Methods for determining the sizes of nanoparticles and loading dyes onto nanoparticles

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Introduction

- Various techniques for detecting, measuring and characterising
- No method is the “best”
- Type of sample, the information required, time constraints and the cost of the analysis.
- Presence of nanoparticles, quantity, size distribution or the surface area
- Chemical content, reactions on the or for the interactions with other chemical species present.
- A divide between techniques that give information amount of nanoparticulate material and those that can look at the individual nanoparticle
- Combination of techniques for one sample.
Introduction

- Some techniques require sample to be: aerosol, suspension or liquid sample, solid. There may be a sample protocol to be followed
- In situ measurements, treatment of the sample before analysis (decompose or react)
- Various amount of sample required (restrict choice of technique)
Transmission Electron Microscopy (TEM)

- Measures: particle size and characterization
- Sample preparation: < 1 μg thin film and stable under an electron beam and a high vacuum
- Sample preparation difficult (thin sample on a support grid)
- Time consuming and costly
- Sensitivity: down to 1 nm
TEM

- Principle: electron beam to interact with a sample to form an image on a photographic plate or specialist camera.
**Scanning Electron Microscopy (SEM)**

- Measures particle size and characterization
- Sample: conductive or sputter coated
- Easier to prepare than TEM
- Samples mounted on a stub of metal with adhesive, coated with 40 - 60 nm of metal such as Gold/Palladium
- Sensitivity: down to 1 nm
- **Principle**: uses a high energy electron beam but the beam is scanned over the surface and the back scattering of the electrons is looked at.
- The sample under a vacuum
• Sputtering device

• SEM
SEM image (nanoparticles)
Atomic Force Microscopy (AFM)

- Particle size and characterization
- Samples must adhere to a substrate, rigid and dispersed on the substrate
- Air or liquid samples
- Sensitivity: 1 nm - 8 μm
- Form of Scanning Probe Microscopy (SPM): requires less time and cost than SEM and TEM.
AFM

- **Principle:** uses a mechanical probe to feel the surface of a sample
- A cantilever with a nanoscale probe is moved over the surface of a sample and the forces between the probe tip and the sample measured from the deflection of the cantilever
- The deflection moves a laser spot that reflects into an arrangement of photodiodes
- 3D visualization
Photon Correlation Spectroscopy (PCS)

- Measures: average particle size and size distribution
- Sample preparation: very dilute suspension (otherwise, unclear scattering of light)
- Sensitivity: 1 nm - 10 μm
- Sensitive to impurities, the viscosity of the sample must be known
**PSC**

- **Principle:** measures the scattering pattern produced when light is shone through a sample

- It combines this with calculations of the diffusion caused by Brownian Motion in the sample in relationship described in the Stokes-Einstein equation

- This gives the radius of a particle, therefore an estimation of the average particle size and distribution of particles through the sample
Other techniques

- X-Ray Diffraction (XRD)
- Dynamic light scattering (DLS)
- Nanoparticle Surface Area Monitor (NSAM)
- Condensation Particle Counter (CPC)
- Differential Mobility Analyzer
- Scanning Mobility Particle Sizer (SMPS)
  e.t.c.
Loading nanoparticles onto dyes
UV/vis spectroscopy

- Weigh a known amount of the conjugate.
  (Very small amount)
- Disperse in a known volume solvent, agitate with sonicator
- UV spectrum of dispersed mixture
- Noting the absorbance of the band
UV/vis spectroscopy

• \( A = \varepsilon c l \)
• \( A = \) absorbance (known)
• \( E = \) extinction coefficient of dye
• \( C = \) concentration (unknown)
• \( L = \) path length of the cuvette (cm)
• Calculate concentration, then moles of dye
• E.g. mole/volume
• The volume of solvent
• Calculate the mass of dye
Once you get the mass of dye
The mass obtained is from the known amount of conjugate e.g.
0.02\textmu g in 0.5\text{mg} of conjugate
1\text{mg} of conjugate will then give us \(1 \times 0.02/0.5\)
That would give us the mass loading of the Pc per mg of conjugate
Thermogravimetric analysis (TGA)

- method of **thermal analysis** in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time

- Measure the TGA profile of the NPs alone

- Measure the TGA profile of the conjugates having the NPs with other molecules

- Compare % loss of NPs alone with conjugates

- Difference will account for loading efficiency
Fluorescence Spectroscopy

- Measure the fluorescence of the photosensitizers before conjugation and after conjugation.
- Compare the fluorescence intensity of the photosensitizers before conjugation and after conjugation (supernatant-same solvent must be used).
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