Dynamic Light Scattering (DLS) for nanoparticle size-distribution acquisiton III

Date: 2021-05-05
Tags: *PSD DLS Article 1 Synthesis Optimisation*
Created by: James Bird

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 suspensions, of concentrations given in [Experiment] Optimisation of MXene synthesis - Yield calculations, are diluted from to give a volume of ~1.2mL at 0.1 wt%. Dilutions are detailed in the table below, where suspension density is approximated to 1 g cm^{-3}

Run N° / #	Volume of bulk suspension / μL	Volume of deionised water / μL
1	51	1149
2	98	1102
3	50	1150
4	1091	109
5	62	1138
6	51	1149
7	308	892
8	250	950
9	52	1148
10	308	892
11	500	700
12	156	1050
14	107	1093
15	194	1000
16	65	1135

- Dilution is carried out by extracting a quantity of the bulk supension using a

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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

DLS operation

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

- Narrow band filter fitted
- Material properties (RI = 1.7, absorption = 0.9)
- Water dispersant (= 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*

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 one

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sample did meet the data quality criteria defined in the analysis program (Zetasizer) : all data quality indicators are highlighted in green in that row (Run #7).

Run N° / #	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	3641	515.3	2051	425.1	0.480	0.266	9771.5	3579.9	0.952	0.021	77.1	1.64
2	2888	633.1	344.4	97.41	0.986	0.025	8416.3	1262.7	1.42	0.194	81.9	1.18
3	1779	338.6	938.4	123.9	0.406	0.211	4813.3	702.7	1.01	0.091	81.2	1.01
4	286.2	65.6	92.83	35.67	0.440	0.053	599.3	25.2	0.837	0.008	89.2	1.83
5	1750	314.3	852.5	212.9	0.599	0.088	2910.5	353.7	0.920	0.045	77.1	2.35
6	2208	268.7	463.7	147.7	0.945	0.017	5305.3	913.4	1.17	0.095	76	5.14
7	540.5	6.086	277.3	15.43	0.409	0.031	373.9	0.8	0.841	0.003	96.7	0.84
8	812.9	134.4	251.8	26.57	0.691	0.040	15138.3	1336.4	1.07	0.035	88.5	1.80
9	2952	199	773	227.1	0.377	0.011	277.5	8.9	0.769	0.01	91.3	2.5
10	902.9	195.3	198.1	34.78	0.82	0.162	109.6	20.7	0.968	0.062	72.6	2.86
11	4935	942.2	2419	233.1	0.574	0.378	4473.5	821.3	0.965	0.086	69.7	1.2
12	1728	251.8	685.6	173.5	0.737	0.169	6584.3	871.2	1.04	0.122	77.9	0.85
14	3264	261	1148	642.7	0.534	0.438	2058.7	1107.1	0.956	0.057	65.5	2.62
15	1628	248.3	626.7	66.04	0.789	0.133	1459	225.8	0.991	0.061	76.1	2.46
16	2591	201.2	859.2	328.7	0.743	0.235	640.4	293.2	0.913	0.053	67.4	4.34

Runs #4 & 9 are highlighted in yellow, as three of the four quality indicator variables are in a suitable range, and the fourth (percentage in range) is greater than all other run averages. Common to all of Runs 4, 7 and 9 in the intensity PSDs is a sharp drop-off in density at the larger end of the particle dimension scale: this corresponds to a poor cumulants and distribution fit to the correlogram on longer timescales, corresponding to those larger particle dimensions. Run #5 is also highlighted in yellow as three of the four quality indicators are suitable, and shares a similar issue, in that the cumulants analysis fits well, but distribution analysis does not on longer timescales.

.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

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number-based particle size distribution for each synthesis run. 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:

The intercept regularly exceeding 1, PDI exceeding 0.6 and low in-range percentages suggest any one of a number of issues with the samples, including large or sedimenting particle presence, unsuitable suspension concentration or sample polydispersity too high for DLS measurements. In combination with the zeta-potential measurements (detailed in [Experiment] Zeta-potential measurement of MXene suspensions II), the first option can be ruled out for 11 of these measurements. Certainly, concentration optimisation should have been carried out for each individual synthesis run to ensure no impact stemmed from this variable, as this has a particle-size dependence on the optimal value. However, the fact that nearly all samples give a high multimodal fit error does suggest that this sample type may be inherently too polydisperse. The software does have an option to extend duration for large particles, which also should have been tested.

Attached files

2021-05-06_Size.csv
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2021-05-06.dts
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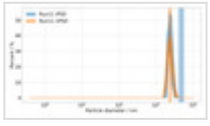

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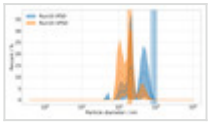

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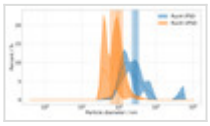

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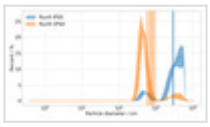

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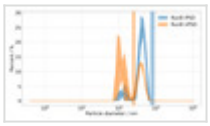

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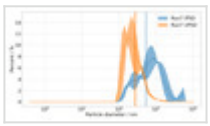

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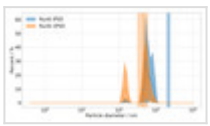

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DLS_SynthOpt_PSDComp_Run5.png
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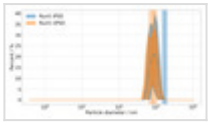

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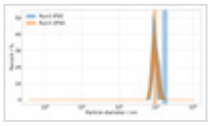

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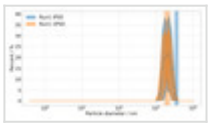

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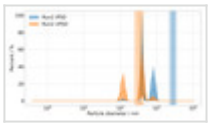

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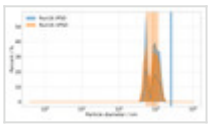

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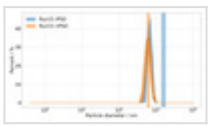

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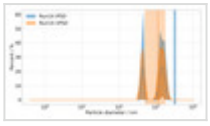

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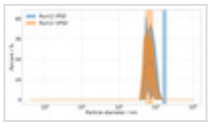

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