Elemental quantification of wet digested Nanocarbons and characterization of their residues

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Abstract

It is estimated that Nanocarbons will be incorporated in our daily life in about three decades. Despite the accumulated knowledge on these materials, a pressing need to develop viable strategies for industrial-scale standardization persists. In addition, it is recognised that even minute amounts of other elements beyond carbon (e.g. transition metals used as growth catalysts) could have a deleterious effect not just on the aggregated physicochemical response of these materials but also in the environment and human health.[1]

Currently, the "gold standard" technique to quantify impurities in Nanocarbons is the neutron activation analysis.[2] However, as it relies on a neutron source, a nuclear reactor is needed, and so it is not suitable for routine analysis. On the other hand, inductively coupled plasma (ICP) techniques are widely available in chemistry laboratories and commonly applied to the study of vestigial quantities of metals in solid materials. Unfortunately, its applicability to Nanocarbons (e.g. graphene and nanotubes) has suffered from the lack of efficient digestion steps and certified reference materials (CRM).[3]

In this study, various commercial and certified graphitic carbon materials were subjected to a "two-steps" microwave-assisted acid digestion procedure. Following this, the concentration of up to 18 elements was analysed by ICP coupled to optical emission spectrometry (OES). Successful quantification of the certified elements was achieved, hence validating our wet digestion approach. Its applicability was further confirmed for a commercial single-walled carbon nanotube (SWCNT) sample. Where possible, the digestion residues were characterised by Raman spectroscopy, transmission electron microscopy and nuclear magnetic resonance (NMR) as we strived to understand the structural disintegration mechanism for graphitic carbons. It was seen that NMR, despite being less commonly used in the study of Nanocarbons, may effectively be the most valuable tool to access the digestion degree of these materials.

References

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