

# Zeta-potential measurement of MXene suspensions

## IV

Date: 2021-09-29

Tags: Zeta 18/08/2021Synth Nanoplexus 400 2021 LCC

Created by: James Bird

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Goal : Use Zetasizer to measure zeta potential of  $\text{Ti}_3\text{C}_2$  MXene nanoparticles in aqueous suspension

Procedure :

### Sample preparation

- Sample preparation is detailed in [\[Experiment\] Dynamic Light Scattering \(DLS\) for nanoparticle size-distribution acquisition V](#)
- Target concentration suspensions transferred to DTS1070 folded capillary cell with 1mL Luer syringe - cuvette exterior gently dried if necessary

### Zetasizer instrument operation

Standard Operating Procedure (SOP) settings (zeta measurement type) in Malvern Zetasizer NanoSeries Red Nano-ZS, using Zetasizer software program:

- Narrow band filter fitted
- Water dispersant ( $\eta = 0.8872$  cP,  $\text{RI} = 1.330$ , dielectric constant = 78.5)
- Smoluchowski Model for  $F(\eta a)$  calculation
- Use dispersant viscosity as sample viscosity
- Temperature = 25 °C with 120 s equilibration time
- DTS1070 folded capillary cell
- Automatic measurement duration
- Three measurements per sample
- Automatic attenuation and voltage selection
- Auto mode analysis model

Results :

Some useful measurement outputs for which measurements were successful (i.e. data quality criteria (DQC) are met) are given in the table below. Quality factors >>

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1 indicate very good signal. Green-highlighted zeta potentials indicate that the mean value suggests a stable suspension due to  $\approx -30$  mV, yellow-highlighted suggests suspensions could be stable within uncertainty, and red-highlighted indicates instability.

Fraction sedimented under rotation speed of $\omega$ / krpm	Concentration / wt %	$\zeta$ potential / mV	$\zeta$ deviation / mV	Electrophoretic mobility ( $U_E$ ) / $\mu\text{mcm/Vs}$	$U_E$ deviation / $\mu\text{mcm/Vs}$	Quality factor	QF StdDev
< 1.5	$1.3 \times 10^{-2}$	-11.8	4.77	-0.9247	0.3742	4.06	1.48
< 1.5	$1.6 \times 10^{-4}$	-37.5	7.43	-2.936	0.5825	6.09	2.78
$1.5 < \omega \leq 2$	$1.6 \times 10^{-2}$	-39.9	7.35	-3.131	0.5763	5.70	0.614
$1.5 < \omega \leq 2$	$2.6 \times 10^{-4}$	-30.8	8.40	-2.409	0.6584	1.68	0.849
$2 < \omega \leq 3$	$1.3 \times 10^{-4}$	-41.0	6.20	-3.212	0.4863	5.70	1.41
$3 < \omega \leq 4$	0.11	-14.7	5.03	-1.152	0.3944	2.34	nan
$4 < \omega \leq 5$	$5.7 \times 10^{-3}$	-19.7	4.59	-1.542	0.36	3.80	0.535
> 5	$1.5 \times 10^{-3}$	-25.0	5.19	-1.961	0.4066	4.2	1.63

Raw datafile is found in simultaneous experiment (see [\[Experiment\] Dynamic Light Scattering \(DLS\) for nanoparticle size-distribution acquisition V](#)) 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; apparent zeta-potential is plotted against the total counts for that zeta-potential interval. ZetaStats.xlsx contains data on all zeta-potential measurements, collected to date, in columnar format in an Excel spreadsheet. Vertical lines spanning the whole plot height are mean zeta-potential values, where the regions of matching colour spanning left and right of this value correspond to its standard deviation. Plotted zeta-potential distributions are only shown for the measurements which met DQC for a specific sample, hence the lack of errorbars for the  $3 < \omega/\text{krpm} \leq 4$  interval is due to the fact that only a single measurement of three met DQC.

Only the first three fractions, acquired from sedimentation of nanoparticles at the lowest centrifuge speeds, result in definitively stable suspensions. The average PDI of the three largest and three smallest particle fractions are  $0.528 \pm 0.201$  and  $0.508 \pm 0.118$ , respectively, and no correlation is found between PDI and zeta-potential. One feasible explanation may be that the smaller particles are more

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completely oxidised as a result of the extended period between synthesis and measurement. The smaller particles are likely to have a larger percentage  $\text{TiO}_2$  coverage due to the kinetics of oxidation.

### Conclusions:

These findings indicate that zeta-potential measurements may be more sensitive to suspension concentration than presumed previously, considering the range of concentrations used for measurement in this work. Oxidation and agglomeration due to sample storage issues may have contributed to suspension instability, with a dependence on particle size range.

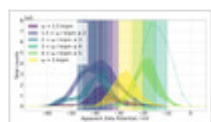
### Attached files

2021-09-29\_Zeta.csv

sha256: 374f32663a561c5f748f416be926dec6cc901034a03e6bdadc40dec1d9096c45

Zeta\_2021-09-29.png

sha256: 3fb923668956e35ef4491cd2a213605eef5e9575ed966991b6eb3abe859e3f26



ZetaStats.xlsx

sha256: 745ec7b19651f490765f879a05dc6fa5af3eef5c938279998c81599a5c5e72d1



Unique eLabID: 20230317-41573115ab304bdb9e1c54922e056115d712511e  
Link: <https://frankel-elab.manchester.ac.uk/experiments.php?mode=view&id=99>