

Freeze-casting of Ti₃C₂ MXene suspension I

Date: 2021-10-18

Tags: Freeze-cast 11/10/2021Synth Nanoplexus 200 2021

Created by: James Bird

1 / 4

Goal : Freeze-cast Ti₃C₂T_z aqueous nanoparticle suspensions to give well-aligned nanoparticles in a sample within the target dimensions and tolerances

Procedure :

- Add necessary quantity of lyophilizer beakers (Labconco) to ultra-low temperature freezer at - 81 °C and note room temperature (15 °C)
- MXene synthesised in [\[Experiment\] MXene synthesis VIII](#) is speed-mixed with the protocol below, to give a smooth, homogeneous paste with no signs of oxidation

Spin rate / rpm	500	1000	1500	2250	3000
Time / s	30	30	30	120	120

- Assemble template using Vaseline jelly to seal joints and to adhere to aluminium top-plate
- Ensure excess Vaseline is removed with a clean cotton bud
- Pour homogenous suspension into a section of the template, flatten with a spatula and place atop the freeze-plate
- Set target temperature to - 70°C on liquid nitrogen/water flow controller with a ramp rate of - 5 K/min
- Allow uni-directional freezing to complete, so that the freeze-front has visibly reached the entire sample surface
- Don waterproof cryo gloves
- Remove a lyophilizer beaker from freezer
- Remove top-plate with the attached template, deconstruct the frame and remove the sample
- Add sample to beaker and return to freezer - remove cryo gloves
- Set target temperature to room temperature on flow controller with a ramp rate of +10 K/min

The above procedure is repeated, with thorough cleaning of the template and top-plate after each sample is made. Cleaning is carried out with deionised water and isopropanol-soaked disposal paper towel, in that order. The two samples have an

Freeze-casting of Ti₃C₂ MXene suspension I

Date: 2021-10-18

Tags: Freeze-cast 11/10/2021Synth Nanoplexus 200 2021

Created by: James Bird

2 / 4

initial suspension concentrations of 1.287 ± 0.086 wt% (13.0 mg/mL). Once all samples have been made, they are attached to the lyophilizer in their beakers.

Results :

Attached .JPEG files are photographs of the samples, as detailed here:

- IMG_1658 shows Sample #1 prior to freeze-casting, hence the suspension held in the template atop the top plate
- IMG_1661 shows Sample #2 prior to freeze-casting

Details of the lyophilized samples are summarised in the table below, where 'dim.' is an abbreviation for 'dimension':

Sample / #	Length (long dim. // to freeze-plate) / mm	Width (short dim. // to freeze-plate) / mm	Height (⊥ to freeze-plate) / mm	Volume / cm ³	Mass / mg	Density / g cm ⁻³
1	118	15	15	26.55	456.9	1.72×10^{-2}
2	56	15	< 15	n/a	182.8	n/a

An attempt to calculate the height of Sample #2 was futile, when accounting for the material used previously: 566 mg yield from [\[Experiment\] MXene synthesis VIII](#), minus air-dried masses for yield calculation gives 195.7 mg remaining. $195.7 \text{ mg} / 1.30 \times 10^{-2} \text{ g cm}^{-3}$ gives 15.05 cm^3 estimated volume. With known length and width of the frame, height estimated to 17.9 mm which would exceed the template height. This is unsurprising considered the loss of suspension when transferring across containers.

Weighing of the Sample #2 aerogel gave a mass of 182.8 mg, which would also suggest a height of 16.7 mm by the same logic. These findings, along with the density of Sample #1 with known sample dimensions, suggest either an incorrect initial concentration or incomplete drying of aerogels. Both samples are attached to the freeze-drier at 12:56 pm on the Friday, and removed Monday morning, hence the drying time should be sufficient. Two options then remain: incorrect

Freeze-casting of Ti₃C₂ MXene suspension I

Date: 2021-10-18

Tags: Freeze-cast 11/10/2021 Synth Nanoplexus 200 2021

Created by: James Bird

3 / 4

initial concentration or strongly bound water remains.

Sample #1 cracked in two on removal from the freeze-frame and placing on the benchtop, perhaps due to thermal shock. The cracks propagated from areas where ridges are located in the freeze-template, suggesting a design change is required. Sample #2 is short-filled, so dimensions, volumes and densities are rough. The actual ramp rate for Sample #2 was much slower (-1.54 K/min) than the target ramp rate (-5 K/min) which suggests an insufficient supply of liquid nitrogen for cooling.

Conclusions :

- No leakage occurred now that vaseline is frequently used to seal template joints
- Ramp target temperature of -70 °C appears to be sufficient for complete freezing of sample 15 mm in height
- New freeze-template needs designing to prevent sample cracking
- If in any doubt of liquid nitrogen supply it must be replenished

Attached files

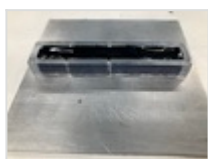
IMG_1661.JPEG

sha256: 1ae5297cf3e6725732a44495128eb347a83524d0fbc0c2cd6dc30596915aed7b



IMG_1658.JPEG

sha256: fcc0e8ad0eb2dbd2226cc1b398b2623cc740e25c4739e14a7d387a579f511e71



Freeze-casting of Ti₃C₂ MXene suspension I

Date: 2021-10-18

Tags: *Freeze-cast 11/10/2021Synth Nanoplexus 200 2021*

Created by: James Bird

4 / 4



Unique eLabID: 20230415-ac16c0d7d74d89d48a1d6bf320af9835849cec1b

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=103>