Disorder-features in thermally stable exfoliated graphene using variable intercalation times and intercalant-blends

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Abstract

Efficient exfoliation of relatively good-quality graphene with high-yield from graphite is critical for its extensive application in different emerging technologies. The cost-implication when undertaken via electrochemical exfoliation makes upscaling even more likely. In addition, the ability to constructively tune the final graphene properties (in terms of the defect metrics in the graphitic lattice) to fit a specific required application could be achieved by controlling operational parameters adopted during the intercalation and exfoliation steps.
In a recent study, we demonstrated a one-step electrochemical exfoliation process carried out in a blend of inorganic salt electrolytes [1]. A remarkably thermally stable graphene was obtained from intercalated phosphate functional groups. The phosphate functionalization in the present study is obtained during the *intercalation step* of the synthesis process which gives more flexibility to the critical insertion step and modifies the material properties to a desirable stage. Here-in, a systematic evaluation of the nature of defects is elucidated with respect to varying intercalation times and intercalant mix using an acid-blend containing sulphuric acid (H_2SO_4) and phosphoric acid (H_3PO_4). The type and location (edge or grain boundary-type), and relative concentration (%) of available defects is discussed with respect to increasing the intercalation times slightly beyond the point it attains a threshold intercalation voltage. Thermal stability of the graphene has also been explored based on the quantity of phosphoric acid content in the intercalant-mix. The exfoliated graphene sheets were characterized using Scanning Electron Microscopy, X-ray Diffraction, Thermal Gravimetric Analysis, X-ray Photoelectron and Raman Spectroscopy in detail to quantify defects and phosphorus contents in the doped-graphene framework. A thermally stable graphene was obtained with a constant "*vacancy-type*" defect signature retained for increasing phosphoric acid in the blend. Interestingly. a shorter time to achieve the threshold intercalation voltage was observed with increasing the H_3PO_4 content.
This study provides a facile and energy-efficient recipe for synthesizing graphene nanostructures with signature defects relevant for use in energy storage and water treatment applications.

References

[1] Sharif, F., Zeraati, A.S., Ganjeh-Anzabi, P. Yasri, N, et al, Carbon (2019), 157, 681-692.

Figures

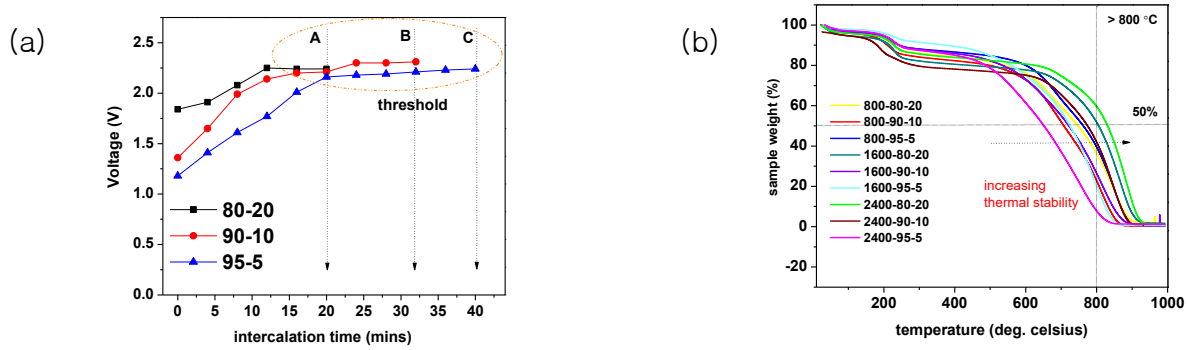

Figure 1: (a) intercalation threshold voltage as a function of time (b) thermal stability profiles in air for graphene samples prepared with different intercalation times and H_2SO_4-H_3PO_4 contents.