

Dynamic Light Scattering (DLS) for nanoparticle size-distribution acquisition IV

Date: 2021-09-17

Tags: PSD DLS 18/08/2021Synth

Created by: James Bird

1 / 5

Goal : Obtain particle size distributions (PSDs) of Ti_3C_2 MXene nanoparticles in aqueous suspension using DLS

Procedure :

Sample preparation

- Bulk concentration Ti_3C_2 product suspension, synthesised in [\[Experiment\] MXene synthesis VII](#) and diluted to 2.92 wt% in [\[Experiment\] Freeze-casting of \$\text{Ti}_3\text{C}_2\$ MXene suspension](#), is serially diluted to give sample suspensions with a volume of ~2mL. Dilutions are detailed in the table below, where suspension density is approximated to 1 g cm^{-3} for concentration calculations.

Concentration / wt%	Volume of previous suspension / μL	Volume of deionised water / μL
1.88	1289	711
9.4×10^{-1}	1000	1000
1.9×10^{-1}	400	1600
9.4×10^{-2}	1000	1000
1.9×10^{-2}	400	1600
9.4×10^{-3}	1000	1000
1.9×10^{-3}	400	1600

- Dilution is carried out by extracting a quantity of the bulk suspension using a Gilson Pipetman P200 micropipette and D200 tips and transferring to a glass vial. The desired quantity of deionised water is measured in the same way.
- If a stated target volume exceeds 1 mL, then a 1mL disposable pipette is used to transfer that quantity
- Suspensions are homogenised throughout with vortex mixing
- Final concentration suspensions transferred to cuvette with Pasteur pipette - cuvette exterior gently dried if necessary

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2 / 5

IMG_1401.JPEG is a photograph of the serially diluted suspension stored in vials. Note they are incorrectly labelled, although they descend in concentration from left to right with concentrations in the table stated above. The lower concentration suspensions appear visibly much darker than anticipated, so some assumptions around concentrations made in [\[Experiment\] Freeze-casting of Ti₃C₂ MXene suspension](#) may be flawed.

Samples were unfortunately stored at the above diluted concentrations for eleven days prior to measurement, from a synthesis started thirty days prior to measurement. So despite degassing samples with inert gas, sealing with parafilm and storing under refrigeration, some oxidation will likely have occurred.

DLS operation

Standard Operating Procedure (SOP) settings (size measurement type):

- Narrow band filter fitted
- Material properties (RI = 1.7, absorption = 0.9)
- Water dispersant (η = 0.8872 cP, RI = 1.330)
- Use dispersant viscosity as sample viscosity
- Temperature = 25 °C with 120 s equilibration time
- DTS1070 folded capillary cell *only*
- 173 ° backscatter measurement angle
- Automatic measurement duration
- Three measurements per sample
- Automatic attenuation selection and positioning method seeking optimum
- General purpose (normal resolution) analysis model *only*

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3 / 5

Results :

All measurement outputs and variables indicative of data quality are detailed in the table below. All individual measurements returned a polydispersity index (PDI) > 0.1, which defines the threshold below which outputs can only be compared quantitatively. Despite this, the combination of three measurements for two sample concentrations did meet the data quality criteria defined in the analysis program (Zetasizer) : all data quality indicators, as well as the concentration are highlighted in green in those rows.

Concentration / wt%	Z-average / d.nm	Z-average std / d.nm	Number mean / d.nm	Number mean std	PDI / dimensionless	PDI std	Derived mean count rate / kcps	Derived mean count rate std	Intercept	Intercept std	In range / %	In range std
1.88	3463	523.1	51.73	85.64	1	0	263.8	115.3	0.308	0.038	60.3	1.73
9.4×10^{-1}	2418	132.1	57	2.07	1	0	144.5	11.2	0.712	0.041	87.1	4.28
1.9×10^{-1}	3213	200.7	132.3	194.3	1	0	70.8	10.3	0.688	0.0423	65.9	6.46
9.4×10^{-2}	5994	1175	102.6	89.05	1	0	140.2	11.0	0.551	0.0895	47.8	5.95
1.9×10^{-2}	2302	54.28	1012	761.7	0.353	0.046	2146.1	67.4	0.948	0.00896	92.5	2.33
9.4×10^{-3}	1181	43.71	789.4	182.4	0.247	0.039	6704.7	100.5	0.942	0.00361	95.2	0.666
1.9×10^{-3}	1177	70.12	801.6	19.82	0.365	0.028	6826.6	298.8	0.949	0.008	90.6	0.802

The yellow-highlighted concentration has three of the four quality indicator variables in a suitable range across all measurements, and hence is deemed reasonably accurate. Common to the intensity PSDs of green and yellow-highlighted samples is a sharp drop-off in density at the larger end of the particle dimension scale: this corresponds to a poor distribution fit to the correlogram on longer timescales, corresponding to those larger particle dimensions which extend beyond the measurement capability of the software under these conditions.

.dts is the raw datafile which can be read into the Zetasizer software program, .csv is an exported, comma-separated summary of the .dts datafile and .png files are the plotted data; particle diameter (on a log scale) is plotted against the mean percentage of that diameter interval contributing to the intensity-based and number-based particle size distribution for each concentration, labelled iPSD and

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4 / 5

nPSD, respectively. Vertical lines spanning the whole plot height are mean Z-average and number mean values (quoted above), where the regions of matching colour spanning left and right of this value correspond to its standard deviation.

Conclusions:

Z-average values for the measurements that met all DLS criteria do not overlap, although both of the most dilute concentration samples have overlapping number-based and intensity-based average particle sizes. The shape of each of the PSDs (both intensity and number-based) do however vary for each of these concentrations (see DLS_2021-09-17_PSDComp.png). These findings again cast doubt on the use of DLS to produce PSDs of $\text{Ti}_3\text{C}_2\text{T}_z$ synthesis products, even with concentrations $\leq 1 \times 10^{-2}$ wt%.

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5 / 5

Attached files

IMG_1401.JPEG

sha256: 38726b0830fbbf64a6a6661f4ad04835d033d5256bebf015595ab26aed71fdc1



2021-09-17.dts

sha256: a2c1d9a0af15e2c7f73615b3adcea18ba95bb4845b5a1f2d9f44abf83fe6ee6d

2021-09-17_Size.csv

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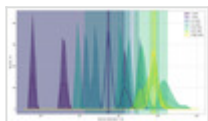
DLS_2021-09-17_iPSD.png

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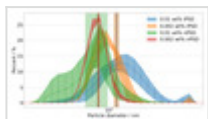
DLS_2021-09-17_nPSD.png

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DLS_2021-09-17_PSDComp.png

sha256: a9d47d1a6098800a1ff4f089a8c1840348ca8d1d3ccc5026598ad94457c5d590



Unique eLabID: 20230310-970aecc0ad871381fb283dad005a6bc71dc2096c
Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=92>