

MXene synthesis II

Date: 2019-08-28

Tags: Synthesis Barsoum 2019 28/08/2019Synth

Created by: James Bird

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Goal : Synthesise double-sized (6g) batch of Ti_3C_2 MXene

Procedure :

Reaction

- 31 mL of 37 w/w% HCl (aq) added to 29 mL H_2O (l) in LDPE reaction vessel to give 60 mL total reaction volume
- 6.0047 g of Ti_3AlC_2 (Barsoum Group, Drexel University 2019 batch, 98%, < 75 μm) added
- 4.0033 g of LiF added
- Temperature controller set at 30°C and stirrer hotplate at 60°C
- Leave reaction to stir at temperature for 24 hours

Centrifugation

1. Washing phase: 10,000 rpm, 5 minutes, 15 °C, 2 repeats
2. Suspension phase: 3500 rpm, 5 minutes, 15 °C, 21 repeats
3. Concentration phase: 11000 rpm, 30 minutes, T = 15 °C

Homogeneisation

Speed-mix program

Spin rate / rpm	2000	2000	0	2000	2000
Time / s	120	120	120	120	120

Storage

Final products degassed with inert gas (N_2), sealed with parafilm and stored in fridge. 1 L bottles of supernatant degassed and sealed, but stored at room

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temperature prior to concentration step.

Results:

The first litre of collected suspension was suspended immediately after the brief washing phase. During the suspension phase, collection of MXene to be classified as product should only be collected once the supernatant appears notably more viscous and darker in colour, this typically coincides with inflation/swelling of the sediment (see InflatedMXene.JPG). Supernatant collected before this step is liable to flocculate (see AcidFlocculation.JPG). pH neutrality (≈ 6) should be verified with Universal Indicator paper before collecting supernatant. The characteristic inflation of the material is a visual cue that water has intercalated sufficiently between MXene layers to encourage delamination. Supernatant collected after this point will appear as in the attached image (MXeneSupernatant.JPG). The flocculation in the attached image was triggered by the degassing step.

Continuation

Sediment and product collected, homogenised and stored as above.

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Attached files

InflatedMXene.JPG

sha256: 5a05acc6cb2e3d82ee0b6302c8cb7d106d2a81ba1d0ed4dd44be5e1602d422d9



AcidFlocculation.JPG

sha256: e1b71831a5041d5aaece1e076e31d98f095728b051576538671bbf12b5dce2f3



MXeneSupernatant.JPG

sha256: 8b7fe67d2d2735330fc07d8f1aee5e6f8ae1c563b761b26f4ade8b8d280f683d



Unique eLabID: 20221007-9926d7a67e6ea5170a01c36317911580f3c1afea

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=10>