Óbuda University

Doctoral School on Materials Sciences and Technologies

2nd semester progress report: Autumn 2017/18

"Application of sensors in the detection of heavy metals ions in the environment"

By: Larbi Eddaif

Supervisor: Dr. Shaban Abdul

Place of work:
MTA TTK AKI
Hungarian Scientific Academy of Science
Research Centre for Natural Sciences
Institute of Materials and Environmental Chemistry

January 26 2018

Outlines

Introduction,

Topic of research,

Results of the actual semester,

Future planned tasks,

Conclusions.

Topic of research

"The application of sensors in the detection of heavy metals ions in the environment"

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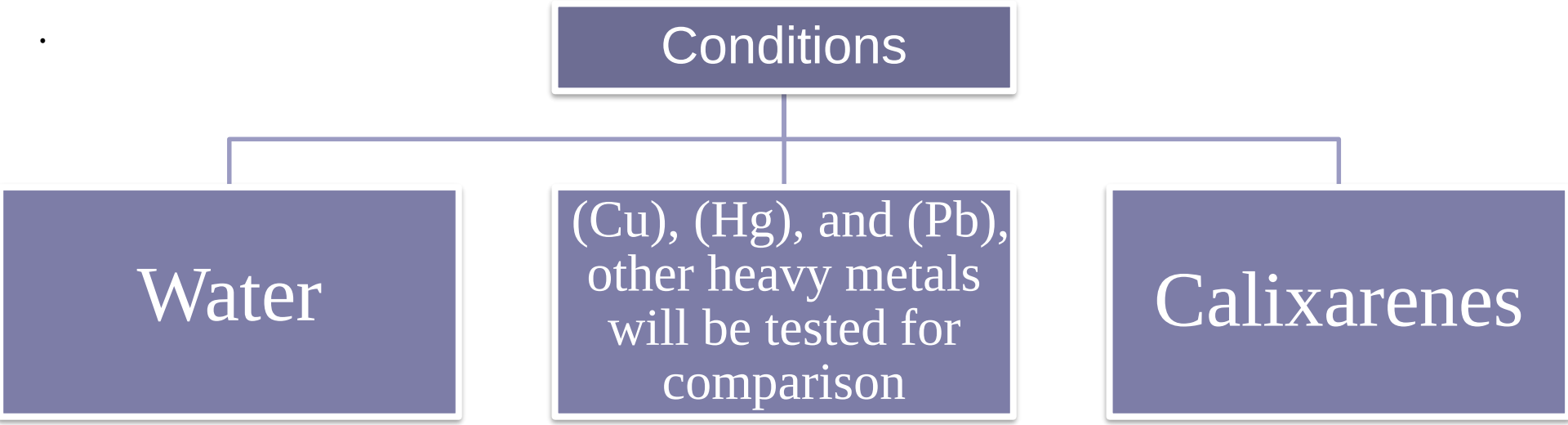


To improve the theoretical and experimental background, more than 70 references have been studied so far.

Results of the actual semester

- **Compelted courses**

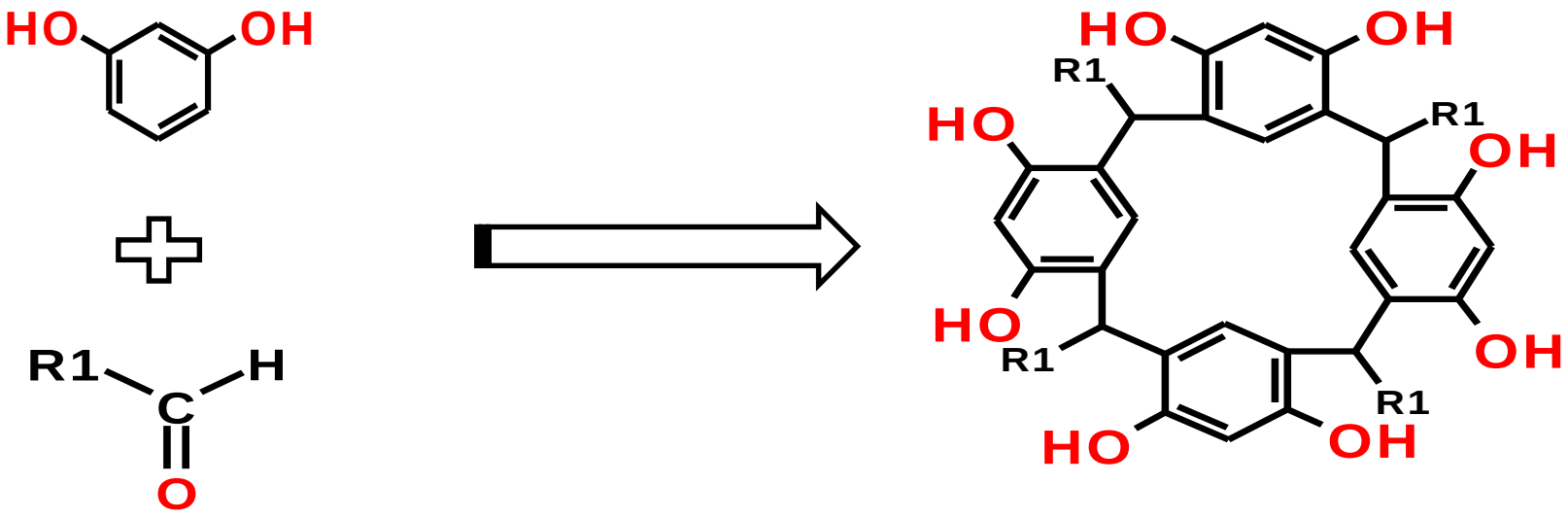
Subject Title & Neptun codes	Lecturers	No. of credits
Chemistry and Physics of Polymers OATPKFC1ND	Dr. Pekker Sándor	6
Selected chapters on the methods of analysis of materials II OATVFAM2ND	✓ Adsorption- Dr. Klébert Szilvia ✓ XPS- Dr. Miklos MOHAI ✓ XRF - Dr. Zoltan MAY	6
Research project	Dr. Shaban Abdul	10
Research Report	Dr. Shaban Abdul	6
Total credits		28

Results of the actual semester

- Research Progress

1. Polymer synthesis

The aromatic components are derived from phenol, resorcinol, or pyrogallol. For phenol, the aldehyde most often used is simple formaldehyde, while larger aldehydes, like acetaldehyde, are usually required in condensation reactions with resorcinol and pyrogallol.



Results of the actual semester

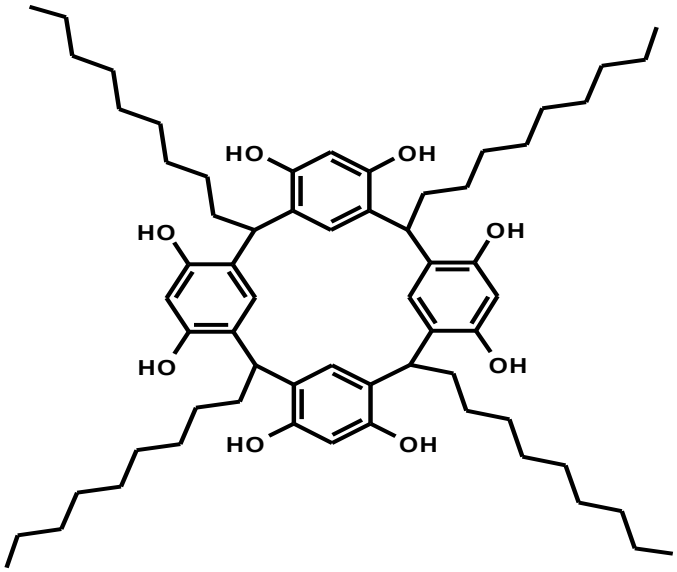
- Research Progress

1. Polymer synthesis

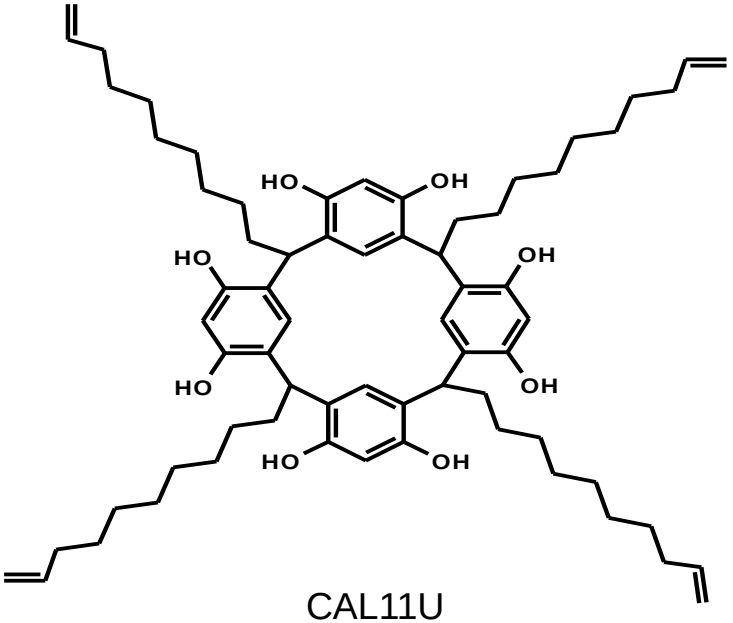
CAL9U – calixresorcinaren synthesized form trans-2,cis-6-Nonadienal.

