Multifrequency ultrasonic exfoliation of graphite in water: Process optimisation by controlling the acoustic pressure and solution temperature

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Liquid phase exfoliation is a widely used method in the production of two dimensional materials, with the addition of ultrasonic induced cavitation being a promising method to enhance and optimise the process for large scale development. However, due to the complex nature of acoustic cavitation, and the poorly understood mechanisms of bulk layer material exfoliation, a robust, efficient procedure has yet to be developed. One important parameter that has not been systematically studied in ultrasonic assisted liquid phase exfoliation is frequency, which effects cavitation bubble size, oscillatory forces, bubble implosion dynamics and more. For this research, the use of a multifrequency system consisting of a 24 kHz sonotrode and a 1174 kHz membrane ultrasonic device were used to exfoliate graphite powder for 1 hour. During the process, a calibrated needle hydrophone measured acoustic pressure in 5 minute intervals, while 7 ml samples were taken for UV-Vis characterisation every 10 minutes. The analysis indicated that changes in the acoustic spectrum with temperature and treatment time correlated with the sharpening of the characteristic absorption peak of graphene, which in combination could be used to approximate graphene production, and develop an efficient system for exfoliation. Therefore, a trade-off between these parameters should be found to ensure high quality graphene.