UV/VIS analysis and optical band gap identification of NM suspension

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Version
1.0 English

Content

1 Scope

This Standard Operating Procedure (SOP) describes the UV/VIS analysis and band gap calculation for nanomaterial (NM) suspensions within the nanOxiMet project. The analysis provides the absorption spectra and the maximum absorption value of the different NM in suspension. This information is helpful for the calculation of the NM suspension concentration as well as for the band gap identification.
Note: The SOP should be taken as guideline and informative advice for UV/VIS analysis and has to be adapted specifically for other UV/VIS instruments.

2 Basics

The aim of this Standard Operating Procedure is the description of the UV/VIS analysis procedure as well as the band gap identification via UV/VIS analysis. Within the nanOxiMet project for each NM at least three different concentrations of the same NM were analysed three times (n = 3) and the absorption spectra recorded. For metal materials via the concentration row (spectra comparison) additional qualitative information about particle/agglomerate size changes (agglomeration) are detectable (plasmon resonance).

3 Materials & Instruments

3.1 Materials

The following materials and chemicals are required:
- HPLC Grade water
- Nanomaterial suspension
- Cuvette (Disposable)
- Pipette

3.2 Instruments

The following instruments are required:
- UV/VIS instrument
- Liquid handling apparatus / Pipette

Note: For the analysis a Specord200 from Analytik Jena was used to record the UV/VIS spectra. The usage and maintenance of the instruments will not be described in detail in this SOP. Please refer to the manual.

4 Experimental procedure

4.1 Suspension preparation

The suspension preparation is described in the SOP –_Dispersion protocol_sonication_cup horn_1.1

4.2 Measurement and Instrument settings

For analysis, a minimum of 3 mL of the prior prepared suspension has to be transferred (using a pipette) into a 4.5 mL disposable plastic cuvette, placed in the analysis device and subsequently analysed for absorption maximum / absorption spectra.

4.2.1 Instrument parameters/settings (Analytik Jena Specord 200)

Initialize instrument: UV lamp on
Settings:
- Cycle mode: Automatic
  → Numbers: 3
- Display: Absorbance
- Correction: Reference

Device:
- Interface: USB
- Slit: 1 nm
- Lamp change: 320°
  → HL automatically

Mode:
- Scan mode
  - Range: 190 nm – 1100 nm
  - Delta Lambda: 1 nm
  - Speed: 50 m/s
  - Integration time 0.02 s

Accessories: none

4.2.2 Measurement procedure (Analytik Jena Specord 200)

- The instrument and the lamps have to be switched on at least 15 min before the first measurements (warm up)
- After 15 min the UV/VIS software “Winaspect” has to be started and the parameters described above have to be recorded into the software
- Initialize the instrument (UV lamp on)
- Place a blank/reference sample in the rear and front cuvette holder and start a reference measurement, for the determination of the background signals

- Replace the blank sample in the front cuvette holder with a sample and start the measurement
- Save the data

5 Data Evaluation / Reporting

For data evaluation write down the maxima of the absorption wavelength and give the absorption spectra as graphs (x-axis wavelength in nm, y-axis absorption 0-1). The graph should express the mean absorption spectra of the three different concentrations measured. For the determination of the suspension concentration, perform a linear correlation between the absorption intensity of one specific wavelength (y-axis) vs. NM suspension concentration (x-axis).

5.1 Optical band gap identification

The band gap is defined as the energy difference between the top of the valence band and the bottom of the conduction band. Electrons are generally able to jump from one band to another as long as a specific minimum amount of energy for the transition, the band gap energy, is provided. The optical band gap was calculated using absorption edge values (λ edge, in nm) from absorption spectra (Figure 1) and using following eq.:

Band Gap Energy (E) = h*c/λ

h = Planks constant = 6.626 x 10-34 Joules sec
c = Speed of light = 3.0 x 108 meter/sec
λ = Cut off wavelength/absorption edge in nm x 10^{-9} meters
1eV = 1.6 X 10-19 Joules (conversion factor)

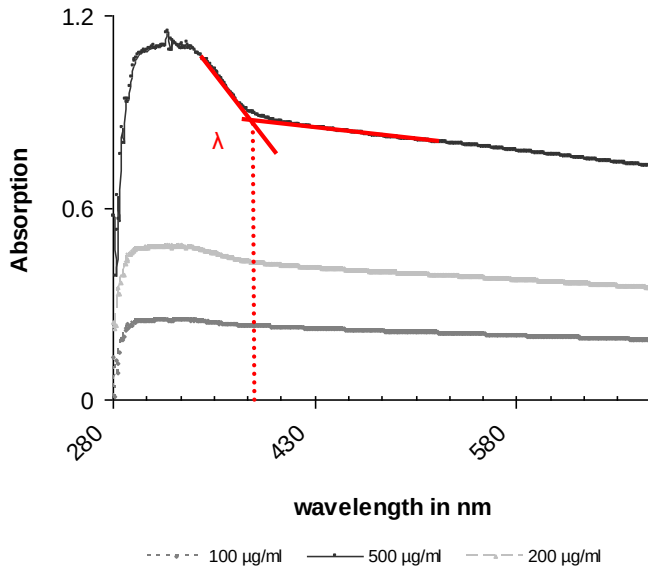

Figure 1 Optical absorption edge determination for NM101 at three different concentrations

6 Quality control

The results are presented as absorption maxima (wavelength in µm) and/or absorption spectra.

7 Safety precautions

In general when handling NMs, protective clothing and suitable masks and gloves have to be worn at any time and the working area, as well as the used materials and instruments, have to be labelled. Please follow the safety information of the instrument manufacturer and material provider.

8 Waste disposal

Please follow the disposal advice of the material provider, if available.

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Responsible for the implementation of this SOP: Measurement personal

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