Bachelor Proef

Semester II:

Gold Nanoparticles:

* synthesis
* characterisation
* radiosensitization of DNA with GNP

Namelore: 2030 Lies: 50 Week: 15/02 - 21/02

Summary: first acquaintance + literature study

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Goal:

1) Make/synthesize GNP
2) characterize them with the different techniques
3) check/analyze the positive radiosensitization effect of GNP on DNA

⇝ very analogue to the masterthesis of Lennaert Wouters
→ we preform an extra test
→ extra data.

Hannelore + Lies: 5u30 22/02/'16

Summary: first acquiantance with the lab + synthesis GNP

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* guided tour around the lab
* Synthesis GNP → protocol from Jennaert Wouters.
  1. A solution of 100 mL 0.01% $HAuCl_4$
  2. boiling Temperature & stirring
  3. Tree aprions:

15nm ← 2.5 ml
30nm ← 1.24 ml } 1% $Na_3C_6H_5O_7$
45nm ← 0.8 ml

  4. boiling + stirring for 35 min.
  5. cooling down + protecting from light for 60 min
  6. Storing; 4°C + no light

Calculation:

% → $\frac{m}{m}$ → we took ✓ ✓

↳ NO GNP since wrong calculation (To little $HAuCl_4$)

* first acquaintance with characterization techniques
  ↳ ~ overview
* try out with DLS (Dynamic light scattering)

Overview thesis

1. Make GNP → protocol Lennaert + functionalize (PEG) Wouters
2. Characterize GNP
   - a. UV-Vis → core diameter
   - b. DLS → hydrodynamic radius
   - c. ξ-pot. → stability colloid
   - d. TEM → image + more exact core diameter
   - e. TGA → weightpercentage difference + PEG capping density Pc

   ↳ which one shall we use? NH_2 ← neutral / charged → OCH_3
3. Mix with DNA and irradiate
4. gelelectrophoresis to study the radiosensitization effect.

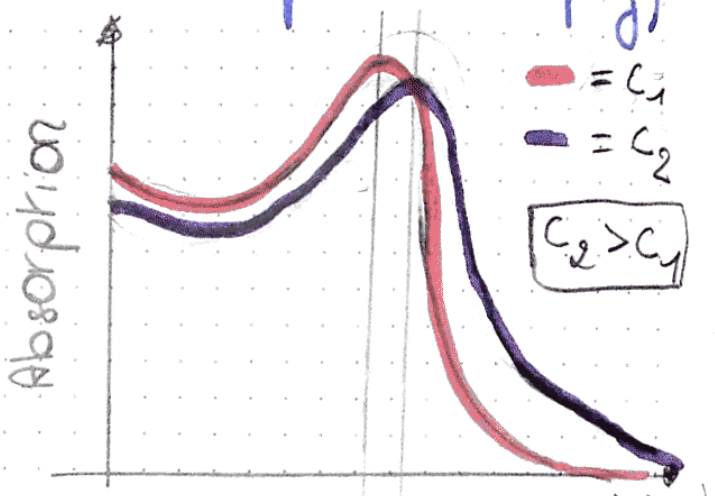
Characterization techniques

① UV-Vis (Ultraviolet-visible spectroscopy)

⇒ core diameter

* UV-radiation trough sample
* surface plasmons
  → resonant freq.
  ↳ depends on the size GNP

bigger → lower forces → lower freq → bigger λ

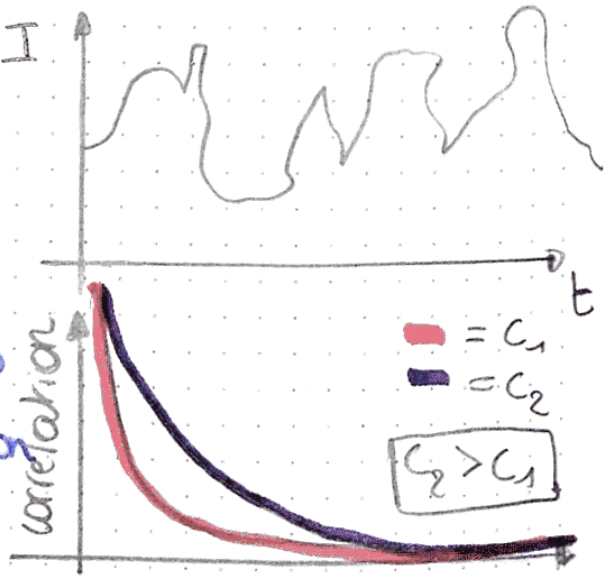


② DLS (dynamic light scattering)

⇒ hydrodynamic radius

* infrared radiation trough sample
* Rayleigh scattering
* measure I
  ↳ variance due to brownian motion GNP
* Determine auto-correlation

$$g^2(q;\tau) = \frac{\langle I(t) I(t+\tau) \rangle}{\langle I(t) \rangle^2}$$



* different methods to analyze the data:
  a) Cumulant method/Algorithm
     → monodisperse solution
  b) Padé-Laplace Method/Algorithm
     → polydisperse solution (PDI ≥ 0.1)
     polydispersity index

