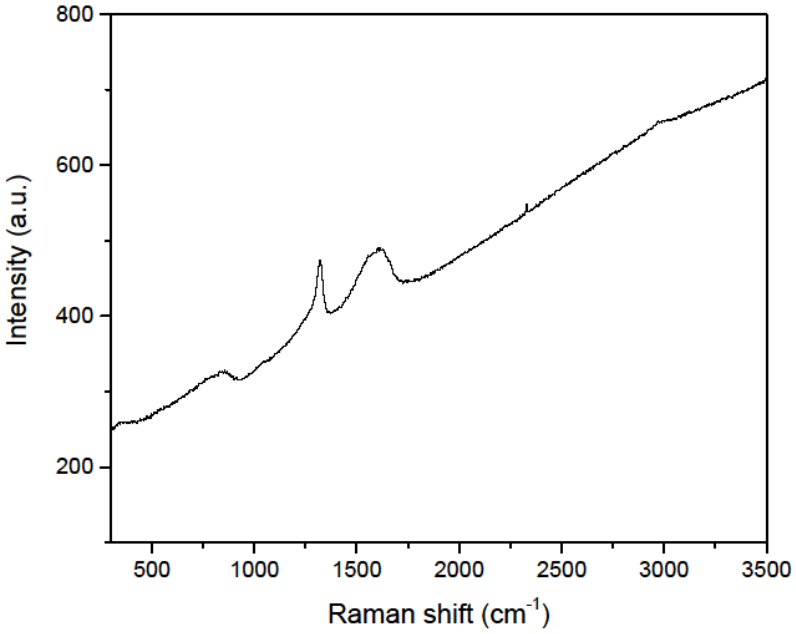

Fig. S1 Raman spectrum of DND

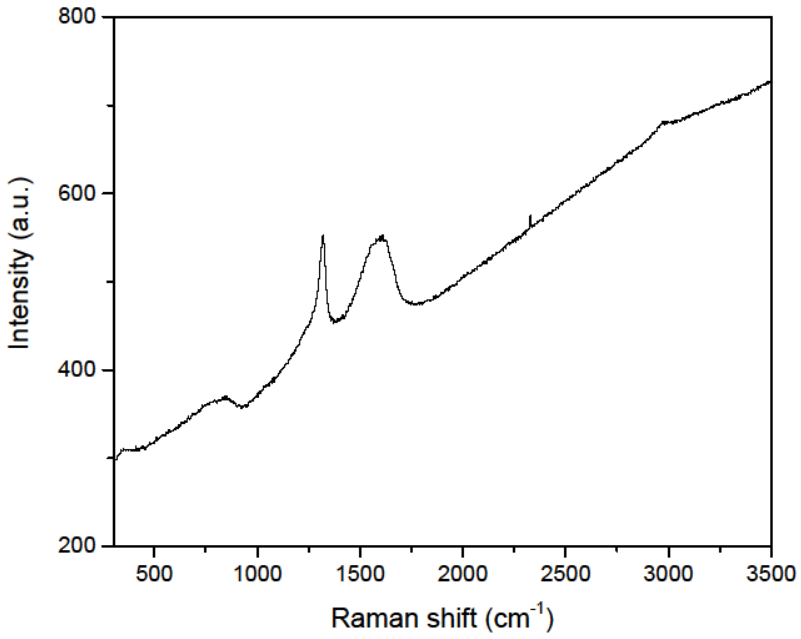

Fig. S2 Raman spectrum of DNDox

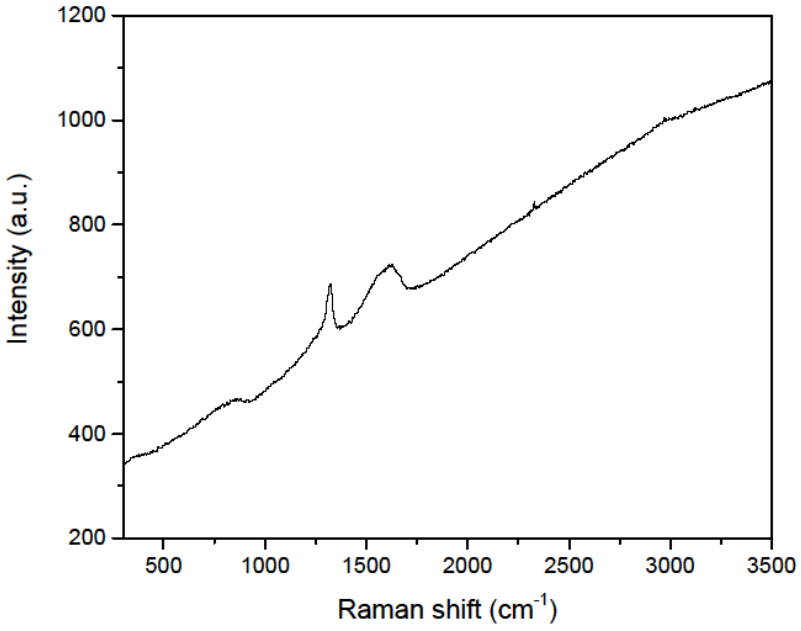

Fig. S3 Raman spectrum of DNDred

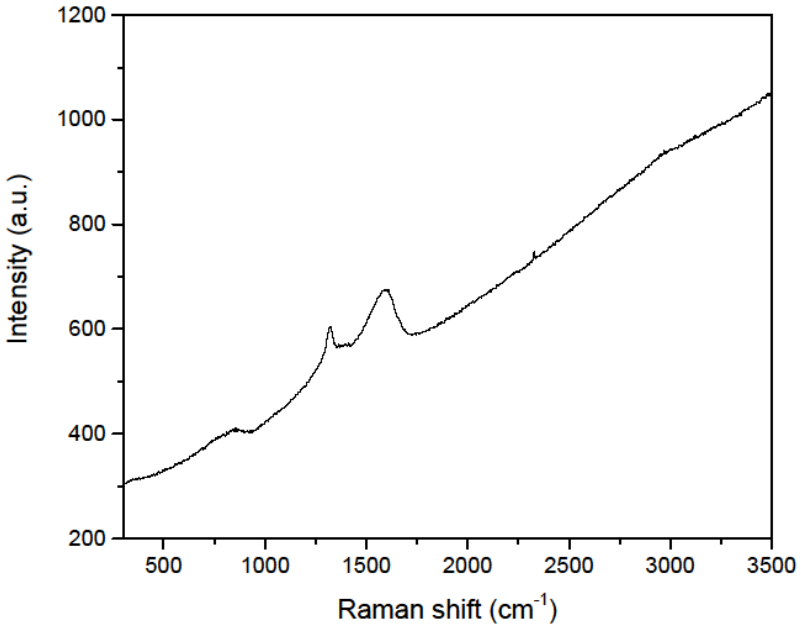