CAL10 – calixresorcinaren.

CAL11U – calixresorcinaren synthesized form undecylenic aldehyde.



CAL10

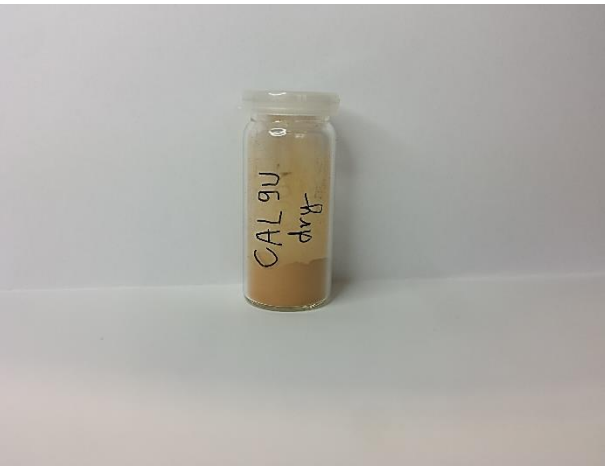


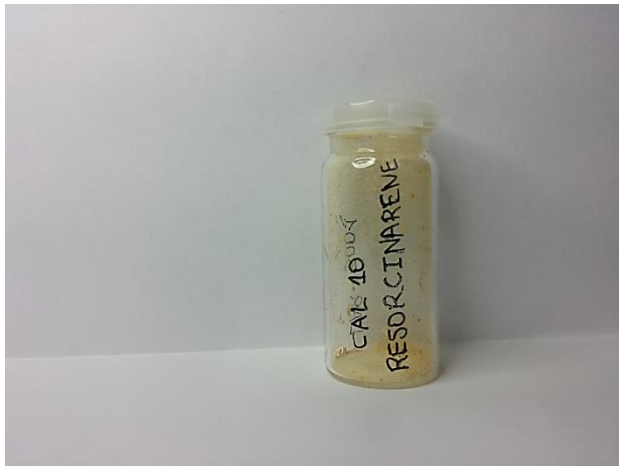
CAL11U

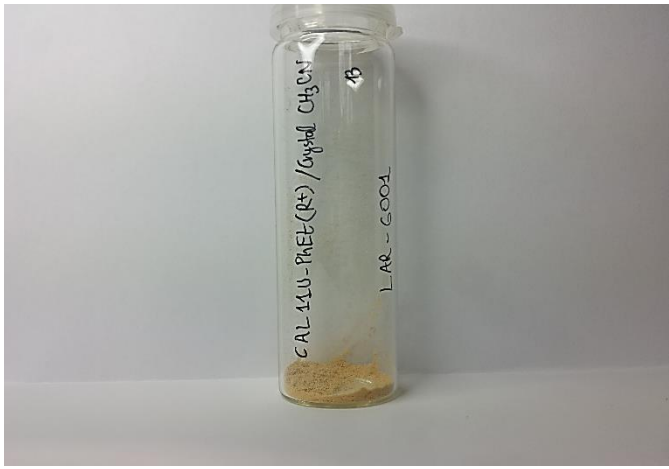
Results of the actual semester

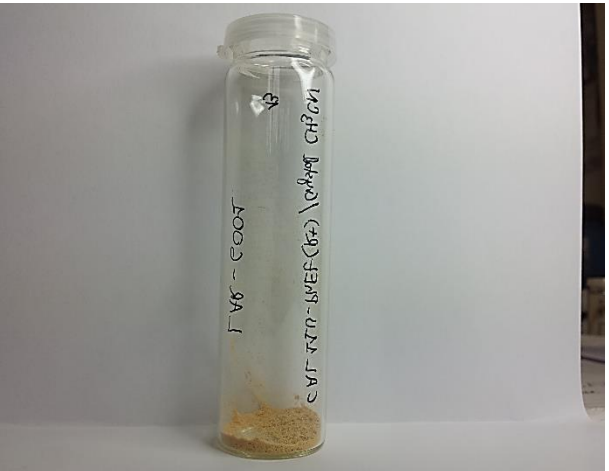
- Research Progress

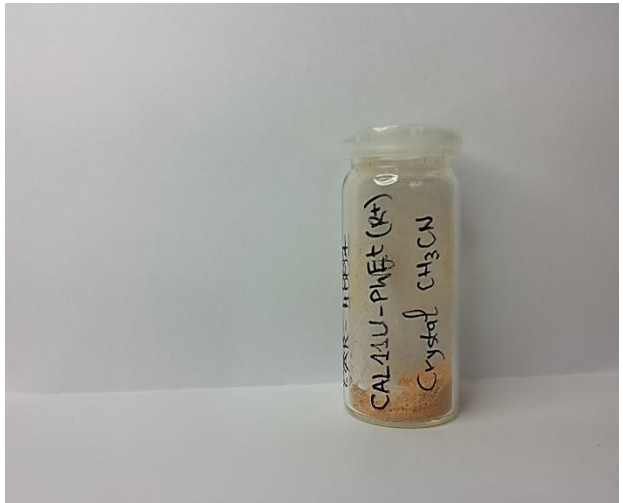
1. Polymer synthesis

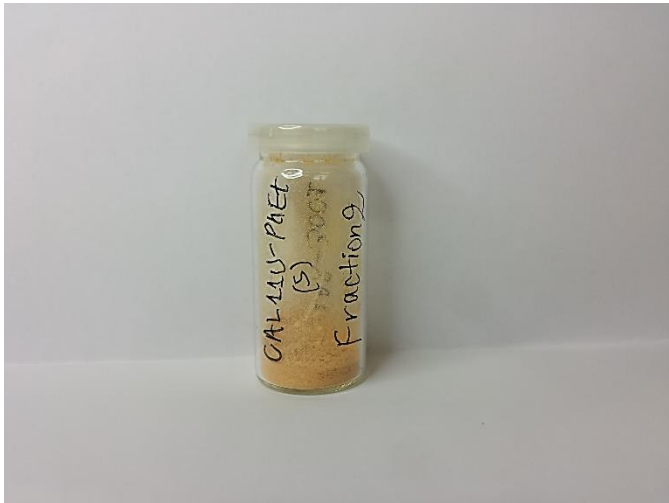












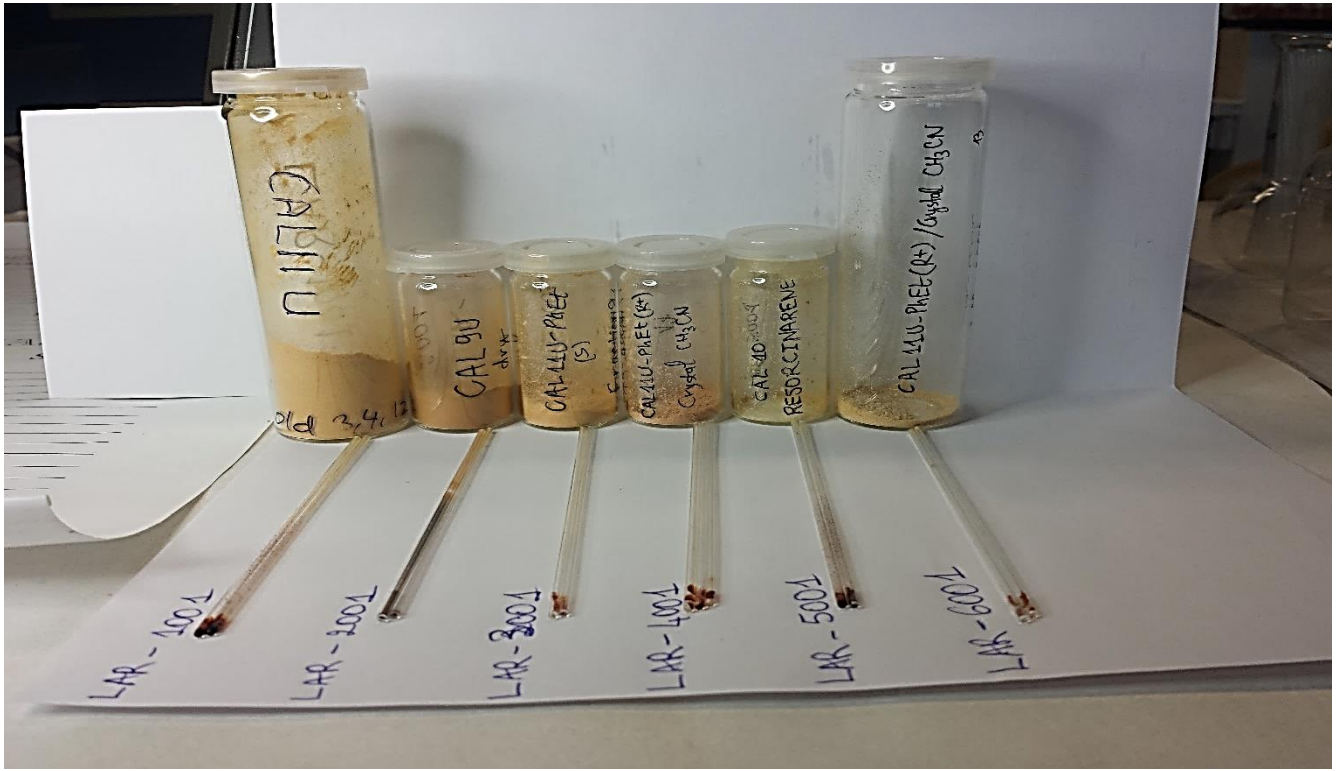
Results of the actual semester

- Research Progress

2. Polymer characterization

a. Boiling point

The **boiling point** of a substance is the temperature at which the vapor pressure of the liquid equals the pressure surrounding the liquid and the liquid changes into a vapor.



Results of the actual semester

- Research Progress

2. Polymer characterization

a. Boiling point

Code	Polymer name (formula)	Boiling Points ^{0}C	Weight (g) after grinding
LAR 2001	CAL9U	314: black color without melting	1.0644
LAR 5001	CAL10 Resorscinarene	284.6	0.0300
LAR 1001	CAL11U	277	2.9434
LAR 6001	CAL11U-PhEt (R+)/Crystal CH_2CN	70	0.2528
LAR 4001	CAL11U-PhEt (R+)/Crystal CH_3CN	75.6	0.1613
LAR 3001	CAL11U-PhEt (S)/Fraction 2	70	0.5723

Results of the actual semester

- Research Progress

2. Polymer characterization

b. Langmuir Blodgett isotherms and B.A.M. investigations

A **Langmuir–Blodgett** film contains one or more monolayers of an organic material, deposited from the surface of a liquid onto a solid by immersing the solid substrate into (or from) the liquid. A monolayer is adsorbed homogeneously with each immersion or emersion step, thus films with very accurate thickness can be formed.

A **Brewster angle microscope** (**BAM**) is a microscope for studying thin films on liquid surfaces, most typically Langmuir films.

