

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

Date: 2021-04-23

Tags: *PSD DLS Synthesis Optimisation*

Created by: James Bird

1 / 4

Goal : Identify optimal concentration in DLS to obtain particle size distributions (PSDs) of Ti_3C_2 MXene nanoparticles in aqueous suspension

Procedure :

Sample preparation

- Bulk concentration Ti_3C_2 product suspension (Run #13 in [\[Experiment Optimisation of MXene Synthesis - Execution of Plackett-Burman Screening Design\]](#)) is serially diluted from 4.52 wt% to give a minimum of 1mL suspension for each target concentration interval. Dilutions are detailed in the table below, where suspension density is approximated to 1 g cm^{-3}

Target suspension concentration / wt%	Volume of previous suspension / μL	Volume of deionised water / μL
4.52	-	-
1	660	1340
0.5	1000	1000
0.1	400	1600
5×10^{-2}	1000	1000
1×10^{-2}	400	1600
5×10^{-3}	1000	1000
1×10^{-3}	400	1600

- Dilution is carried out by extracting a quantity of the more concentrated, last-made 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
- Suspensions are homogenised throughout with vortex mixing
- Final concentration suspensions transferred to cuvette with Pasteur pipette - cuvette exterior gently dried if necessary

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

Date: 2021-04-23

Tags: PSD DLS Synthesis Optimisation

Created by: James Bird

2 / 4

DLS operation

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

- Narrow band filter fitted
- 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*

Results :

All measurement outputs 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 sample did meet the data quality criteria defined in the analysis program (Zetasizer) - highlighted in green in the table below.

Concentration / wt%	Z-average / d.nm	Z-average std / d.nm	PDI / dimensionless	PDI std
1×10^{-3}	2112	627.1	0.961	0.068
5×10^{-3}	1714	380.2	0.857	0.126
1×10^{-2}	2192	1049	0.778	0.030
5×10^{-2}	1746	201.5	0.562	0.109
0.1	1951	38.22	0.234	0.052

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

Date: 2021-04-23

Tags: *PSD DLS Synthesis Optimisation*

Created by: James Bird

3 / 4

0.5	2925	289.3	0.776	0.222
-----	------	-------	-------	-------

.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 is the plotted data; particle diameter (on a log scale) is plotted against the mean percentage of that diameter interval contributing to the intensity-based particle size distribution. Vertical lines spanning the whole plot height are mean Z-average values (quoted above), where the regions of matching colour spanning left and right of this value correspond to its standard deviation.

Conclusions:

The low PDI resultant for a concentration was used as justification to set the concentration of all synthesis product suspensions at the same concentration. Unfortunately, as mentioned previously, concentration optimisation for one product suspension may not be applicable across all. It could also still simply be the case that these suspensions are too polydisperse for the algorithm to reliably fit the data to a log-normal distribution.

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

Date: 2021-04-23

Tags: *PSD DLS Synthesis Optimisation*

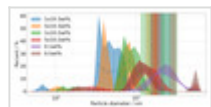
Created by: James Bird

4 / 4

Attached files

DLS_2021-04-23_Run13_ConcOpt.png

sha256: e25fdd68676710fa0ceda29d709308fb3e628f0224b4e390b72de4108a4f130c



2021-04-23.dts

sha256: 076babd6fb4ec1da1f946f80210766eb3ec8580bc01accfd9c46d5bb2921c284

2021-04-23_Size.csv

sha256: f592f3c4ce404664c4bfbcd1d72ffde6af553d19179db34a32886eff10d6be7a3



Unique eLabID: 20230119-0d9e5a16685164e941003b783c0859e6ecffcdb0

Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=78>