

Microanalysis of MXene synthesis products I

Date: 2022-01-17

Tags: Nanoplexus 400 2021 11/10/2021Synth 19/10/2021Synth Microanalysis 16/11/2021Synth 06/12/2021Synth KTH Collab Nanoplexus Ti3C2Tz 2021

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Goal :

Obtain composition of $\text{Ti}_3\text{C}_2\text{T}_z$ synthesis products and sediments using microanalytical techniques

Materials:

Offcuts of MXene from punch-pressing in [Experiment - Punch pressing of freeze-cast, lyophilized and calendered MXene aerogels](#), with MXene synthesized in [Experiment - MXene synthesis VIII](#).

Ready-ground powder from [Experiment - DSC & TGA Sample preparation](#), with MXene synthesized in [Experiment - MXene synthesis IX](#).

Failed calendering of a aerogels in [Experiment - Calendering of freeze-cast MXene aerogel III](#), where the MXene is synthesized in [Experiment - MXene synthesis X](#).

1 mL of suspension, at 48.2 mg/mL, fabricated in [Experiment - MXene mixing, dilution, homogenization and freeze-drying](#), with MXene synthesized in [Experiment - MXene synthesis XI](#).

The sediments of the latter two syntheses ([Experiment - MXene synthesis XI](#) & [Experiment - MXene synthesis X](#))

Procedure :

Chemical microanalysis is offered as a service, so submission of freeze-dried and ground powder is required only.

Suspension sample is added to an ultra-low temperature freezer at $-81\text{ }^{\circ}\text{C}$, then attached to a freeze-drier once frozen, and left until dry.

All dry powders are ground in an alumina pestle and mortar until visually fine. Each sample product collected in a vial, with average mass $52.4 \pm 22.1\text{ mg}$. Using the combination of three analytical techniques (ICP-AES, CHN analysis and halide analysis titration), technicians derive concentrations of elements C, H, Cl, Ti and Al and Li.

Results :

Bal. denotes the balanced summation to 100% of the total sample weight. The only undetectable elements that are expected to be present in the sample are O & F.

Synth Date	Type	Composition						
		C / wt%	H / wt%	Cl / wt%	Ti / wt%	Al / wt%	Li / wt%	Bal. / wt%
11/10/2021	Product	12.92	1.15	4.12	63.64	0.42	0.36	17.39
19/10/2021	Product	8.64	0.56	3.01	48.7	0.32	7.44	31.33
16/11/2021	Product	10.59	0.61	4.71	66.07	0.35	0.37	17.3
06/12/2021	Product	9.96	0.62	4.89	66.99	< 0.3	0.72	16.82
16/11/2021	Sediment	10.51	0.39	4.3	69.59	2.08	0.33	12.8
06/12/2021	Sediment	10.23	0.36	3.4	69.25	3.85	< 0.3	12.91

After some calculations (see uploaded .xlsx), it appears that the synthesis product 19/10/2021 is oxidized, considering the marked Ti:Bal. stoichiometric ratio of 0.55, where the balance component is presumed to be a 50/50 split of O & F, as the only elements predicted to be present which cannot be directly measured. The same sample also carries a large quantity of lithium, which is probably due to the peculiarities of the etch (see [Experiment - MXene synthesis IX](#)) meaning that initial concentrations may have been inaccurate. The Ti:Bal. ratio of all other dry products is in the range 1.37 ± 0.08 , which is in the correct range on the presumption from theory that the $Ti:T_x$ stoichiometric ratio is 1.5 if $x = 2$. If, however, chlorine and hydrogen are considered T_x , then the average $Ti:T_x$ ratio of the non-oxidized products decreases to 0.72 ± 0.13 , meaning that $x = 4.2$. This finding suggests that not the entire fraction of any of the Bal. elements (O & F), Cl or H can be attributed to the MXene, and are instead present in other phases. This finding hinders the ability to calculate an accurate purity value for any synthesis product derived from stoichiometry. The minimum MXene concentration by weight percentage, for when $x = 0$, can however be calculated on the assumption that any residual Al is still bound within the parent MAX phase. The average across the three, non-oxidized syntheses is 75.1 ± 1.6 wt%.

Noticeably, although the sediment analyses do contain more aluminium, and hence presumably more MAX phase, from the ratio of Ti:Al there could still be > 50 % MXene in each sample. Separation techniques could be optimised to further enhance yield.

Conclusions:

Good values of purity (> 75%) for MXene synthesis products are obtained for those synthesised via conventional routes.

Attached file

2022-01-17.xlsx

sha256: a4e998e5159b8254146e7c1269d7dd401d5ec97b71dc124e17fb08ff577831c6



Unique eLabID: 20230527-6e4f70506536d93fbbd45c4c769d13bb10aad271

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