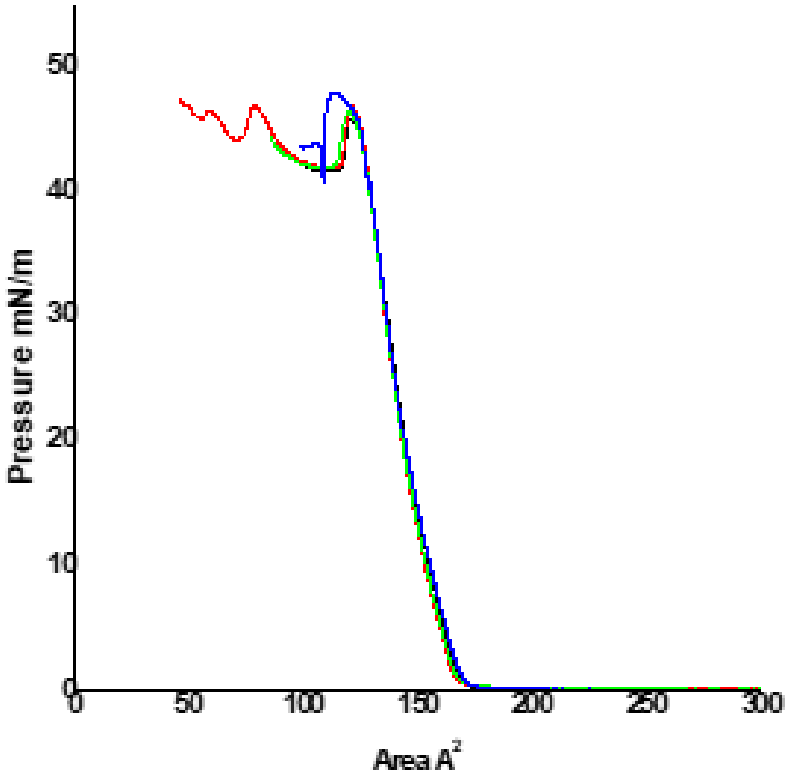
Results of the actual semester

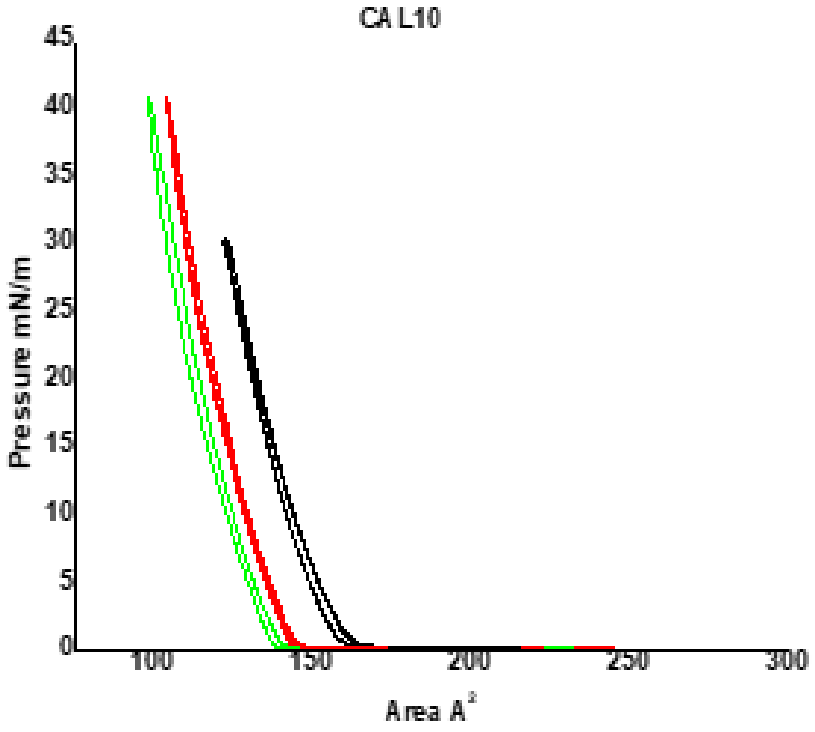
- Research Progress

2. Polymer characterization

b. Langmuir Blodgett isotherms and B.A.M. investigations

Cal 10





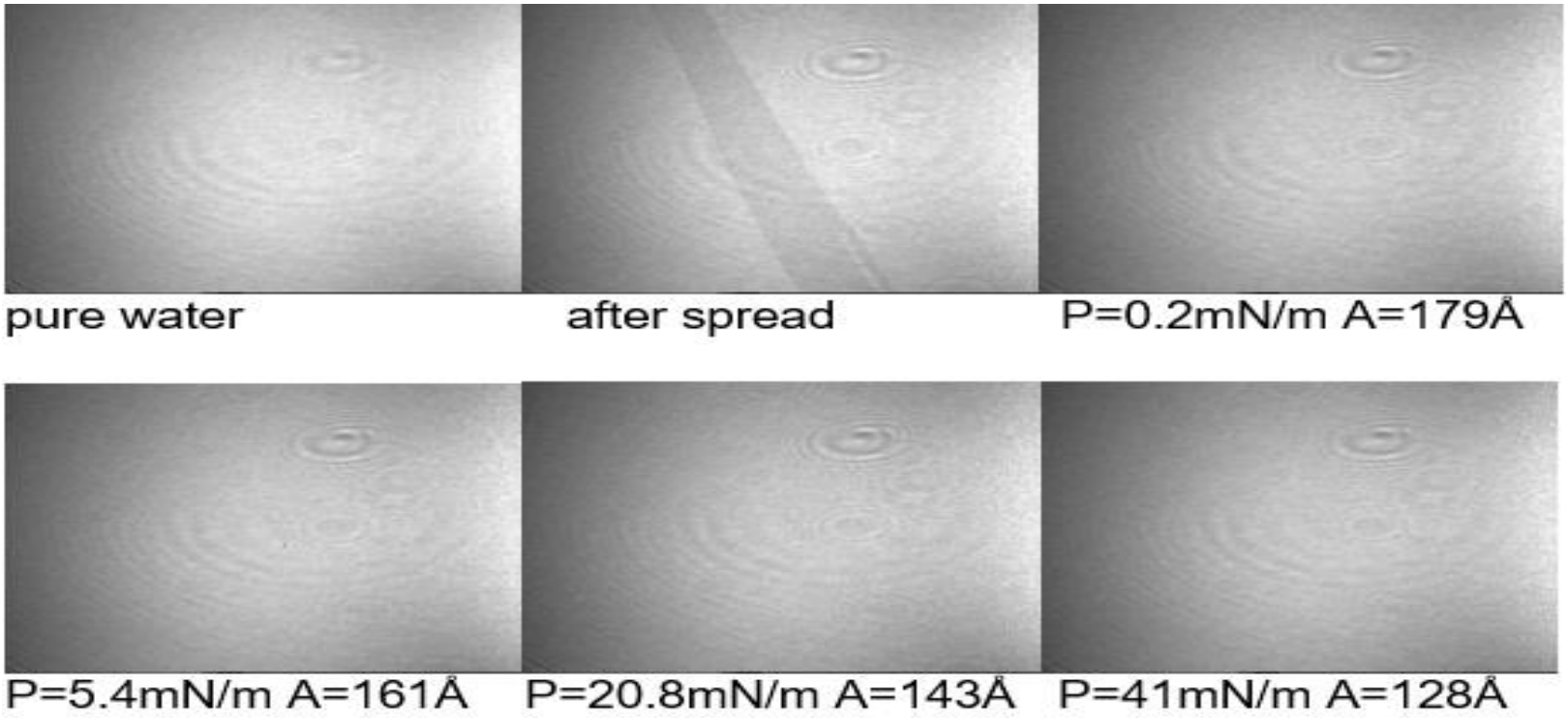
Results of the actual semester

- Research Progress

2. Polymer characterization

b. Langmuir Blodgett isotherms and B.A.M. investigations

Cal 10



Results of the actual semester

- Research Progress

2. Polymer characterization

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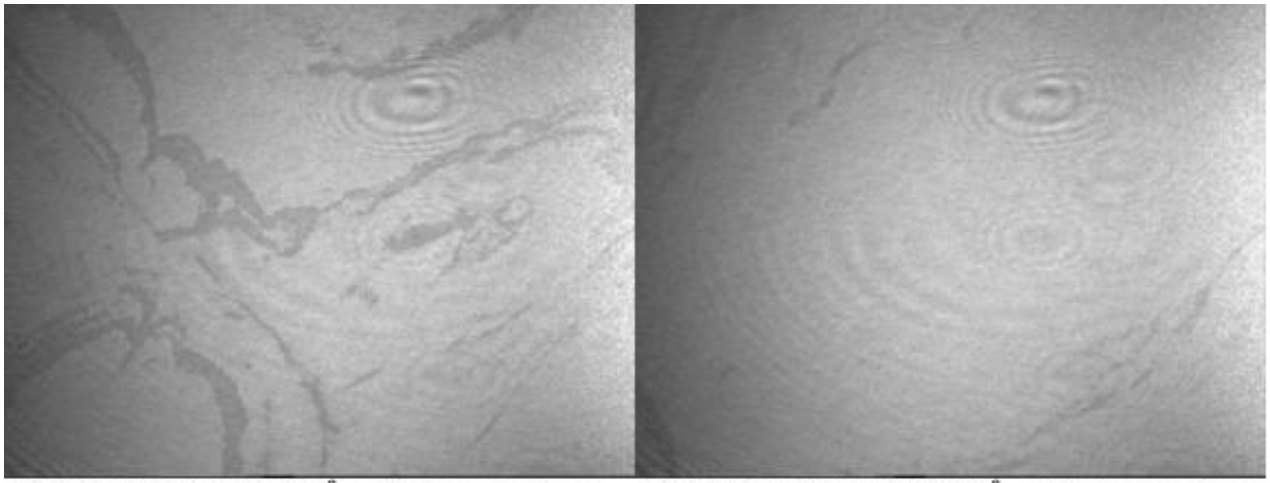
Cal 10



