**Preparation methods for Carbon Materials Possessing Turbostratic Graphite and Graphene Nanodomains via Combustion of Magnesium in Carbon Dioxide**

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Introduction.This study presents a detailed method for synthesis of a carbon material aligned in a turbostratic manner containing regions of graphite and few-layer graphene located in carbon-only nanodomains. The material, obtained from the combustion of magnesium in a carbon dioxide atmosphere, displays 2D and layered 3D amorphous architectures.

Aims. This study presents methods and procedures for producing a carbon sample on the gram-scale in detail. The method and procedures covers are the following steps; 1. The preparation of carbon in CO2 atmosphere; and 2. The purification of the by-products to obtain pure turbostratic graphite.

Methods. Magnesium was combusted in a carbon dioxide atmosphere sublimated from dry ice blocks.[1] The Mg ribbon was ignited on a dry ice block and the combusting Mg was covered by the other half of the dry ice block. The Mg ribbons allowed to completely combust. After a complete combustion, a grey-black product consisting of a mixture of magnesium oxide (MgO) and carbon material was collected. The product containing MgO and carbon was mechanically ground to a fine powder for a digestion process. The resulting ground material was transferred to a 5 L beaker and 2.5 L of HCl was introduced and stirred overnight to digest the MgO by-product enabling the removal of Mg, based on the stoichiometric equation: MgO (s) + 2HCl (aq) → MgCl2 (aq) + H2O (l). The remaining carbon samples in the digest were sedimented, filtered and rinsed copiously with deionised water until pH reached 7 following this, the digested carbon was calcined in air for four hours in a furnace at 570 °C afterward to remove amorphous carbon. The calcinated carbon was digested again under the same digestion procedures followed by filtration and drying to obtain turbostratic graphite.

Results and Discussion. Although one digestion process removes most Mg based constituents for the 1st digestion, our XPS study suggested that an extra digestion process may be beneficial for obtaining cleaner sample. We also note that excessive repetition of digestion processes would not contribute to the purity if repeated over three times as there are material losses for each digestion process. The degree of purity of the as synthesised carbon can be improved by multiple acid digestions, but more than 6 atomic % Mg was still observed, likely due to trapped MgO located within various carbon matrices (e.g. MgO trapped in amorphous carbon) which could not be digested. Upon further processing including calcination and digestions, no traceable Mg was detected. Relatively small amount of oxygen was observed from the calcinated and re-digested sample in comparison with pre-calcined sample. This is attributable to the removal of MgO which were indigestible from the initial digestion.

Conclusion. The XPS characterisation confirms that digestion process removes most Mg based constituents. However, a calcination process and subsequent digestion process are required to obtain purer carbon containing turbostratic graphite samples. This gram scale process may be favourable to the applications require large volumes of samples.

**References**

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