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INAA of elemental admixtures in carbon-based nanomaterials for battery electrodes

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Carbon-based nanomaterials (nanocarbons) exhibit exceptional mechanical, electrical, thermal, and optical properties. These materials are attractive in various fields of science and technology, including energy storage. Despite the interest in nanocarbons as electrodes for supercapacitors and batteries, additional elements present at variable quantities could influence their electrochemical response. These admixtures often originate from the production process of the nanocarbons and details on concentration is often unavailable. Absence of such information is especially bothersome in the case of expensive commercial materials. Due to the high chemical resistance of nanocarbons, elemental characterization is a challenging task, particularly if total sample decomposition and solubilization is required. Moreover, while routine digestion methods are being developed worldwide, results validation is complicated due to a lack of well-characterized reference materials for many of the known nanocarbons. Instrumental neutron activation analysis (INAA) is an excellent method to establish reference values of total mass fractions in many types of materials. Recognized as a primary method of chemical analysis, INAA has high potential for accuracy, matrix independence, and very low limits of detection for many elements. Moreover, INAA is a non-destructive method and does not require sample dissolution.

We examined six different nanocarbon samples by INAA, i.e., carbon black, expandable graphite (purified and non-purified), reduced graphene oxide, and two specimens of single walled carbon nanotubes. The test portions, each with a mass of about 30 mg, were irradiated in the LVR-15 experimental reactor (Research Centre Řež, Ltd.) at a thermal neutron fluence rate of $3 \cdot 10^{13}$ cm^{-2} s^{-1}. The irradiation time was 1 min or 2 hrs to be able to determine elements forming radionuclides with half-live from 2.5 min to several years. Quantification was performed by co-irradiation of a set of synthetic multielement comparators. Quality control of the results was demonstrated by analysis of the SWCNT-1 Certified Reference Material (NRC Canada) and US NIST Standard Reference Materials SRM1633b, SRM2711, and SRM1547. Comparing the results determined for these six nanocarbons highlights how different their composition truly is, even when sold as (or presumed to be) "pure"carbon materials. Mass fraction of some elements span from sub-μg g^{-1} to units of wt %. The INAA results can now be used to validate the results from more routine analytical tools. A comparison with the values determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) at KAUST for some nanocarbons is presented.

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