P=42mN/m A=98Å after collapse P=43mN/m A=108Å after collapse

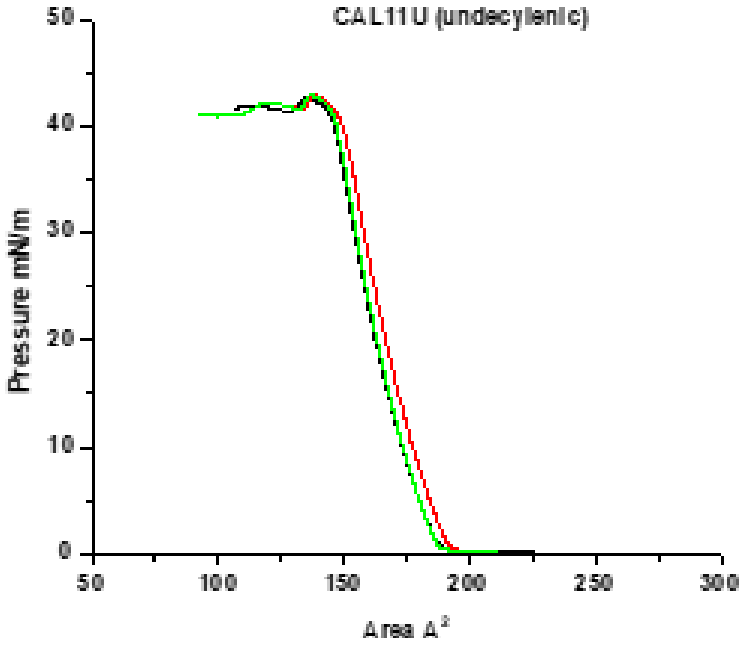
Results of the actual semester

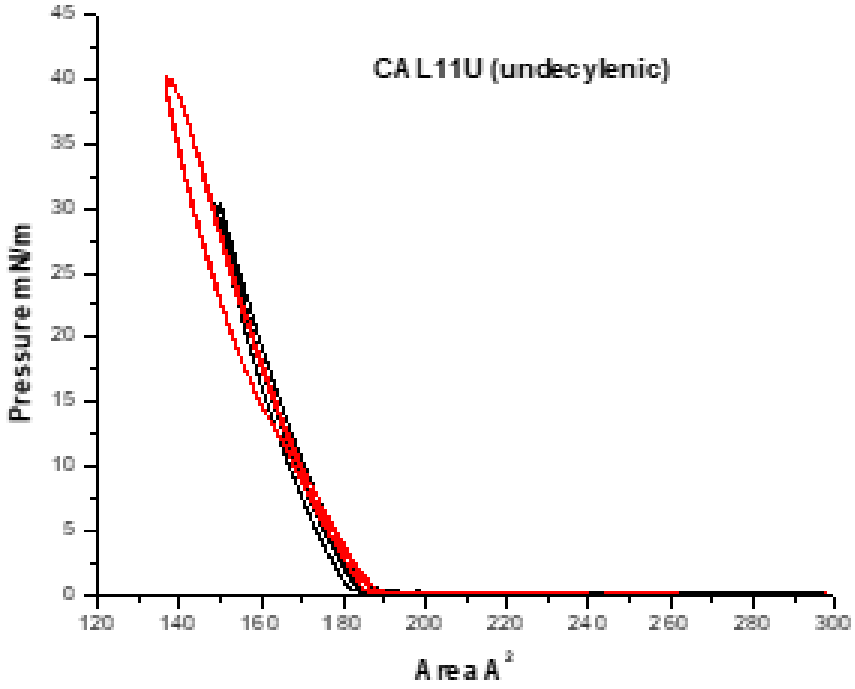
- Research Progress

2. Polymer characterization

b. Langmuir Blodgett isotherms and B.A.M. investigations

Cal 11U





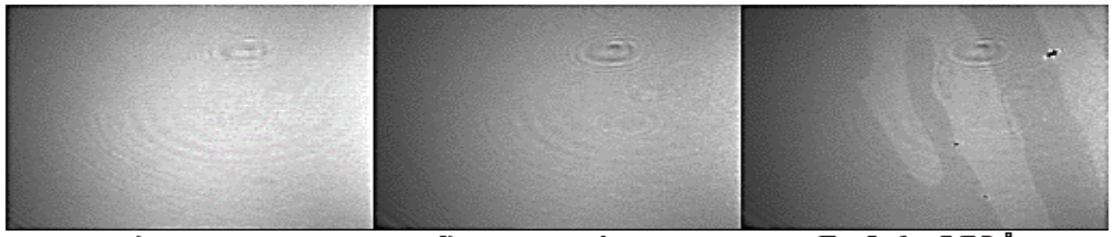
Results of the actual semester

- Research Progress

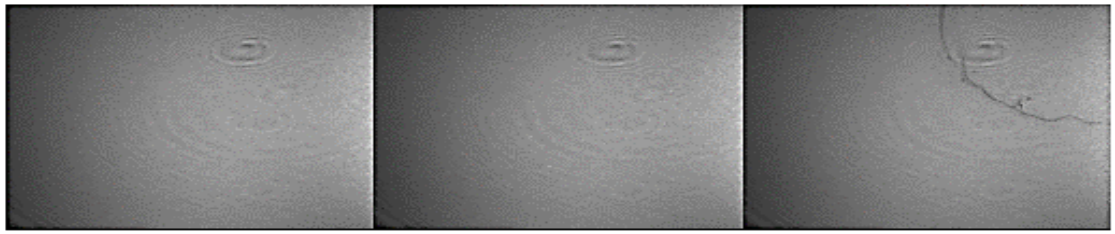
2. Polymer characterization

b. Langmuir Blodgett isotherms and B.A.M. investigations

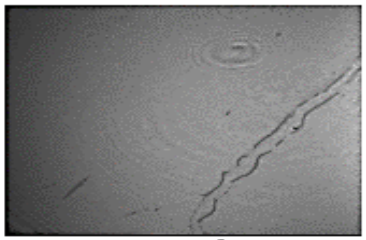
Cal 11U



pure water | after spread | P=0 A=252Å



P=10mN/m A=166Å | P=30mN/m A=146Å | P=42mN/m A=135Å col.



