Nanomaterial FAte and Speciation in the Environment (NanoFASE)

This project receives funding from the European Union's Horizon 2020 research and innovation programme under grant agreement N° 646002

NanoFASE Scientific Protocol

Characterisation of NMs by means of TEM

Written by:

Sophie M Briffa, Iseult Lynch and Eugenia Valsami-Jones[1]

[1] University of Birmingham

as part of the FP7 project NanoMILE

Edited for NanoFASE by Iseult Lynch, June 2019

Note: Template developed as part of Deliverable 5.4 under the European Commission's FP7 project NanoImpactNet (Grant Agreement 218539)

Contents

1. **Name(s) of scientific protocol:**

   Characterisation of NMs by means of TEM

2. **Scope and Domain:**

   Size and morphology characterisation of project NMs by means of TEM.

3. **Principle of the scientific protocol:**

   Suspended particles are deposited on a grid and sized by TEM imaging based on electrons reflected from the sample. By counting 200+ particles, an accurate measure of size and size distribution can be obtained.

4. **Description of scientific protocol:**

   Samples were prepared on copper grids coated with a thin film of holey carbon (Carbon film on 300 mesh Copper grids 3.05 mm) using the drop method. This involves placing a drop of sample on the surface of the grid which was held stationary and suspended by means of TEM tweezers. The drop was allowed to stand on the grid for a period of half an hour to an hour to allow the nanomaterials to adhere to the surface of the grid. Following this the grids were gently dipped repeatedly in ultrapure water to remove any loose material and excess salts from the grid. The grids were allowed to air-dry and kept covered to prevent any contamination from taking place. Imaging was carried out by means of a JEOL 1200EX TEM (accelerating voltage 80 kV). Image J was used to analyse the data. In order to obtain a size distribution (plotted as a histogram), at least 200 particles of each sample must be analysed.

5. **Environment:**

Benchtop

6. Biological and/or environmental models used:

N/A. Particles are typically deposited from solvent, but can be deposited from cell culture medium or various environmental matrices.

7. Chemicals and reagents used:

N/A – Particles are deposited on the grid from Deionised (DI) water, acetone or equivalent.

8. Nanoparticles used:

All project NPs.

TEM works extremely well for electron dense particles such as metals and metal oxides, but is also applicable to carbon-based materials and polymeric materials, althoguh some additional contrast (staining) may be required.

9. Apparatus and equipment used:

Carbon film on 300 mesh Copper grids 3.05 mm

JEOL 1200EX TEM

General plastics and glassware.

10. Health and Safety Precautions:

The protocol should follow Control Of Substances Hazardous to Health (COSHH) standards, and general health and safety precautions apply.

Instrument: View instrument manual for health and safety precautions regarding general, electrical warnings and manual handling precautions. Users should be fully trained before utilising TEM instrument.

11. Data analysis and Reporting the Data:

Image J (https://imagej.net/Welcome) is used to analyse the data. In order to obtain a size distribution plotted as a histogram, at least 200 particles of each sample have to be analysed using this software.

12. Abbreviations:

Control Of Substance Hazardous to Health – COSHH

Transmission Electron Microscopy – TEM

13. Limitations:

TEM works extremely well for electron dense particles such as metals and metal oxides, but is also applicable to carbon-based materials and polymeric materials, althoguh some additional contrast (staining) may be required.

14. Publications:

Cui X, Fryer B, Zhou D, Lodge RW, Khlobystov AN, Valsami-Jones E, Lynch I. Core–Shell NaHoF4@TiO2 NPs: A Labeling Method to Trace Engineered Nanomaterials of Ubiquitous Elements in the Environment. ACS applied materials & interfaces, 2019, 112119452-19461.

Briffa SM, Lynch I, Hapiuk D, Valsami-Jones E. Physical and chemical transformations of zirconium doped ceria nanoparticles in the presence of phosphate: Increasing realism in environmental fate and behaviour experiments. Environmental Pollution, 2019, Online published. Available online 7 June 2019.

15. References:

ISO 13322-1, Particle size analysis — Image analysis methods — Part 1: Static image analysis methods.

ISO 14488, Particulate materials — Sampling and sample splitting for the determination of particulate properties