Fig. S4 Raman spectrum of aDND

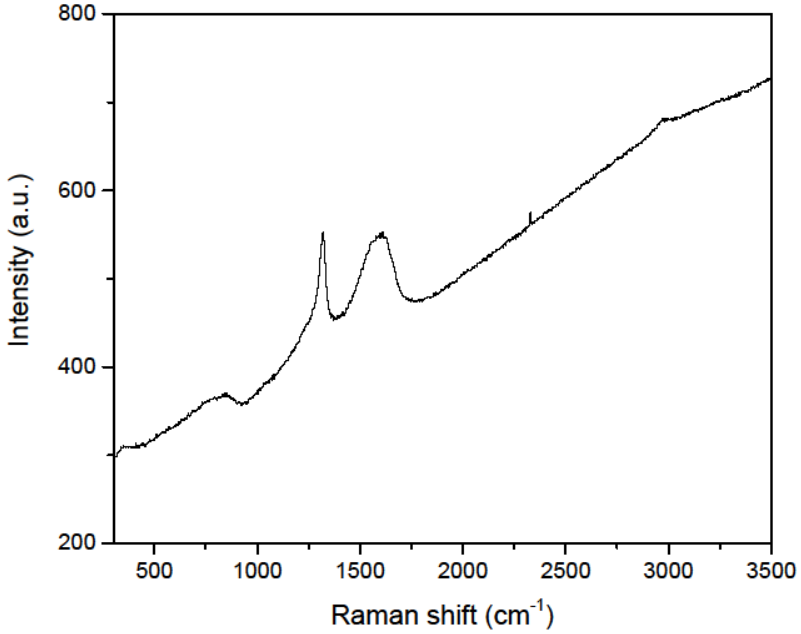

Fig. S5 Raman spectrum of aDNDox

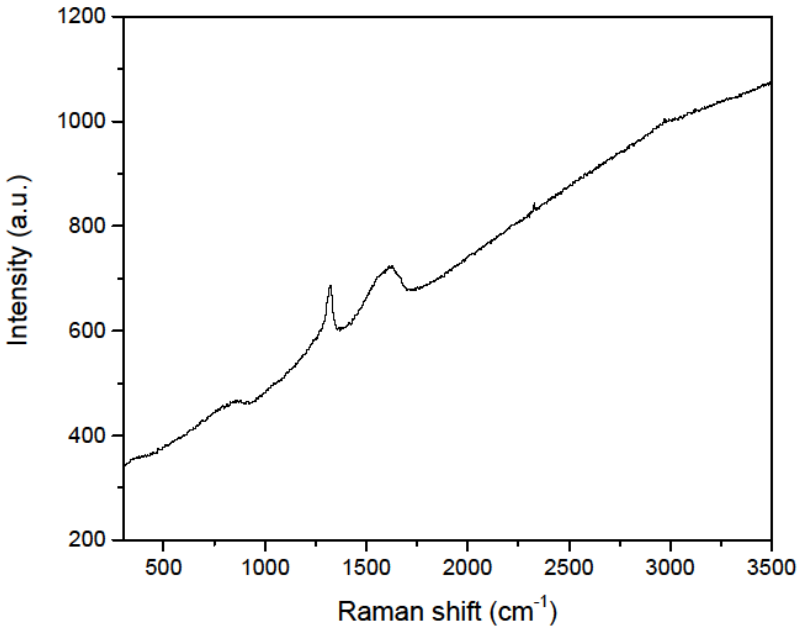

Fig. S6 Raman spectrum of aDNDred

XRD spectra:

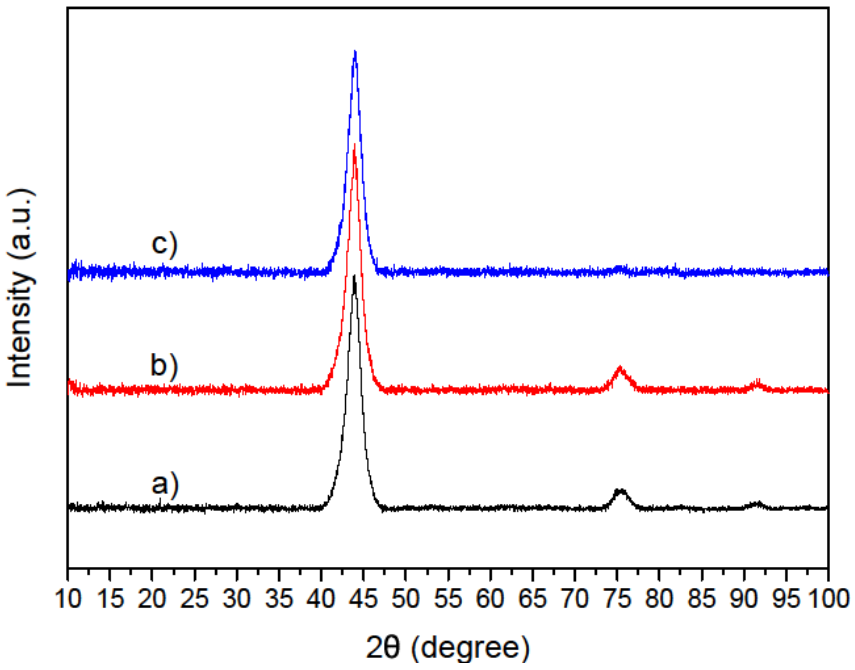

Fig. S7 XRD spectra of a) untreated DND b) DND treated