P=42mN/m A=108Å after collapse

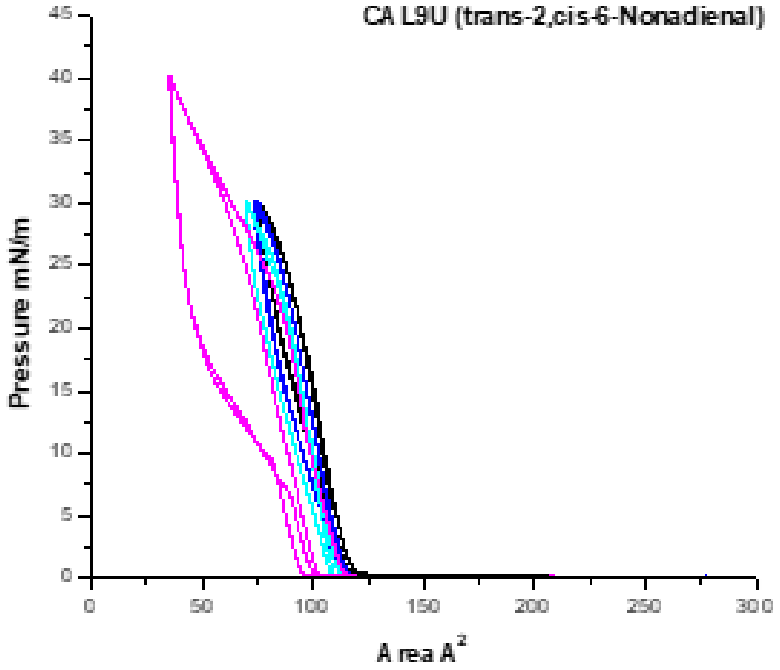
Results of the actual semester

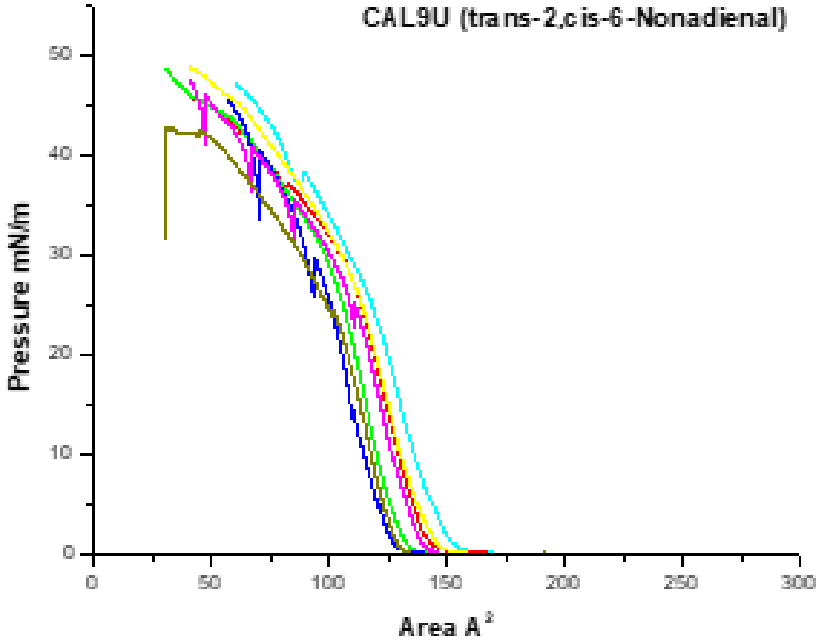
- Research Progress

2. Polymer characterization

b. Langmuir Blodgett isotherms and B.A.M. investigations

Cal 9U





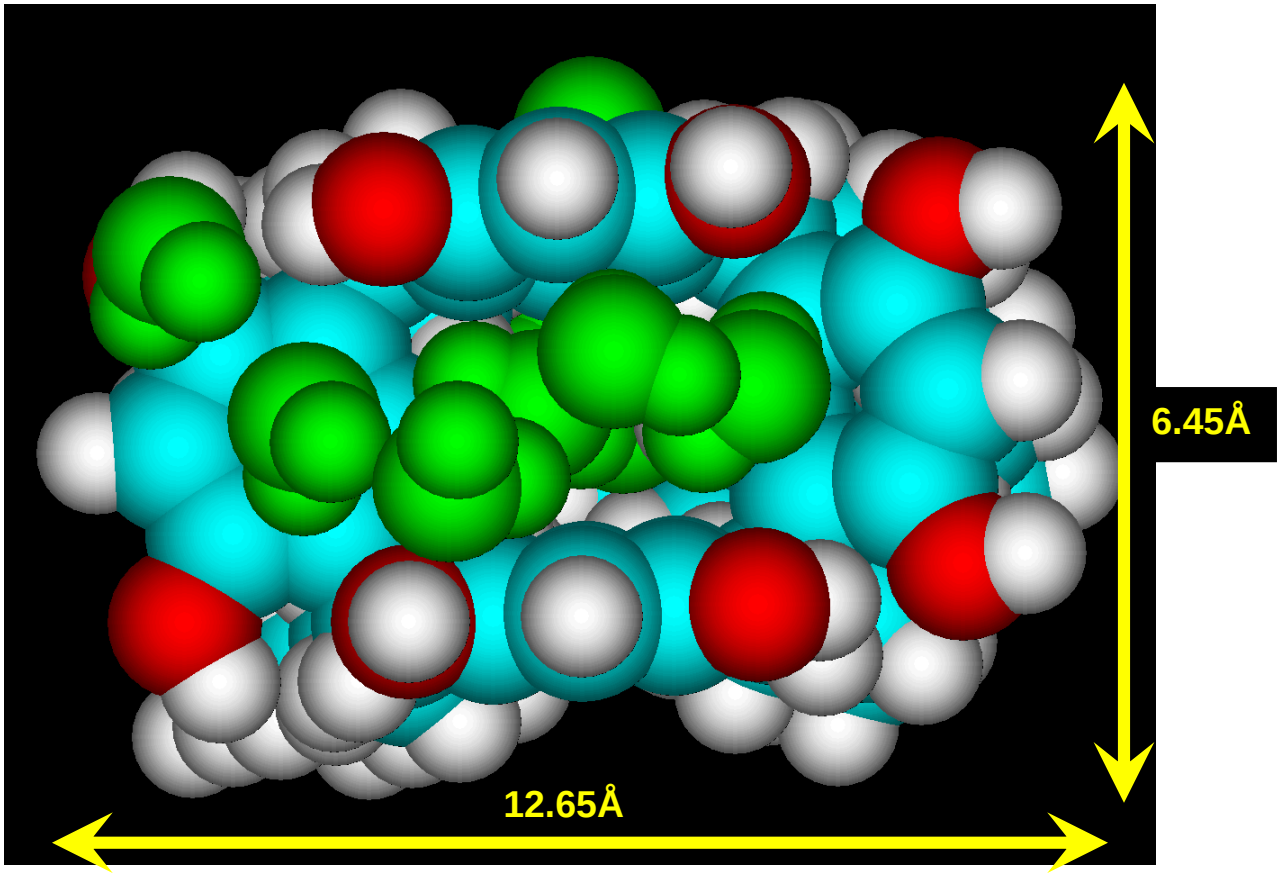
Results of the actual semester

- Research Progress

2. Polymer characterization

c. Structure simulation

Cal 10



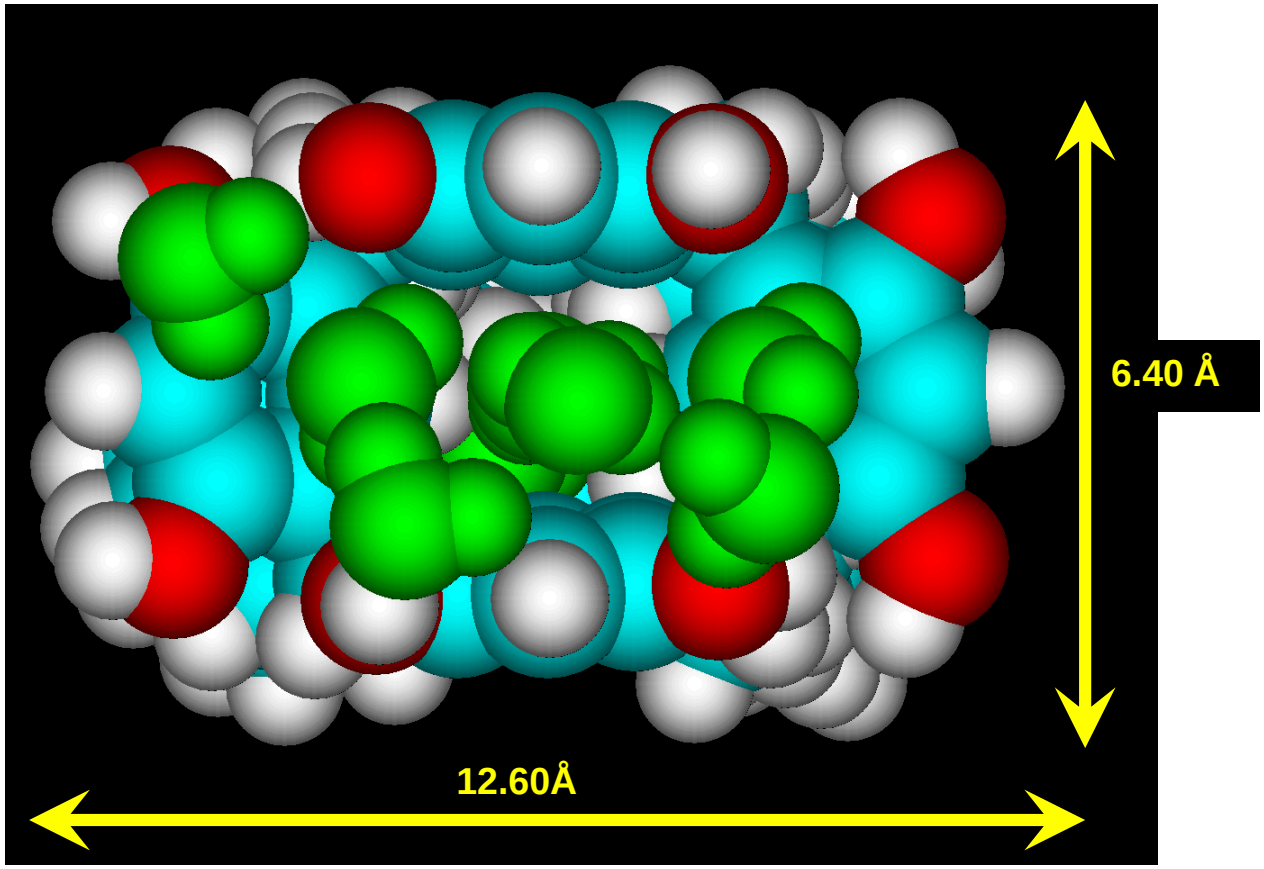
Results of the actual semester

