

## Critical Parameters in Exfoliating Graphite into Graphene

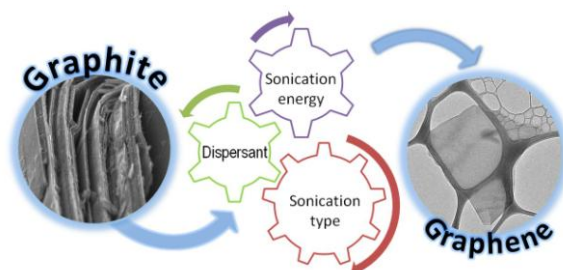
Matat Buzaglo, Michael Shtein and Oren Regev

Department of Chemical Engineering and Ilse Katz Institute for Nanoscale Science and Technology, Ben-Gurion University of the Negev, Beer-Sheva, Israel

[matat.buzi@gmail.com](mailto:matat.buzi@gmail.com)

### Abstract

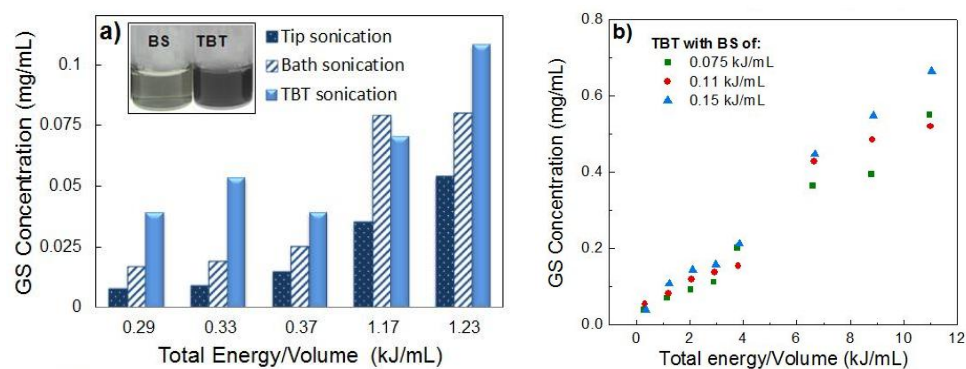
Dispersing graphite into few-layers graphene sheets (GS) in water is very appealing as an environmental-friendly, low-cost, low-energy source of graphene. Very high GS concentrations in water ( $0.7 \text{ mg}\cdot\text{mL}^{-1}$ ) are obtained by optimizing the nature of dispersant and the type of ultra-sonic generator. We employed a wide range of dispersant types: anionic, cationic, and nonionic surfactants and found no clear trend in GS concentration with the polarity of the head-group of the dispersant or the solution surface tension. In contrast, the nature of the hydrophobic part is critical for an efficient dispersion. Triton X-100 gave the highest GS concentration because it includes a benzene ring with strong  $\pi$ - $\pi$  interactions with the aromatic structure of GS. We found that a multi-step sonication procedure involving both tip and bath sources considerably enhances the yield of exfoliated GS. Tip sonication (TS) and bath sonication (BS) differ considerably in the power they supply. Therefore, the trivial assumption would be that the substantially higher power of TS would be more efficient than the weak BS. Fig. 1a compares the efficiency of either TS or BS alone, or their combination (TBT). Clearly, in terms of integrated sonication energy, TS alone is the least efficient option, and the weaker BS leads to much higher GS concentration. Still, combining the two sources gives the highest GS concentration. This marked difference is ocularly visible: a much darker supernatant (higher GS concentration) is obtained by TBT than by BS alone (Fig. 1a, inset). Although the BS contribution to the total energy is rather small ( $\sim 1\%$  of the total energy), it has a substantial contribution to the final GS concentration (40%) as shown in Fig. 1b. Both Raman spectroscopy and transmission electron microscopy indicate few-layers graphene patches with typical size of  $\sim 0.65\mu\text{m}$  in one dimension and  $\sim 0.35\mu\text{m}$  in the other (Fig. 2).



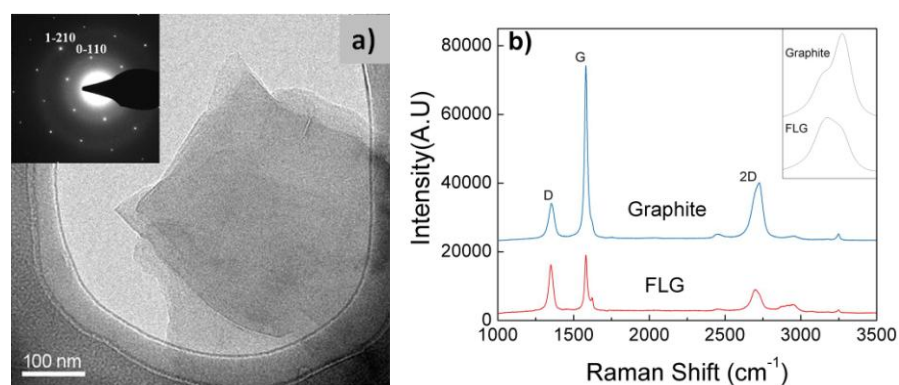
### Reference

[1] Matat Buzaglo, Michael Shtein, Sivan Kober, Robert Lovrincic, Ayelet Vilan and Oren Regev, *Phys. Chem. Chem. Phys.*, **15** (12) (2013), 4428 - 4435.

## Figures



**Figure 1** - Optimization of sonication procedure, dispersant and energy/volume. **(a)** GS concentrations upon bath sonication (BS), tip sonication (TS) and tip-bath-tip sonication (TBT). *Inset*: Image of the supernatant of the GS dispersions after centrifugation; **(b)** Concentration of GS (with TX-100) as a function of integrated sonication energy/volume, as measured by UV-vis absorption. These solutions were treated by TBT cycles.



**Figure 2** - Indication for few layers of graphene showing **a)** Room temperature TEM micrographs of GS stacks from GS-TX-100. The diffraction pattern (*inset*) indicates that the GS are less than 5 layers thick,<sup>31</sup> **b)** the Raman spectra of graphene film on quartz substrate at 514 nm. *Inset* shows a zoom-up of the 2D peak.