

# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: Training XRD Synthesis Optimisation Rigaku

Created by: James Bird

1 / 10

**Goal : Run typical, coupled  $\theta$ :2 $\theta$  scan of dropcast  $\text{Ti}_3\text{C}_2$  MXene synthesis product to confirm successful synthesis**

Procedure :

## Sample preparation

- Dropcast bulk concentration product suspensions from each synthesis run in [\[Experiment\] Optimisation of MXene Synthesis - Execution of Plackett-Burman Screening Design](#) on to zero-background holders (ZBH) made of oriented single-crystal silicon and ensure coverage of area 10 x 10 mm.
- For runs #4-12, measurements were started before all solvent (water) had evaporated
- For all runs, measurements were also taken once the dropcast suspension dried and all solvent evaporated

## Instrument set-up

Geometry	Bragg-Brentano
Goniometer	SmartLab (In-plane) with $\theta$ $\phi$ Z cradle and xy-20mm attachment
Detector	HyPix 3000(H)
X-ray source	Copper
Radiation	$K_{\alpha 1}$ = 0.154059 nm, $K_{\alpha 2}$ = 0.154441 nm, $K_{\alpha}$ ratio 0.4970, $K_{\beta}$ = 0.139225 nm
Incident beam optics	CBO parallel beam, 5 ° Soller, 1 mm incident slit and 10 mm length limiting slit
Diffracted beam optics	20 mm receiving slit, 5 ° Soller, 38.5 mm receiving slit #2
2 $\theta$ start:finish:step / °	3:100:0.02
Dwell time / s	1.32

# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: Training XRD Synthesis Optimisation Rigaku

Created by: James Bird

2 / 10

## Results :

Run / #	Condition	Filename
1	Dry	Dry_Ti3C2xaq_Run1_20210420
2	Dry	Dry_Ti3C2xaq_Run2_20210420
3	Dry	Dry_Ti3C2xaq_Run3_20210420
4	Dry	PB_5mmSS_10mmDS_Ti3C2Tx_aq_DRY_IS1mm_5mmSS_20210322
5	Dry	PB_5mmSS_10mmDS_Ti3C2Tx_aq_5_Dry_IS1mm_5mmSS_20210322
6	Dry	PB_5mmSS_10mmDS_Ti3C2Tx_aq_6_Dry_IS1mm_5mmSS_20210322
7	Dry	PB_5mmSS_10mmDS_SynthOpt_Dropcast_Run7_Dry_IS1mm_5mmSS_20210329
8	Dry	PB_5mmSS_10mmDS_TiC2Tx_aqt_Dropcast_Run82_Dry_IS1mm_5mmSS_20210329
9	Dry	PB_5mmSS_10mmDS_Ti3C2Tx_aq_Dropcast_Run9_Dry_IS1mm_5mmSS_20210330
10	Dry	PB_5mmSS_10mmDS_Ti3C2Tx_aq_Dropcast_Run10_Dry_IS1mm_5mmSS_20210330
11	Dry	PB_5mmSS_10mmDS_SynthOpt_Dropcast_Run11_Dry_IS1mm_5mmSS_20210329
12	Dry	PB_5mmSS_10mmDS_SynthOpt_Dropcast_Run12_Dry_IS1mm_5mmSS_20210329
13	Dry	Dry_Ti3C2xaq_Run13_20210420
14	Dry	Dry_Ti3C2xaq_Run14_20210420
15	Dry	Dry_Ti3C2xaq_Run15_20210420
16	Dry	Dry_Ti3C2xaq_Run16_20210420
4	Wet	PB_5mmSS_10mmDS_Ti3C2Tx_aq_IS1mm_5mmSS_20210322
5	Wet	PB_5mmSS_10mmDS_Ti3C2Tx_aq_5_WET_IS1mm_5mmSS_20210322
6	Wet	PB_5mmSS_10mmDS_Ti3C2Tx_aq_6_Wet_IS1mm_5mmSS_20210322
7	Wet	PB_5mmSS_10mmDS_SynthOpt_Dropcast_Run7_Wet_IS1mm_5mmSS_20210329
8	Wet	PB_5mmSS_10mmDS_TiC2Tx_aqt_Dropcast_Run82_Wet_IS1mm_5mmSS_20210329
9	Wet	PB_5mmSS_10mmDS_Ti3C2Tx_aq_Dropcast_Run9_Wet_IS1mm_5mmSS_20210330
10	Wet	PB_5mmSS_10mmDS_Ti3C2Tx_aq_Dropcast_Run10_Wet_IS1mm_5mmSS_20210330

# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: Training XRD Synthesis Optimisation Rigaku

Created by: James Bird

3 / 10

11	Wet	PB_5mmSS_10mmDS_SynthOpt_Dropcast_Run11_Wet_IS1mm_5mmSS_20210329
12	Wet	PB_5mmSS_10mmDS_SynthOpt_Dropcast_Run12_Wet_IS1mm_5mmSS_20210329

Files suffixed with \_Theta\_2-Theta.txt contain xy columnar data of  $2\theta$  vs intensity, .ras files are raw outputs from the diffractometer and .png are images of the plotted data.

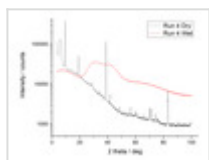
## Conclusions:

These diffraction patterns were primarily collected as qualitative proof of successful (partial)  $\text{Ti}_3\text{C}_2$  MXene synthesis from  $\text{Ti}_3\text{AlC}_2$ , which is confirmed in all instances to various degrees by the varying intensity/area peaks assigned to  $\text{Ti}_3\text{C}_2$  or  $\text{Ti}_3\text{AlC}_2$  diffracting planes. The still wet dropcast product suspension plots are largely uninformative and unremarkable, although in many instances it is evident that the suspension has dried throughout the duration of the 105 minute measurement - indicated by perfectly overlapping plots at higher angles. The unremarkable conclusion to draw is that nanoparticles in suspension need time to settle into ordered diffracting domains as solvent evaporates, or material presence will be dominantly indicated by an amorphous background.

## Attached files

Run4\_DropcastXRD.png

sha256: 362c1269c5abcd92b1b8d0743d2c7327b4ab1c981bac1d415f457df2937c618d



Run12\_DropcastXRD.png

sha256: 908e49a17ef5624b3e6b89b37e93cd2a554ed83e02e4604c9d9b5c920c0bec8f

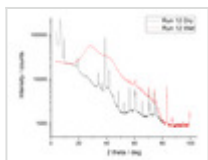
# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: *Training XRD Synthesis Optimisation Rigaku*

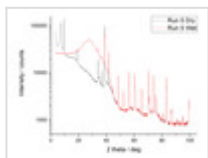
Created by: James Bird

4 / 10



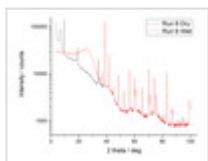
Run5\_DropcastXRD.png

sha256: b4ead7fb11308c3c1d869c71bdd144e0fe9c2a867a3e898bd00245ab5840b05a



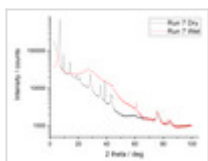
Run6\_DropcastXRD.png

sha256: cbbec725d1eb8be42b0b61683888bf773f74d4c8350c8c259cec2986055dd13b



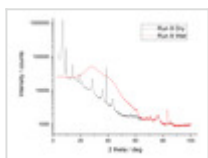
Run7\_DropcastXRD.png

sha256: bc146a929e4ff78789d6164d697f7dbc1a18956de8b11b5f8e3f792a108c1e90



Run8\_DropcastXRD.png

sha256: e9953aa7af5e6ebb8ef64035116afa4b8bde0527e419c56d4b4de353b3dd05f3



Run9\_DropcastXRD.png

sha256: d5c0f4f82b1bc39cc39ff5e208d526625361833e62ab1ddf7ff5615245ca7820

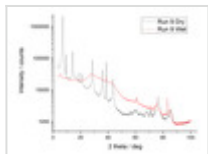
# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: Training XRD Synthesis Optimisation Rigaku