- Research Progress

2. Polymer characterization

c. Structure simulation

Cal 11U



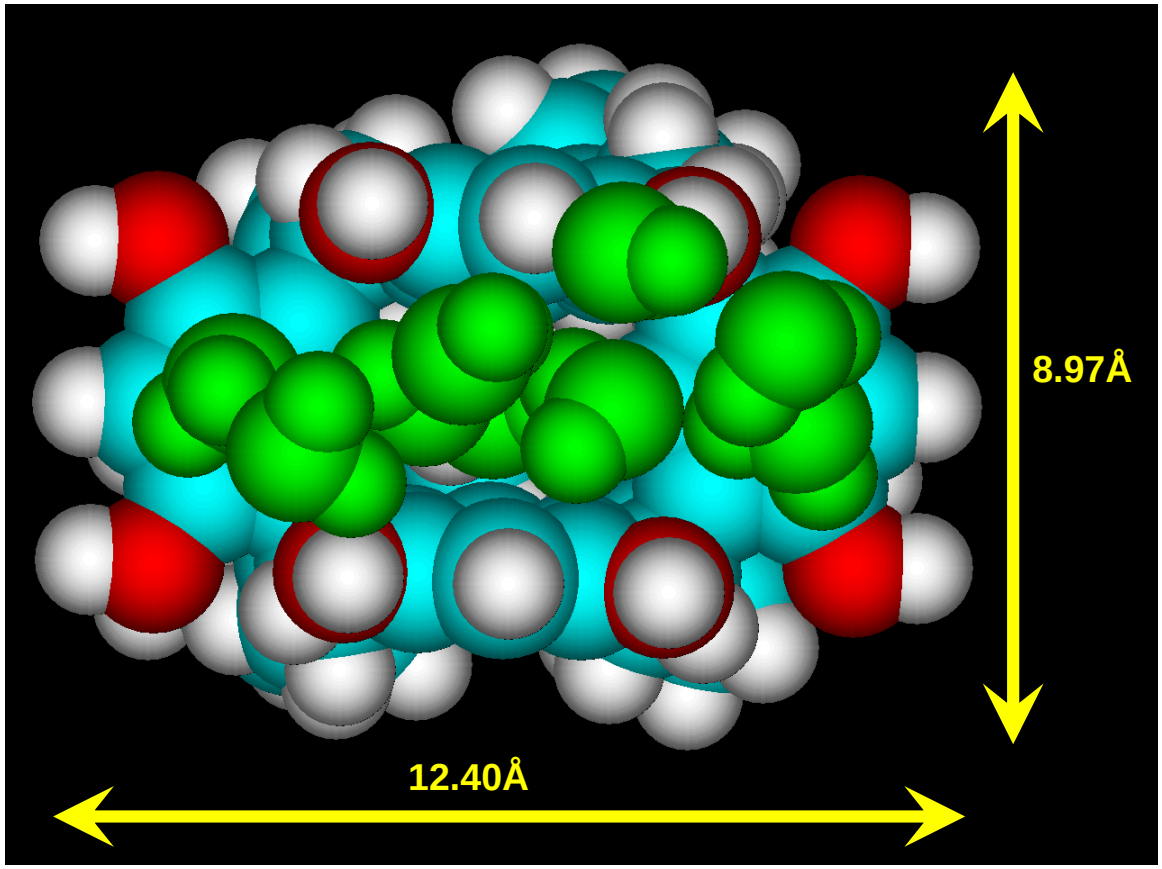
Results of the actual semester

- Research Progress

2. Polymer characterization

c. Structure simulation

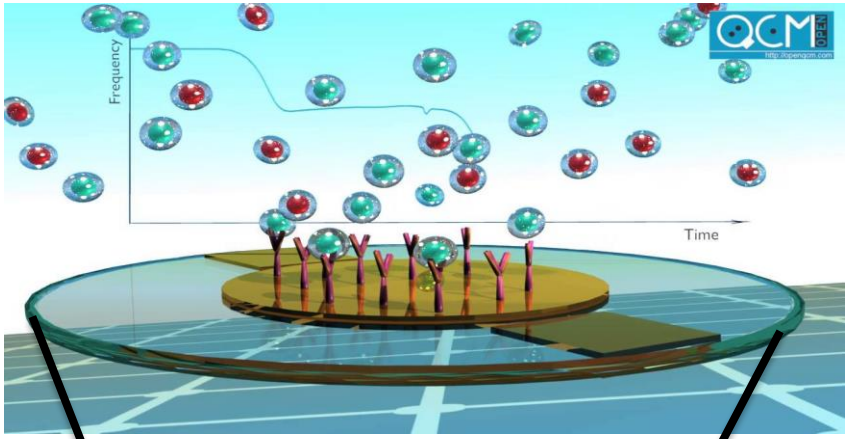
Cal 9U

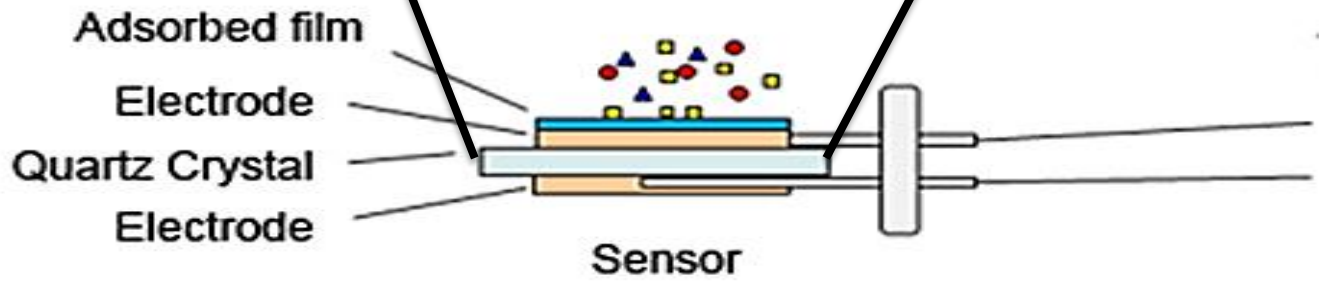


Results of the actual semester

- Research Progress

3. QCM I results





Frequency changes according to adsorption of chemicals on the surface of QCM sensor.