with ozone and oxidized with H_2O_2 c) DND treated with ozone and reduced with $NaBH_4$

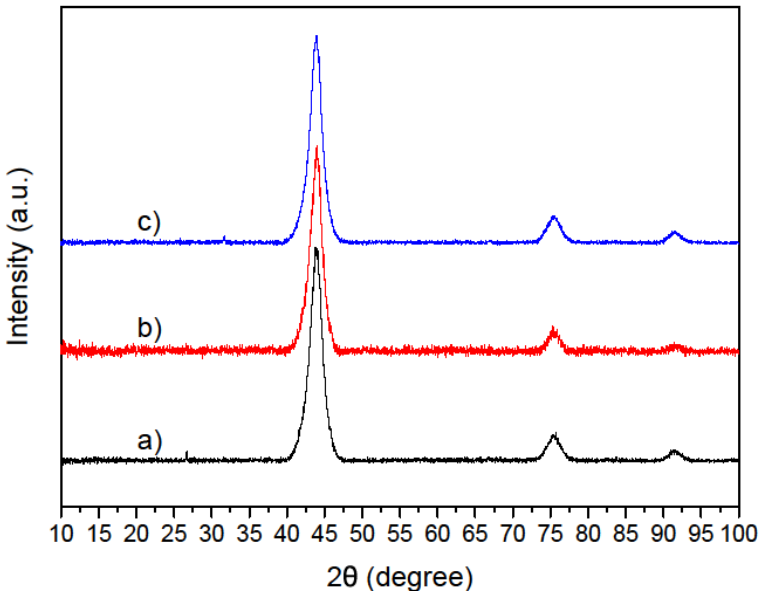

Fig. S8 XRD spectra of a) untreated aDND b) aDND treated with ozone and oxidized with H_2O_2 c) aDND treated with ozone and reduced with $NaBH_4$

