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FAST HIGH-SHEAR EXFOLIATION OF NATURAL FLAKE GRAPHITE WITH TEMPERATURE CONTROL AND HIGH YIELD

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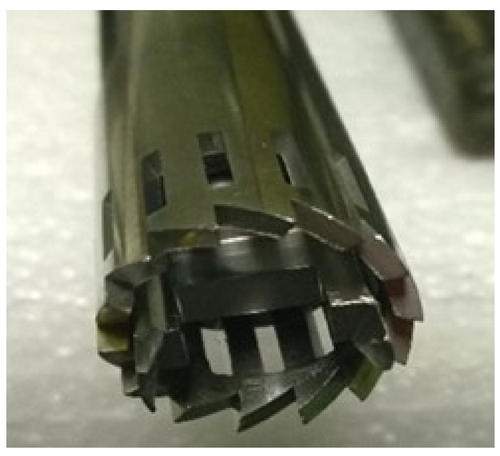
ABSTRACT

- Many of the potential applications demand graphene to be dispersed in liquids [1]
- High-shear exfoliation of graphite is a scalable [2] and cost-effective liquid-phase exfoliation (LPE) method for producing defect-free few-layer graphene dispersions
- The drawbacks of LPE such as low graphene concentrations and solvent or surfactant residuals hamper the applicability of the dispersions
- Here, we show that concentrations as high as 3 mg/ml with a 3 % yield can be produced only after 2 h of shear exfoliation in environmentally friendly aqueous medium using sodium cholate (SC) as surfactant [3]

EXPERIMENTAL

- The rock samples containing flake graphite were retrieved from Haapamäki, Finland
- **Fragmentation and enrichment** of the graphite ore in an in-house process to produce purified graphite containing 99.3 % m/m carbon



Preparation of the dispersions

- High-shear exfoliation in a 5 mM sodium cholate solution using a laboratory mixer with a rotor-stator combination
- Mixing parameters: C(i) = 100 mg/ml, N(rotor speed) = 16 500 rpm, t = 2 h

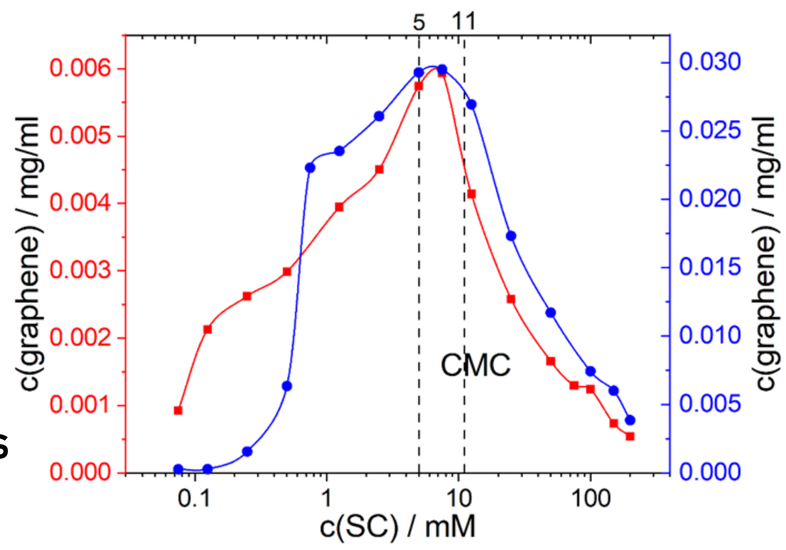
Post-processing of the dispersions

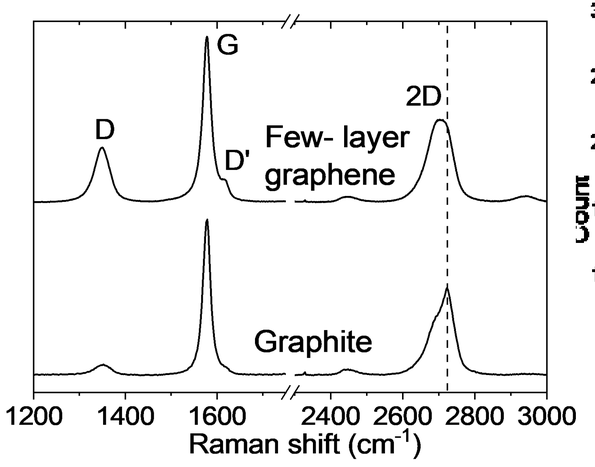
- 24 h **dialysis** to remove extra surfactant
- Thin film (500 nm – 1 µm) fabrication by **spray-coating** on glass substrates under heating
- Electrical **conductivity measurements** with a four-probe meter after drying in a glove box for 3 days

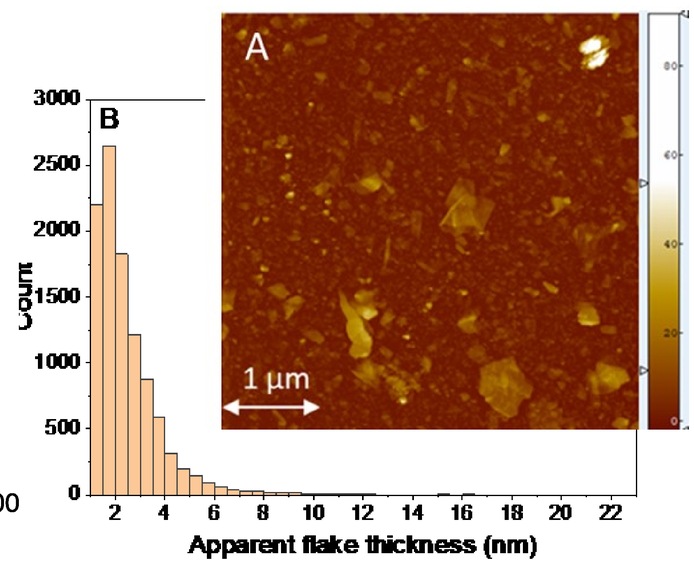
RESULTS

The high graphene concentration of 3 mg/ml with a 3 % yield can be attributed to:

- The appropriate SC concentration
  - The optimal range is 1 – 10 mM just below the CMC of SC
- Low temperature (T=10°C) during exfoliation
  - Concentration increases with decreasing temperature







Quality

- Raman and AFM analysis show that graphite is well exfoliated, the dispersions contain mostly few-layer graphene (<5 layers)

Film conductivity

- Electrical conductivities as high as 17 300 S/m
- Dialysis improves the conductivity by 40 %

CONCLUSIONS & HIGHLIGHTS

- The concentration and yield are significantly higher than in previous reports using water-based exfoliation medium
- The **surfactant concentration** and **temperature during exfoliation** affect the resultant graphene concentration significantly
- Dialysis removes excess surfactant from the dispersions improving the applicability of the produced material

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