Results of the actual semester

- Research Progress

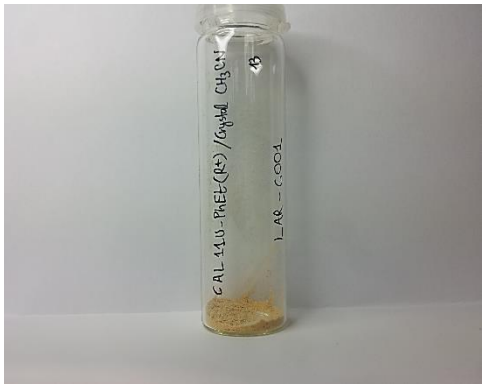
3. QCM-I Results

Results of the actual semester

- Research Progress

3. QCM-I Results

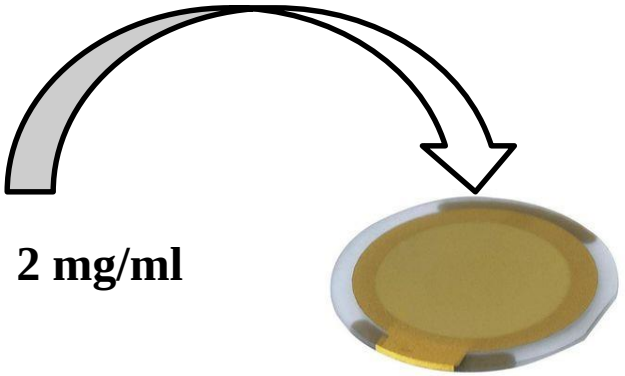
Polymer solution



CAL11U-PhEt (R+)/Crystal CH_2CN

In chloroform **2 mg/ml**

10µl coated



Heavy metal solution

200 ppm of Lead nitrate $Pb(NO_3)_2$

Results of the actual semester

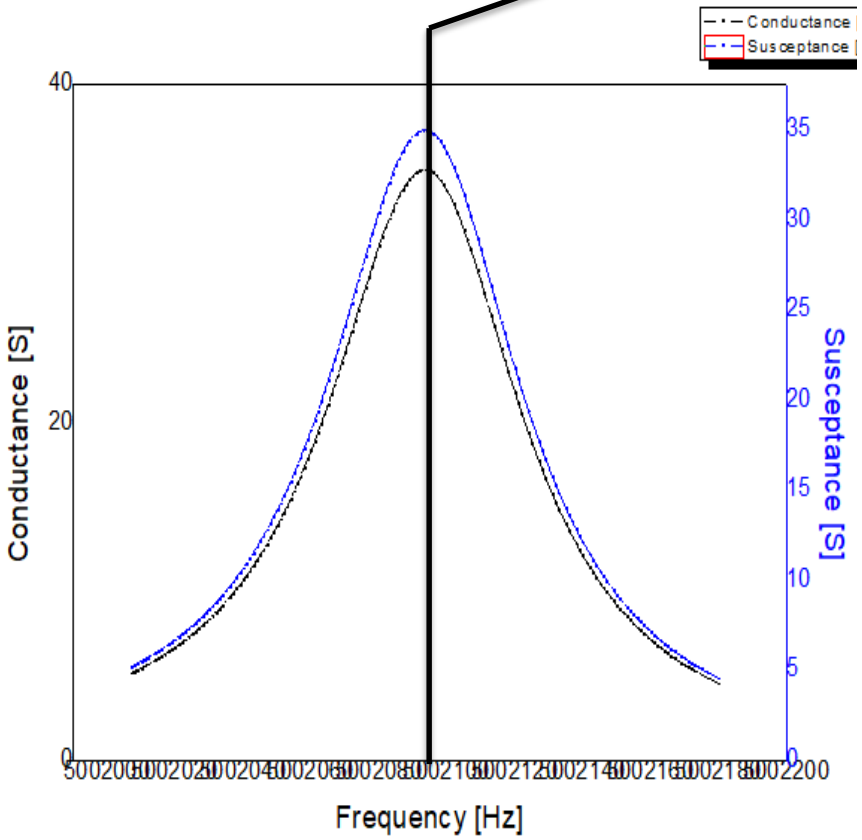
- Research ad Progress

3. QCM-I Results

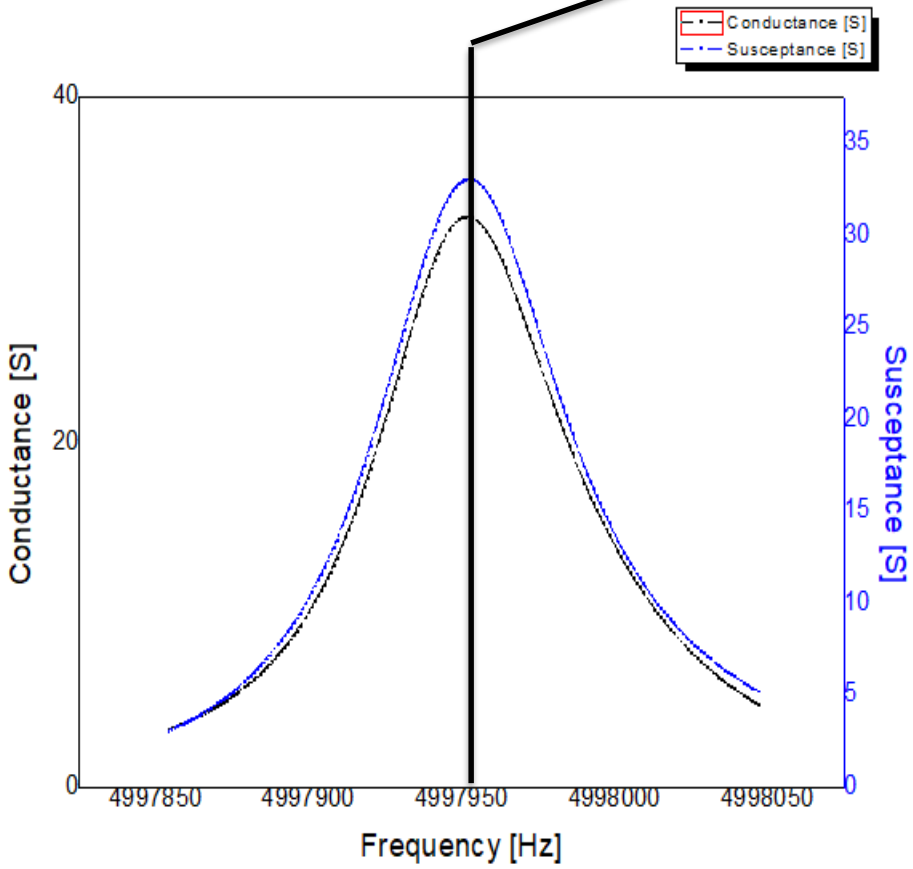
$\Delta F = 4143.3$ Hz

$F_0 = 5002093.9$ Hz

$F = 4997950.6$ Hz



Clean crystal resonance



Resonance after coating

Results of the actual semester

- Research Progress

3. QCM-I Results

$\Delta F = 4143.3$ Hz

Quantitative analysis: Sauerbrey equation:

There's a linear relationship between the frequency and the mass/ surface area

The Sauerbrey equation: $\Delta F = - C_f . (1/n) . \Delta m$

