

Co-solvent exfoliation and suspension of hexagonal boron nitride

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Electronic Supplementary Information (ESI)

Materials and methods

Hexagonal boron nitride (h-BN) was supplied by Momentive Performance Materials, Inc. Two grades were used: NX1, with an average particle size of 1 μm , and PT100, with an average particle size of 13 μm . NX1 was used initially for the UV-vis studies as a model material, and TEM images were taken using exfoliated PT100. Acetone, methanol, ethanol, 1-propanol, 2-propanol, and *tert*-butanol were obtained from Sigma-Aldrich, and all were of $\geq 99.5\%$ purity (as purchased).

All co-solvent mixtures were prepared prior to the addition of h-BN. All calculations and preparations were done volumetrically with deionized water, and solvents were stored in bottles sealed with parafilm to minimize changes in concentration. A B2500A-DTH bath sonicator (VWR) operating at 42 kHz was used for all exfoliations. 10 mg of h-BN and 5 mL of co-solvent mixture, at the desired concentration, were added to a 3 dram vial. The vial was tightly capped and parafilmed to maintain the integrity of the solution during the sonication process. The vials were sonicated for 3 hours and rotated randomly around the space of the sonicator every 30 minutes to correct for any variations within the apparatus. This procedure was carried out for each solvent, 0 to 100% concentrations at 10% intervals. The mixtures reached temperatures of 45 °C during the course of sonication. The resulting mixture was allowed to cool and was subsequently centrifuged using an Allegra X-15R centrifuge (Beckman Coulter) for 20 minutes at 3200 rpm, and the resulting supernatant was carefully extracted for further characterization.

Characterization:

UV-vis spectroscopy: UV-vis absorbance measurements were made using a UV-3101PC UV-VIS-NIR scanning spectrophotometer (Shimadzu). The supernatant samples were pipetted into a quartz cuvette (path length 1 cm, Starna Cells, Inc.) and quickly capped. The samples were analyzed within 2 days of the initial sonication. All samples were analyzed from 700-300 nm, but the absorbance at 400 nm was used for measuring the relative amount of exfoliation for each co-solvent.

Transmission electron microscopy (TEM): TEM micrographs and diffraction patterns were recorded using a Titan S/TEM (FEI) operating at 300 keV. TEM samples were prepared by placing a 400-mesh lacey formvar/carbon copper grid (Ted Pella, Inc.) onto a piece of qualitative filter paper (Whatman “4”, seen in Fig. S1). The supernatant sample of interest was then diluted ~1:10 in water, shaken, and a few drops were added via a Pasteur pipet. The filter paper is necessary to help wick away the solvent as fast as possible. This helps to avoid restacking of the BNNS, ensuring an accurate representation of BNNS found in the co-solvent system.

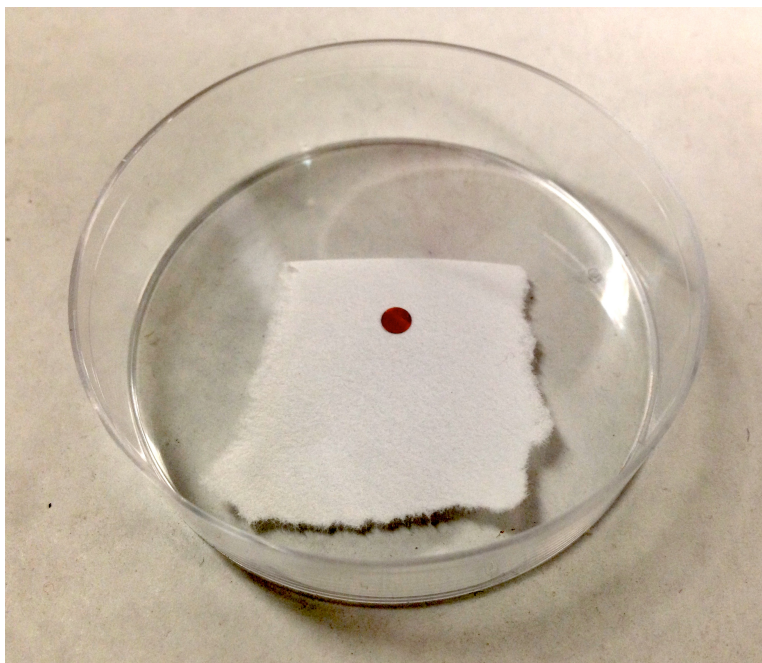


Fig. S1 Preparation of TEM samples. The lacey formvar/carbon copper grid (400 mesh) is prepared on top of a piece of filter paper. This helps to quickly wick away the solvent from the exfoliated BNNS.

