

Neutron Reflectivity to Investigate Graphene Exfoliation

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Hexafluorobenzene, (HFB) and benzene have previously been found to form an alternating columnar-stacked structure. Our research attempts to exploit the unique properties of this binary mixture to exfoliate graphite from natural sources, natural flake graphite and highly ordered pyrolytic graphite, into single layer graphene sheets via ultrasonication. This work was recently published in the Journal of the American Chemical Society [1]. The goal of our work at the Chalk River Neutron facility was to obtain direct experimental evidence for solvent layering nucleating from these exfoliated graphene sheets. To this end highly concentrated samples of varying solvent ratios were prepared in aluminum sample holders and scanned with neutron diffraction. Examples of the results are shown in figure 1.

Pure solvents as well as mixtures were studied to see how spacing in the graphite was affected as well as if

there was any additional ordering of the solvents. The appearance of the peak at higher d-spacings/ lower theta values (figure 2) shows ordered stacking of the solvents that disappears when the temperature is increased. Although we did see stacking in the solution that was dependent on the temperature as anticipated for the stacked system, the results were inconclusive as to whether the stacking was initiated from the graphene sheets or just resultant of natural stacking of the system.

Reference

- [1] Andrew J. Oyer, Jan-Michael Y. Carrillo, Chetan C. Hire, Hannes C. Schniepp, Alexandru D. Asandei, Andrey V. Dobrynin, and Douglas H. Adamson. Stabilization of Graphene Sheets by a Structured Benzene/Hexafluorobenzene Mixed Solvent. Journal of the American Chemical Society. 2012. 134 (11), 5018-5021.

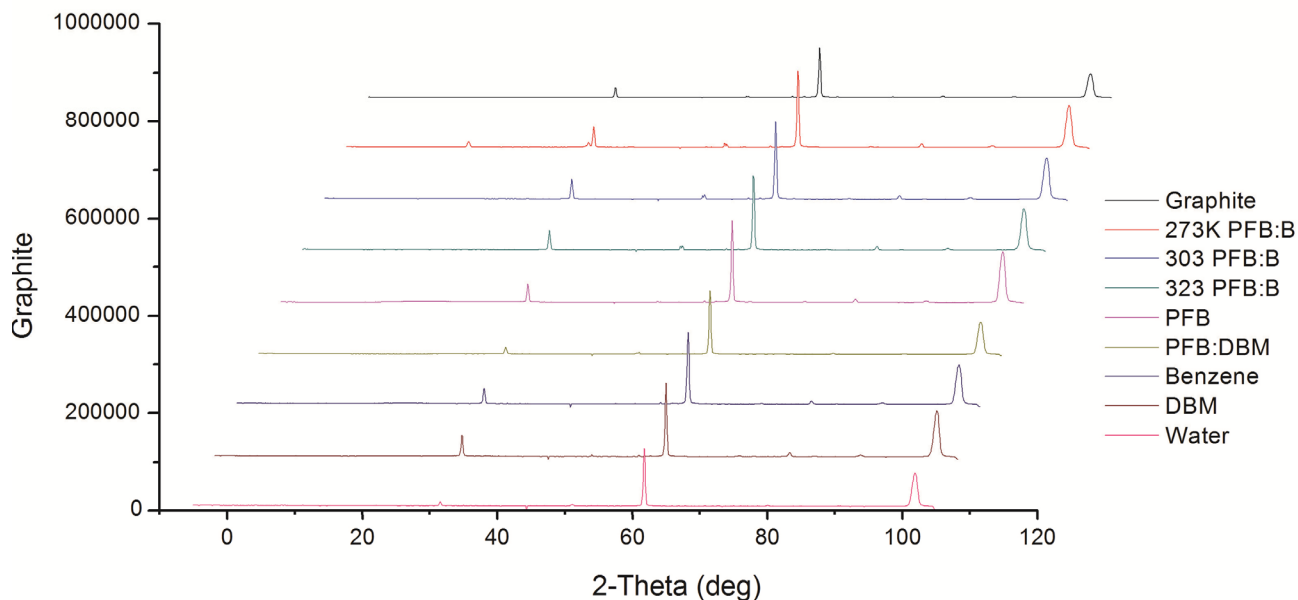


Fig. 1 2-Theta plots of initial studies of graphite with various solvents

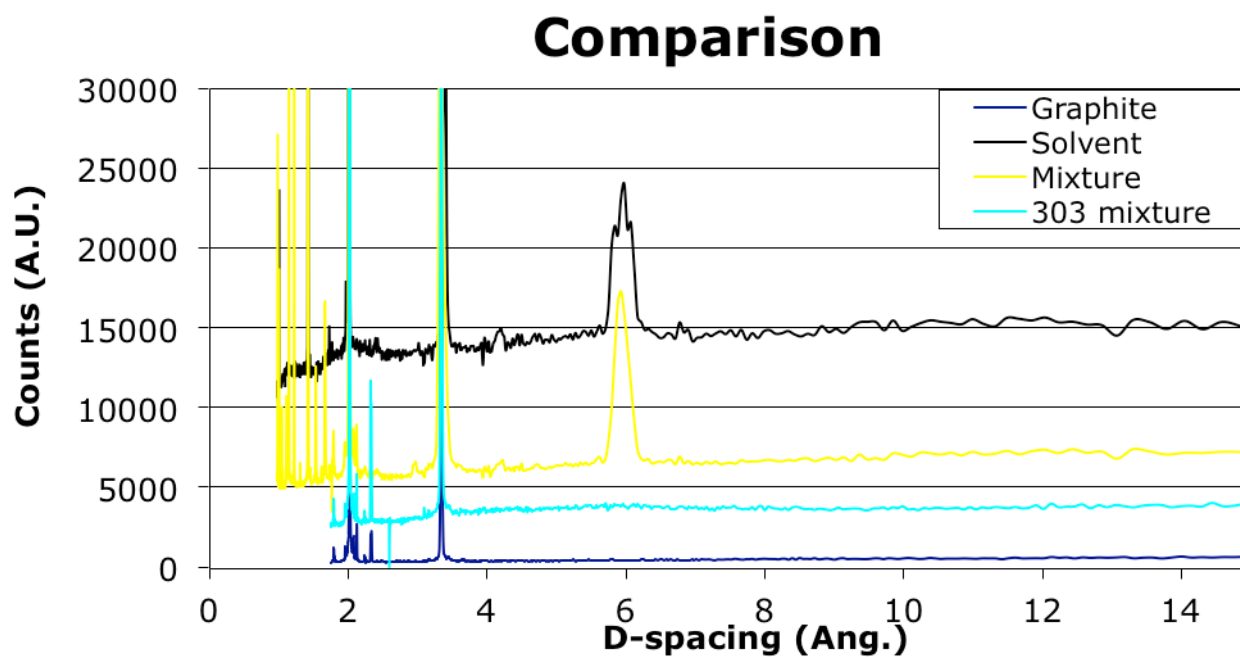


Fig. 2 D-spacing plot of region and solvents of the most interest