FTIR spectra of diamond materials between 1500 and 1800 cm^{-1}

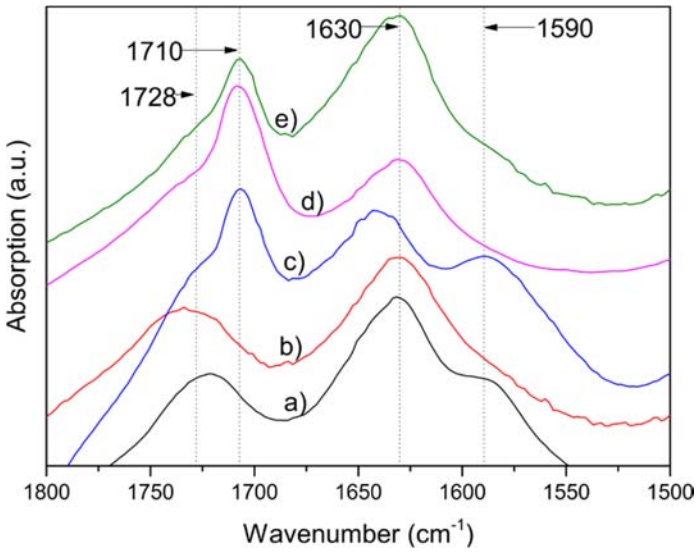

Fig. S9. Magnified FTIR spectra of a) untreated DND b) DND treated with ozone over a period of six hours c) DND treated with ozone for 6 h and oxidation with hydrogen peroxide d) DND treated with ozone for 6 h, oxidation with hydrogen peroxide and washing with dilute hydrochloric acid e) DND treated with ozone for 6 h, reduction with sodium borohydride and washing with dilute hydrochloric acid.

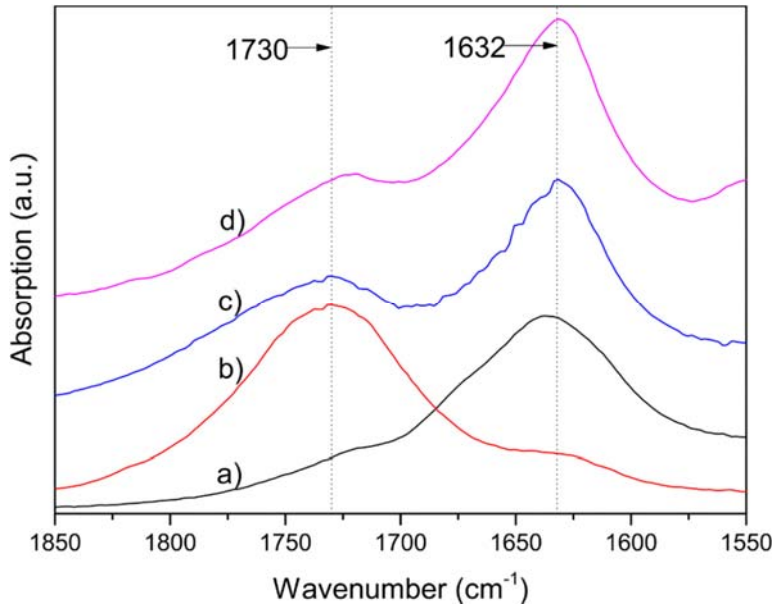

Fig. S10. FT-IR spectra of a) untreated aDND b) aDND treated with ozone over a period of six hours c) aDND treated with ozone for 6 h, oxidation with hydrogen peroxide and washing with dilute hydrochloric acid d) aDND treated with ozone for 6 h, reduction with sodium borohydride and washing with dilute hydrochloric acid**.**

