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Removal of metal impurities from detonation nanodiamonds by heat treatment with chlorine gas

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Detonation nanodiamonds (DNDs) have the potential to be part of a new class of efficient slow neutrons reflectors.[1-3] However, the synthesis conditions lead to some impurities that are unfavourable for such an application due to their high neutron absorption, e.g. hydrogen, sp² C and metal impurities. Thus, ultrapure DNDs are highly required for this application. With the aim of selectively purify DNDs, our approach is based on a combination of gas phase purification treatments: oxidation under air (ODND), chlorination (Cl-DND) and fluorination (F-DND) treatments. The oxidation of DNDs leads to the total combustion of the carbonaceous species, thus concentrating the remaining inorganic impurities which allow their detection (XRD, EDX, FTIR and Raman spectroscopies) and quantification (ICP-MS). Chlorination is used to selectively remove the metal impurities contained in the DNDs samples. The efficiency of the purification treatment is assessed by TGA (Figure 1.A) and FTIR analyses; which is facilitated by applying the selective combustion of the treated DNDs (Figure 1.B). Finally, for the purpose of obtaining ultrapure DND samples, a complementary fluorination treatment is then applied to remove C-H and sp² C bonds and impurities which had not been eliminated as volatile chloride in the temperature range (500-900°C) by chlorination. A comparison of the DND samples submitted to these different chemical treatments is visible in Figure 1.C.

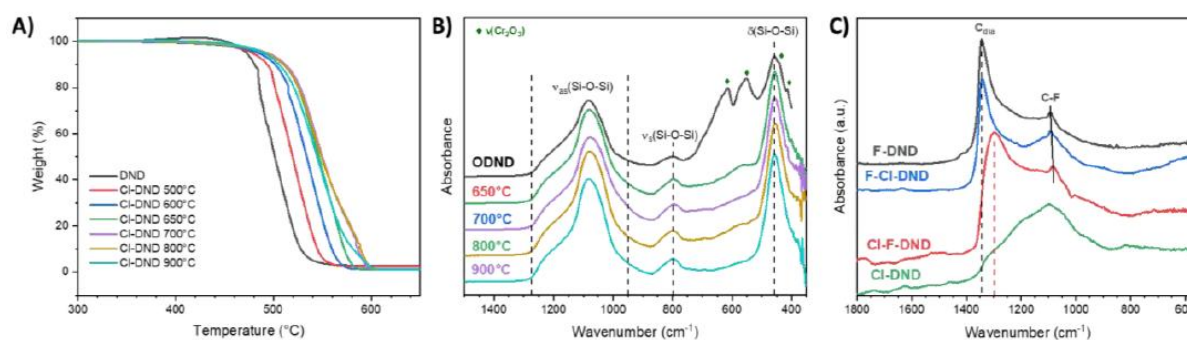


Figure 1. A) Thermogravimetric analysis of DND and Cl-DND samples at different temperatures. B) ATR-FTIR spectra of oxidized Cl-DNDs samples. C) ATR-FTIR spectra of F-DND, Cl-DND, F-Cl-DND and Cl-F-DND samples.

References

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- [3] Herraiz et al J. Phys. Chem. C 2020 124 (26), 14229-14236