Procedure for synthesis of nanomaterials

Friday 20.03.2026

- Synthesis of TiO_2 nanocomposite by sol-gel

In a 50 mL plastic vial, weight 8.5 g of $Ti(OBu)_4$ polymer and mix with 15 mL of absolute ethanol (Solution 1).

In a 100 mL 1-neck flask, mix 7.5 mL of H_2O, 17.5mL of absolute ethanol and the required amount of conc. HNO_3 (Solution 2). Mix the system with a magnetic bar.

Load a 20 mL syringe with Solution 1 and add it dropwise into Solution 2 while stirring.

Wash the vial with 5 mL of ethanol and add it into Solution 1.

After mixing for 10 minutes, leave the system stirring gently until the gel is formed.

DO NOT COVER THE FLASK

If possible, record the time required to form the gel.

- Synthesis of Ti-MCM-41 synthesis

To prepare the micelles:

Weight CTAB into a 100 mL 1-neck flask.

Add H_2O and a magnetic bar. Mount the flask into a dry-syn and heat the system to 40°C.

When CTAB is dissolved, ass the required amount fo the Swelling Agent (if required).

Leave stirring for the weekend, with the flask closed with a cup.

Monday 23.03.2026

- Synthesis of Ti-MCM-41 synthesis

Into a 15 mL plastic vial, weight TBOT and add 2-PrOH.

Under stirring, add in the flask TEOS and 2.5 mL of NaOH 2.0 M.

Wait 10 minutes and add dropwise with solution of TBOT in 2-PrOH.

After stirring for 10 minutes, transfer the gel into the Teflon liners dividing the system into 2 equal parts. Close the vessels tightly.

The gel will be aged under microwave irradiation (120°C for 40 minutes).

Prepare solution for washing the precipitate mixing 100 mL of water with 50 mL of ethanol.

After cooling, transfer the product into 50 mL plastic vials and recover the powder by centrifugation.

After wasting the first mother liquor, add 30 mL of washing solution, shake the powder and leave overnight for the further washing.

- Synthesis of TiO_2 nanocomposite by sol-gel

After aging the gel for at least 24h, put the flask in oven at 120°C overnight to dry the gel.

Tuesday 24.03.2026

- Synthesis of CdS QDs

Mount a 250 mL 1-neck flask on a dry-syn. Add a magnetic bar.

Weight NAC, transfer into the flask and add 55 mL of water. Mix until NAC is dissolved.

Add the solution of Cd^{2+}, mix for 2 minutes.

Adjust pH of solution by adding NaOH 2.0M until pH = 8.0 – 9.0 is reached (control with indicator paper).

Control temperature at the desired value with ice/water bath or dry-syn and stir at that temperature for 30 min.

Add S^{2-} solution and stir for 30 minutes.

Distribute the product into 3 50 mL plastic vials, save one vial for characterization in solution and add 20 mL of acetone in the other 2 vials. Recover the QDs by centrifugation (5000 rpm for 10 min).

Wash each precipitate with 10 mL of acetone, recover the QDs by centrifugation (5000 rpm for 10 min) and leave it drying to open air.

Record UV-vis and fluorescence spectra for CdS QDs in solution and calculate the size of QDs from the maximum of emission spectra.

- Synthesis of TiO_2 nanocomposite by sol-gel

Remove the xerogel from the oven, let it cool to room temperature and grind the powder for calcination.

- Synthesis of Ti-MCM-41 synthesis

Wash the precipitate 3 times using aliquots of 30 mL of washing solution, shake the powder to suspend the precipitate and centrifuge (5000 rpm for 10 min).

Repeat a final washing using 30 mL of pure ethanol, recover the powder by centrifugation.

Dry the powders overnight at 70°C.

Grind the powder and prepare the sample for calcination.