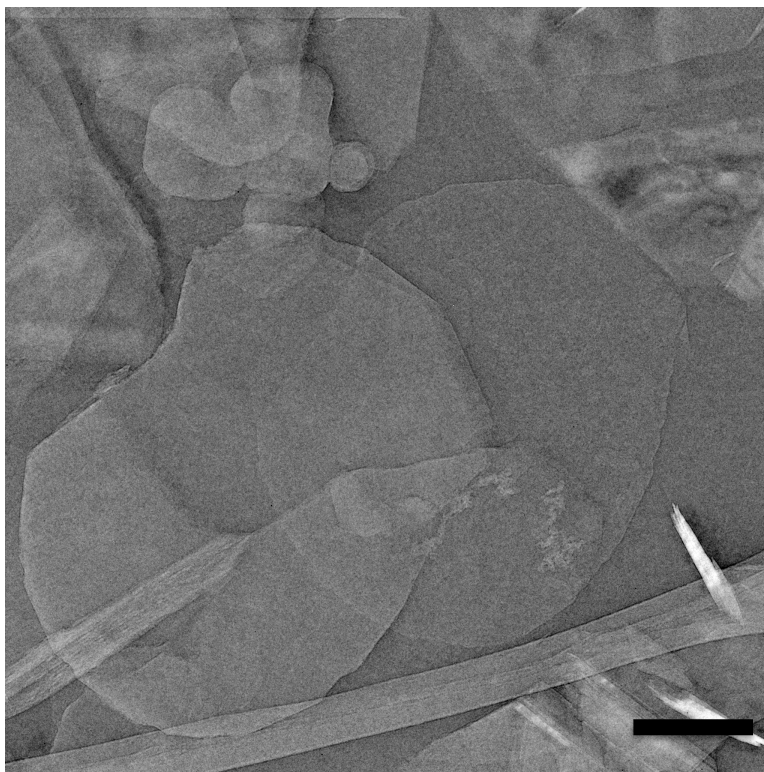


Fig. S2 TEM micrograph of a partially exfoliated BNNS. The two sheets share similar shapes. Scale bar is 200 nm.

**Surface tension in mN/m for various aqueous mixtures of
common solvents at 25°C**

Mass %	Acetone	Methanol	Ethanol	1-propanol	2-propanol	<i>tert</i> -butanol
0%	72.0	72.0	72.0	72.0	72.0	72.0
10%	44.9	56.2	47.5	34.3	40.4	32.7
20%	40.5	47.2	38.0	27.8	30.6	25.6
30%	36.7	41.1	33.0	26.0	26.8	23.7
40%	33.0	36.5	30.2	25.3	25.3	23.2
50%	30.1	32.9	28.0	24.8	24.3	22.6
60%	29.4	29.8	26.2	24.5	23.5	22.0
70%	29.4	27.5	25.0	24.1	22.7	20.9
80%	27.6	25.5	23.8	23.9	22.1	20.4
90%	24.5	23.9	22.7	23.6	21.7	20.1
100%	23.1	22.5	21.8	23.3	21.2	20.1

Fig. S3 Surface tension values of common aqueous mixture at 25 °C as obtained from the *Handbook of Chemistry and Physics*, CRC Press, 2003, 84th edition. Data for *tert*-butanol comes from Gliński *et al.*, *J. Chem. Phys.*, 1995, 102, 1361. Note: 1 mN/m = 1 mJ/m².

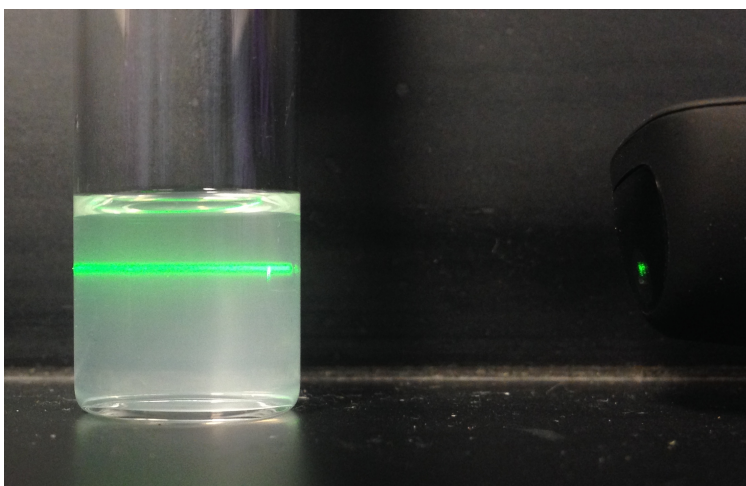
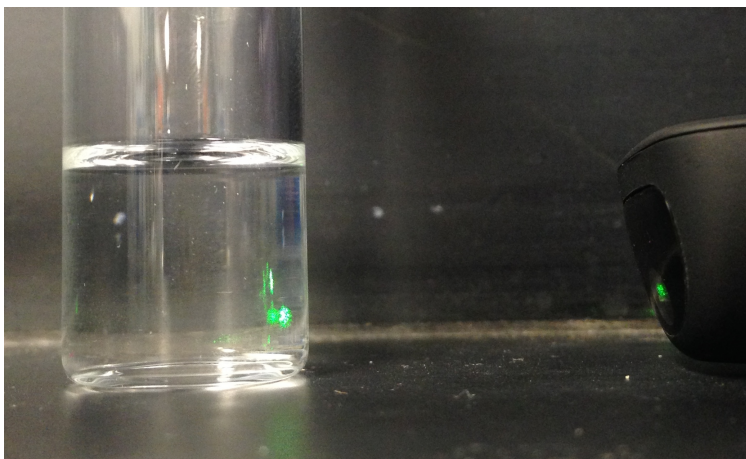


Fig. S4 Top) Green laser shining through 60 w/w% tBA in water. Bottom) Green laser shining through PT100 h-BN dispersed in 60 w/w% tBA in water. Tyndall scattering can be seen in the bottom photo due to the presence of BNNS.