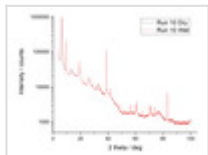
Created by: James Bird

5 / 10



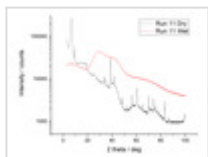
Run10\_DropcastXRD.png

sha256: fba7896ebe47640d8160316a2cec45a160360b8cc29482944020250f2cf9b51b



Run11\_DropcastXRD.png

sha256: 1b3f902913a1385353d8687d6ab89a9a8b9d5c586a5402d9095f1df393f0dd27



PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_6\_Wet\_IS1mm\_5mmSS\_20210322.ras

sha256: a2cec77593597bda4a978f06e203a34f276923a3f89e70b829d42a09b51e8c97

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_6\_Dry\_IS1mm\_5mmSS\_20210322.ras

sha256: dbde33019628b690fd2ad319805ac07032349fb8147d0b60abe4b1273a840d4d

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run9\_Dry\_IS1mm\_5mmSS\_20210330.ras

sha256: 6aa2d050a6a023850e762c2caf4ace0a6f06700bba6d42cb069ab2c5bb84dad7

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run9\_Wet\_IS1mm\_5mmSS\_20210330.ras

sha256: dcf810a727921737ff3d4df84d50a631f5f79c6c9b6564c27360fe236b74ab79

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run10\_Dry\_IS1mm\_5mmSS\_20210330.ras

sha256: 07af16e95b211bf3a2a3747110a4e1a571810c90fc223d4085c8de200fe12ba3

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run10\_Wet\_IS1mm\_5mmSS\_20210330.ras

sha256: de2de2736237c749a4d07af5e2ed7dc4b05e2bbe5a57de71b78443b2cb8e2e6b

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_DRY\_IS1mm\_5mmSS\_20210322.ras

# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: *Training XRD Synthesis Optimisation Rigaku*

Created by: James Bird

6 / 10

sha256: d127307040abed0c4ca3a27a2de5c2f6318e8d519529ecadcb3dc9a50f5734b2

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_IS1mm\_5mmSS\_20210322.ras

sha256: 36eeff797f649f94b5ea04f1aa80bc8f625e59f32cc8daed544a193f9a5ab366

PB\_5mmSS\_10mmDS\_TiC2Tx\_aqt\_Dropcast\_Run82\_Dry\_IS1mm\_5mmSS\_20210329.ras

sha256: 770c8e9f7ad92a3d48ec292e5bf0e1a2b3f5b994e6708a64f5428055177dba19

PB\_5mmSS\_10mmDS\_TiC2Tx\_aqt\_Dropcast\_Run82\_Wet\_IS1mm\_5mmSS\_20210329.ras

sha256: f7f8ff7c9e26e8ecd491fdb8332aff4a4464567fb6ad6cf6316e6dc7456c4

Dry\_Ti3C2Txaq\_Run1\_20210420.ras

sha256: f138d816af4157dbbd6cfd2c39029767ed61e9b153e217e61d7f5e66cea20ba9

Dry\_Ti3C2Txaq\_Run2\_20210420.ras

sha256: 93814fa7af915b599252eade8fb2848dbb575da030165c345e2e4857c39fbd12

Dry\_Ti3C2Txaq\_Run3\_20210420.ras

sha256: cd15be0c834a5133dfbc84f0e57cfd4ca50937357b5c312b73e95e50f4b0f456

Dry\_Ti3C2Txaq\_Run13\_20210420.ras

sha256: 31ae432f960ab86ddcc94ebf1e9edb6559eb54efb318d76d72a9e8dbf62cf074

Dry\_Ti3C2Txaq\_Run14\_20210420.ras

sha256: bb57ed346139111751119d16707b6506e134b10b3823a955f3c1724e797e59b5

Dry\_Ti3C2Txaq\_Run15\_20210420.ras

sha256: f958e3b529322cb6ad89f01b13b56b486f09356063f1f4961af52f6446127314

Dry\_Ti3C2Txaq\_Run16\_20210420.ras

sha256: 91a213797ba6297f488cc4c41b43ee21c06ca070f93e7f43901591f2bb416708

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run7\_Dry\_IS1mm\_5mmSS\_20210329.ras

sha256: a5d4f31abfeffb43d1b40b886adef714d87154a2a684a7596be726db7fa917a

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run7\_Wet\_IS1mm\_5mmSS\_20210329.ras

sha256: 96aef9967d8cd5ab77e9959e2e2506d0ca2feef2c5c9af537ebb35c43037a622

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run11\_Dry\_IS1mm\_5mmSS\_20210329.ras

# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: *Training XRD Synthesis Optimisation Rigaku*

Created by: James Bird

7 / 10

sha256: 984f603bcebe94d8f87bd4c5b48be10d0e45d6536273ee2b7bd42b6cd40b845d

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run11\_Wet\_IS1mm\_5mmSS\_20210329.ras  
sha256: 8a1d7657b3d62e9dff5c0543251384805905d956121edb87762d2f04eff9d67

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run12\_Dry\_IS1mm\_5mmSS\_20210329.ras  
sha256: 0d2f0ba718d6618c9c56e89f3cd6df72609f9466fb5d0ca19c28a6a74a6859f3

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run12\_Wet\_IS1mm\_5mmSS\_20210329.ras  
sha256: e9e4c8e17cbbf3ef5a9aa8d25efe8bb4ba9fe9a502729a6b070e0a1574f5cbf0

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_5\_Dry\_IS1mm\_5mmSS\_20210322.ras  
sha256: c8db7da8efdf7e1aa98ced2895674366ab8875489705f9629130af0e63092d0f

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_5\_WET\_IS1mm\_5mmSS\_20210322.ras  
sha256: cff54fdb4ced623aac5858bd127f988cdfc02ebaa7c2ddf9b1bb2d77430e0b89

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_5\_WET\_IS1mm\_5mmSS\_20210322\_Theta\_2-Theta.txt  
sha256: d0aff317ba6b5e95ce14691864711f437e44ba93e862d9849dfa885b0ffb01a8

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_6\_Dry\_IS1mm\_5mmSS\_20210322\_Theta\_2-Theta.txt  
sha256: 48e25b541c7cb1575b42484986a6ba74add4b71b5b519846bb20b16d3372874b

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_6\_Wet\_IS1mm\_5mmSS\_20210322\_Theta\_2-Theta.txt  
sha256: a8799286bf0c1d03ed250643b2a9f7c07b2ffd315badc2397ed757e332654b53

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run9\_Dry\_IS1mm\_5mmSS\_20210330\_Theta\_2-Theta.txt  
sha256: 574ef64f511d656048ad7838086bff49b87a1765fff2ec3a80dee0bb05060665

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run9\_Wet\_IS1mm\_5mmSS\_20210330\_Theta\_2-Theta.txt  
sha256: 0bb4a3083e8d9cdcb6a4bd9e945247c6de35e3d9876f539307c10ea70b37fa3c

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run10\_Dry\_IS1mm\_5mmSS\_20210330\_Theta\_2-Theta.txt  
sha256: 705bc650503103e6d78836e41c46960b6d1e5837192feb90bf534b434510bdfa

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_Dropcast\_Run10\_Wet\_IS1mm\_5mmSS\_20210330\_Theta\_2-

# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: *Training XRD Synthesis Optimisation Rigaku*

Created by: James Bird

8 / 10

Theta.txt

sha256: c738a18b1b5c3dca2f4cadb1225df2b059a63943943a71c55d7af05b6093bc25

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_DRY\_IS1mm\_5mmSS\_20210322\_Theta\_2-Theta.txt

sha256: 1c0d3283fa84d082d9e73638949301cd1f952f51477f0ee965c77d3310e407f8

PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_IS1mm\_5mmSS\_20210322\_Theta\_2-Theta.txt

sha256: a96795226a99d2095676ae13abe06bb2ea77ceec01e69ef151102ea655687436

PB\_5mmSS\_10mmDS\_TiC2Tx\_aqt\_Dropcast\_Run82\_Dry\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt

sha256: e0cf72af558ffe5131bf1f22b89963933a07bb3cdf40165a1bc8f53b09f02228

Dry\_Ti3C2Txaq\_Run1\_20210420\_Theta\_2-Theta.txt

sha256: 320c1dd6a0017c0bf5ff2e82330aa024f442512746b2292e129b100f833d21f2

PB\_5mmSS\_10mmDS\_TiC2Tx\_aqt\_Dropcast\_Run82\_Wet\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt

sha256: f0b1e886647ceee5d55c0d6863f73d9fd8ea2edc24ca8bb3f683b9f4f6b636ff

Dry\_Ti3C2Txaq\_Run2\_20210420\_Theta\_2-Theta.txt

sha256: e00780605c7f84f8602e753ad324d1bb4983e81a680b226845111eaf606edfba

Dry\_Ti3C2Txaq\_Run3\_20210420\_Theta\_2-Theta.txt

sha256: 93c8194a67a60fce1af743c9733ce78a8b9178e229782f3be7bf3d16cd559439

Dry\_Ti3C2Txaq\_Run13\_20210420\_Theta\_2-Theta.txt

sha256: eee4eb3fc3a93a859c079c435ef37af9a10f94318d6b8c4328c8d0997c25a9f3

Dry\_Ti3C2Txaq\_Run14\_20210420\_Theta\_2-Theta.txt

sha256: 3da5a62be3ef2d0739a2d32e678f045cb4b1508093e02b1ed632514a1c5d6aaa

Dry\_Ti3C2Txaq\_Run15\_20210420\_Theta\_2-Theta.txt

sha256: 876c70606057dc97fd298ab93128198a5a76d0bd93adc85c0677a21055693e99

Dry\_Ti3C2Txaq\_Run16\_20210420\_Theta\_2-Theta.txt

sha256: 95109c4cbc63eda5009c757f67a1463ff0ee6760239a1184df15fa078e3ba94f

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run7\_Dry\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt



# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

Tags: Training XRD Synthesis Optimisation Rigaku

Created by: James Bird

9 / 10

sha256: 3bb4bde3c54b0e98ba10821d524768930b96d58e184e060442f382b01345dd0c

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run7\_Wet\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt

sha256: 8047ae70bd8a6807b410c9e25d51956f315daf45c1b5202a2124f920408081e0

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run11\_Dry\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt

sha256: 89abcc2cee6e6b1854a4b31285b34f5aeb3daf8057a05a9521185c14bc2b2314

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run11\_Wet\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt

sha256: 1958670a13a70402a892f3f185700c084f9957445cb6be719989457b68962ad3

PB\_5mmSS\_10mmDS\_SynthOpt\_Dropcast\_Run12\_Dry\_IS1mm\_5mmSS\_20210329\_Theta\_2-Theta.txt

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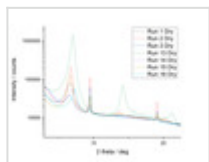
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PB\_5mmSS\_10mmDS\_Ti3C2Tx\_aq\_5\_Dry\_IS1mm\_5mmSS\_20210322\_Theta\_2-Theta.txt

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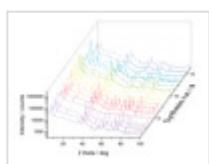
SynthOpt\_DryDropcastOnly\_LowAngle.png

sha256: 18d13c9ae48b69606d96ea64691af061c4a11ebf26948051cbaefea5eddc60a1



SynthOpt\_AllDryDropcast.png

sha256: aed4c0a969db37cfb522cd7e1b440d1d9020c1fc3c3a48989a74ba1b6aa8bd0e



# XRD analysis of dropcast MXene suspensions - Rigaku

Date: 2021-03-22

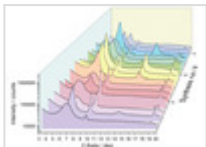
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Created by: James Bird

10 / 10

SynthOpt\_AllDry\_LowAngle.png

sha256: 4b1f1cd755cadb8c597f391943288454d9cda6c9f9fba12a23717bb23073e6fe



Unique eLabID: 20221109-76533da2497e608ca658c6811c15069e08c79a91

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=69>