IR spectra of DND submitted to workup treatment without prior ozonization

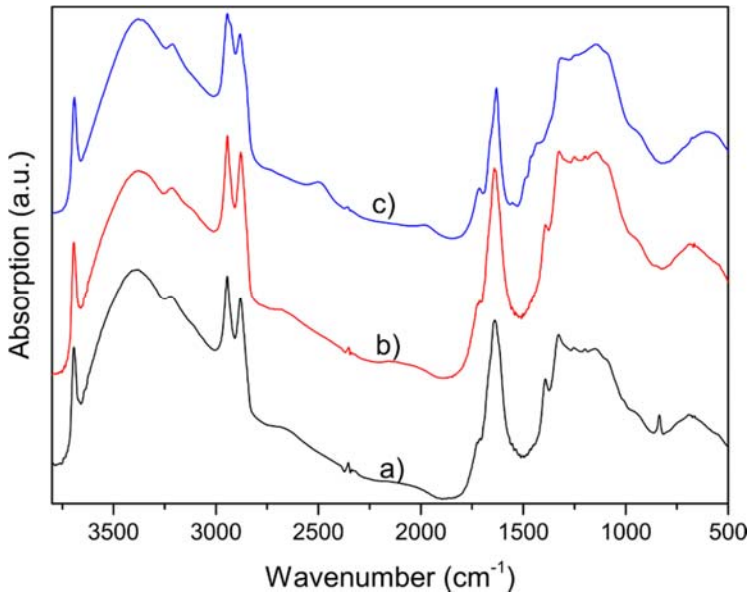

Fig. S11. IR spectra of a) annealed aDND; b) aDND treated with $NaBH_4$ and subsequent washing with dilute HCl; c) aDND treated with H_2O_2 and washed with dilute HCl.

Determination of required reaction time

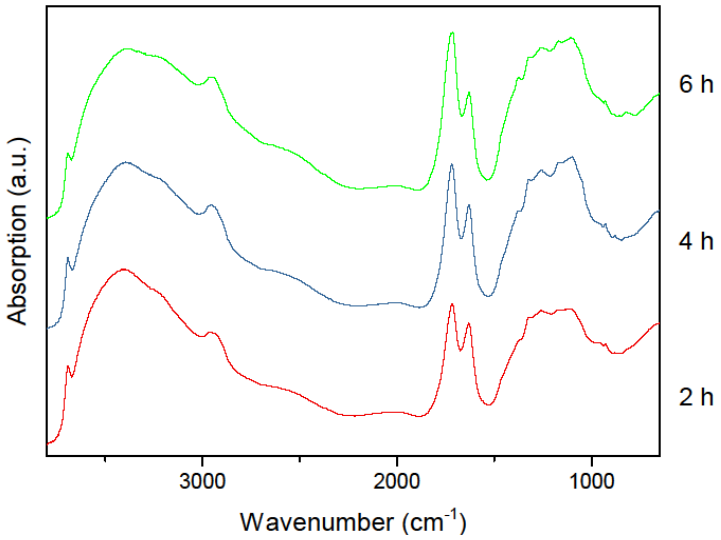

Fig. S12: IR spectra of ozone treated DND after different reaction times for ozonolysis under ambient conditions.

Estimation of the number of surface groups from Boehm titration results

Table S1. *Number of surface groups for untreated DND, DND after ozonolysis and oxidative workup and DND after ozonolysis and reductive workup*

Sample	carboxylic (sites per nm^2)	lactonic (sites per nm^2)	phenolic (sites per nm^2)
DND	0,42	0,07	0,01
DNDox	0,39	0,07	0,09
DNDred	0,34	0,02	0,003

Table S2. *Number of surface groups for thermally annealed aDND, aDND after ozonolysis and oxidative workup and aDND after ozonolysis and reductive workup*

Sample	carboxylic (sites per nm^2)	lactonic (sites per nm^2)	phenolic (sites per nm^2)
aDND	0,09	0,02	0,005
aDNDox	0,38	0,04	0,04
aDNDred	0,35	0,04	0,02

The estimation has been carried out following the example below according to ref. 20:

1. $$COOH\ Sites = \frac{n_{COOH} * N_A}{m_{DND} * S_{BET} * 10^{18}}$$

   e.g. for untreated DND:
   n_{COOH} = 0,000246 mol
   N_A = 6,02 x 10^{23} mol^{-1}
   m_{DND} = 1 g
   S_{BET} = 350 m^2/g from BET isotherm (and assumed to be unchanged after ozonolysis)
   ➔ 0,42 sites per nm^2

Reference [20] from main article:

L. Schmidlin, V. Pichot,, M. Comet, S. Josset, P. Rabu, and D. Spitzer, *Diamond Relat. Mater.*, 2012, **22**, 113-117.