

MXene synthesis yield acquisition

Date: 2019-10-10

Tags: *Synthesis KU Leuven 07/10/2019Synth Yield*

Created by: James Bird

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Goal : Acquire a measure of synthesis yield via drying of suspension using a vacuum oven

Procedure :

- Homogenised product from 07/10/2019 MXene synthesis placed in vacuum oven in speed-mixer pot, and vacuum pump (Vacuubrand MD 4 NT) exhausted oven to ≈ 1 mbar
- Oven set to 25 °C (initially 9 °C with vacuum established)
- Due to material ejection, transferred product to larger 200 mL plastic tub and covered with filter paper secured with elastic band
- Monitor total container mass including product to ensure all water evacuated
- Vacuum pump not intended for continuous use, so vacuum re-established intermittently throughout drying
- Oven set to 50 °C after 19.5 hours of drying
- Oven set to 40 °C after 26.5 hours of drying

Results :

- Whole drying process took 73 hours with hiccups (14:30 10/10/19 to 15:30 13/10/19) for 46.35 g of suspension
- Material ejection occurred when vacuum established - liquid water evaporated causing suspension to bubble (occurred three times before permeable filter paper 'lid' attached)
- Filter paper had to be replaced after first 19 hours due to accidental tearing, provoking additional loss of material from final calculation as some MXene had dried on the paper
- In ~ 4 hours of drying at 50 °C, between hour 19.5 and 23.5, a 20 mbar increase in pressure was observed. 30 minutes after vacuum pump turned back on, baseline pressure re-established and condensed water evacuated.
- In ~ 22 hours of drying at 40 °C, between hour 26.5 and 48.5, a 40 mbar increase in pressure was observed
- Another 24.5 hours of drying saw no visible water condensation nor pressure change indicating fully dry sample
- Dry mass of sample was 600 mg, hence a 20% yield
- Continuous film formed (see attached .JPG files) with metallic appearance

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Attached files

VacuumOvenDrying.JPG

sha256: 2a8113fe91e7b400ad12a86f90f8eadfebe8e7c7a8e5223e655c024475978643



VacuumOvenDrying_Torn.JPG

sha256: 0b5755535a26655d38f334390c549159a1c554fbc2fd24c24e2601d7495e0c4b



Unique eLabID: 20221011-5d9ac47b35e32fe0dfab1aaec16fc056171c29fa

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=22>