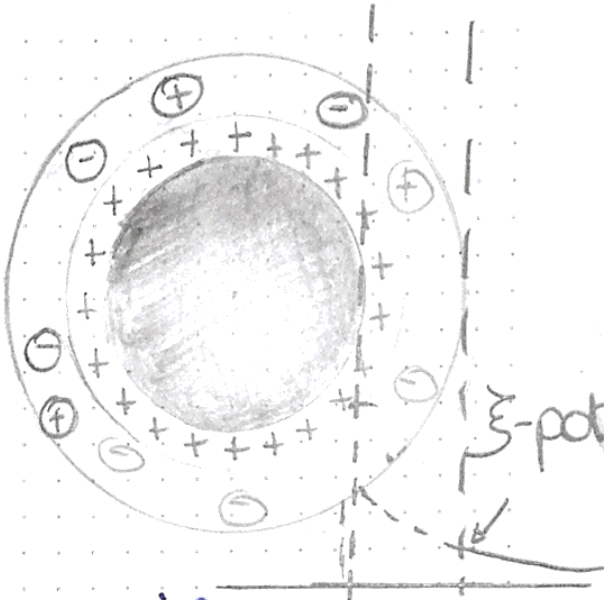
Background:

Brownian motion + Stokes-Einstein

$$P(r,t) = (4\pi D)^{-3/2} \exp(-r^2/4Dt) \qquad D = k_b T / 6\pi\eta R_h$$

③ ξ-potential
⇒ stability of colloid

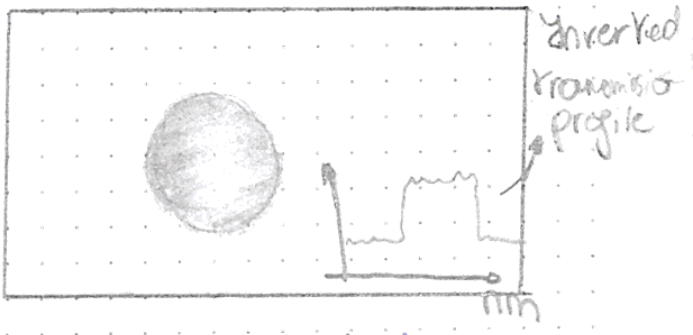
* electrophoresis
  → 2 electrodes
* measure constant v
  → laser doplen
∴ $E \cdot q = \alpha \cdot v \Rightarrow v = \mu_e \cdot E$
with $\mu_e = q/\alpha$
* theoretical link ξ-pot:
$\xi = \alpha \eta \cdot \mu_e / 3\varepsilon$ (Smoluchowski approximation)
η → viscosity medium; ε → dielectric constant medium



⇒ if ξ ≥ 30mV
stable colloid

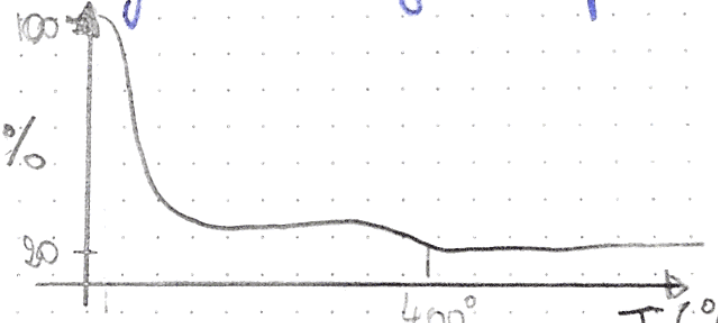
④ TEM (Transmission electron microscope)
⇒ size/radius particles
↳ which one?

* heating cathode (tungsten filament)
* e^- more to anode (with hole → beam e^-)
* beam through sample
* transmitted beam → fluorescent plate
→ electronic picture



! Resolution depends on wavelength

⑤ TGA (Termogravimetric analysis)
⇒ weight percentage + PEG capping density ρ_c
* Weight monitoring as a function of Temp.
⇒ mass ratio $\frac{\text{gold}}{\text{PEG}}$
→ M_{PEG} + ρ_{gold} → num. % ligands/particle
→ + surface area: ρ_c



Mushroom vs. Brush ⇒ $s = 2 \cdot \sqrt{\frac{1}{\pi \rho_c}}$ | $s \geq R \approx a N^{\nu}$ Flory radius

Mannelore: 6030 Lies: 29/01/2016

Summary: Synthesis GNP + Characterization; UV-vis, DLS, ζ-potential

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Synthesis GNP:

1. Solution of 0,00998 g $HAuCl_4$ in 99,997 ml H_2O
2. Solution put at boiling T + stirred
3. a) solution 0,100234 g $Na_3C_6H_5O_7$ in 9,943 ml H_2O (10 ml 1% sol)
   b) add 2.5 1.24 0.8 ml
      15 30 45 ; nm
4. Solution stirred for 15 min
5. Cooled down & protected from light for 60 min
6. Stored at 4°C & protected from light

REMARKS

* We started from a solution of $HAuCl_4$
  → 1 g $HAuCl_4$ in 25 ml
  ↝ took the right volume to get 0.00998 g $HAuCl_4$ and filled up till 100 ml
* boiling T: bubbles
* 2.5 ml & 1.24 ml were injected in the solution with an doktor syringe (after careful measurement with an pippet)
* Important (to reduce errors); better twice the same amount pipetting
* Protection from light: silverpaper

Brand of thermometer: eversave

Characterization techniques:

DLS login pc: 31415
program: Malvelore (Expert)
31415

SNR: 1.05 % (noise)
→ hydrodynamic radius measurement
≈ 45nm

! you can improve the measurements by forcing the high accuracy

Besides Cumulants, Pade-Laplace you also have SBL
↳ sort of combination

! Only Intensity is interesting of both.

Device settings:

Time Interval: 1
Numb. channels: 300 (→ 600 with 45nm/0.8ml

UV-Vis → check for no aggregation.

Wavelength accuracy:
≤ ± 1.5 nm λ > 315 nm
≤ ± 0.8 nm λ < 315 nm
(for 25 flashes)

! Make sure no excel file is open
→ he will open a new one himself

Mannelore: 6u30 Lier: 29/02/2016

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