For a 5 MHz AT cut QCM:

$C = -17.7$ Hz. ng^{-1}. cm^2. and n = 1,3,5,... overtones.

$$\Delta m \text{ (ng. cm}^{-2}) = - \Delta F(\text{Hz}) / C \text{ (Hz. ng}^{-1}\text{. cm}^2)$$

The film thickness $\delta = \Delta m/\rho . A$

where '**A**' is the area and '**ρ**' is the density

If $\Delta D \sim 0$ the Sauerbrey equation will give a correct mass estimation.

If $\Delta D >> 0$ the Sauerbrey equation will underestimate the mass.

Results of the actual semester

- Research Progress

3. QCM-I Results

Then the mass is $\Delta m\ (ng.\ cm^{-2}) = 4143.3(Hz) / 17.7\ (Hz.\ ng^{-1}.\ cm^{2})$

$= 234.08\ ng.\ cm^{-2}$

For a d = 12 mm crystal , A = $1.13 cm^2$ then $\Delta m = 264.51$ ng

The film thickness $\delta = (\Delta m / \rho\ (g.\ cm^{-3}))/A(cm^2) = (264.51 * 10^{-9}/0.002) /1.13$

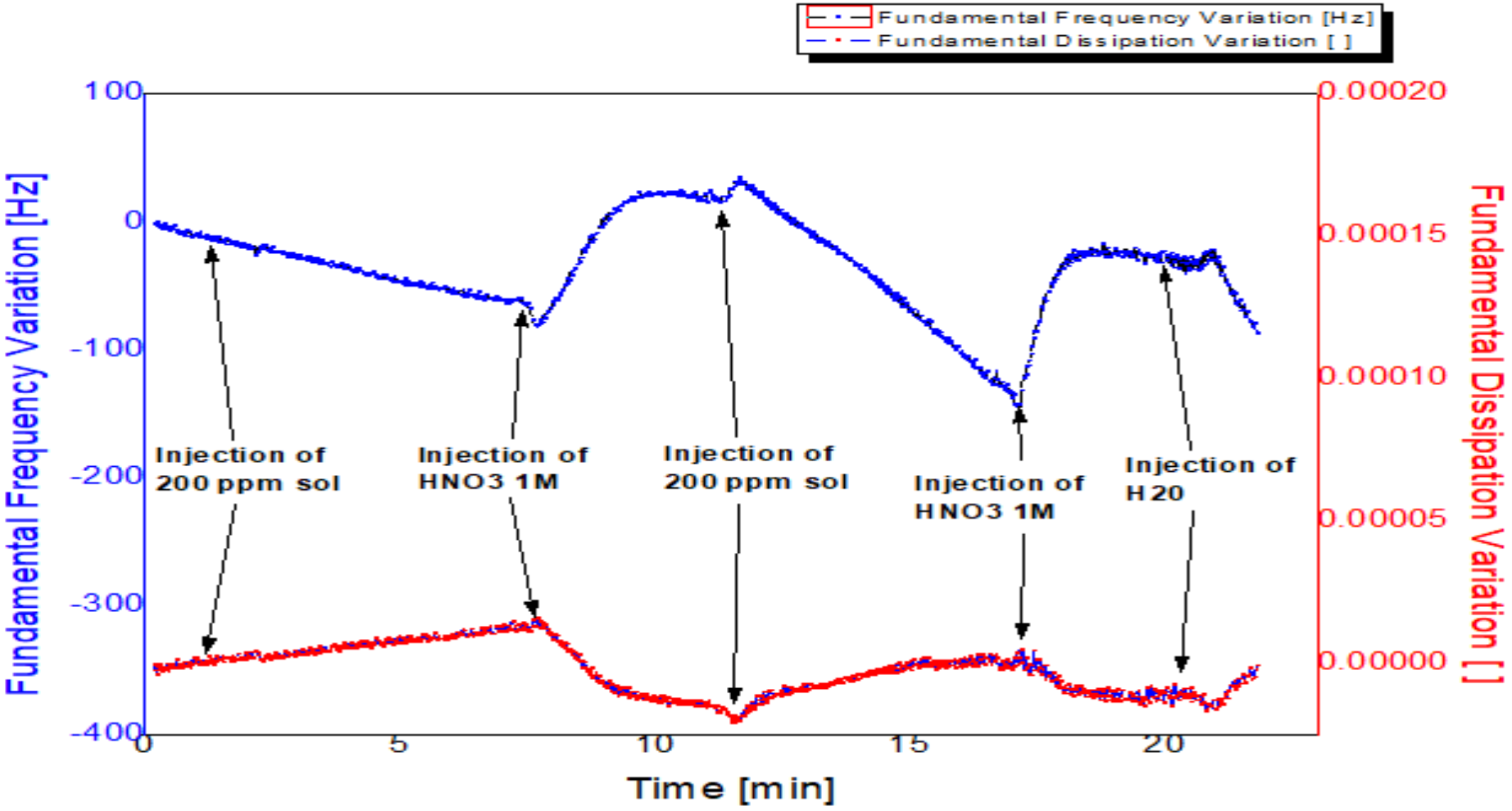
$= (1.32*10^{-4}\ )/1.13$

$=1.17*10^{-4}$ cm

Results of the actual semester

- Research Progress

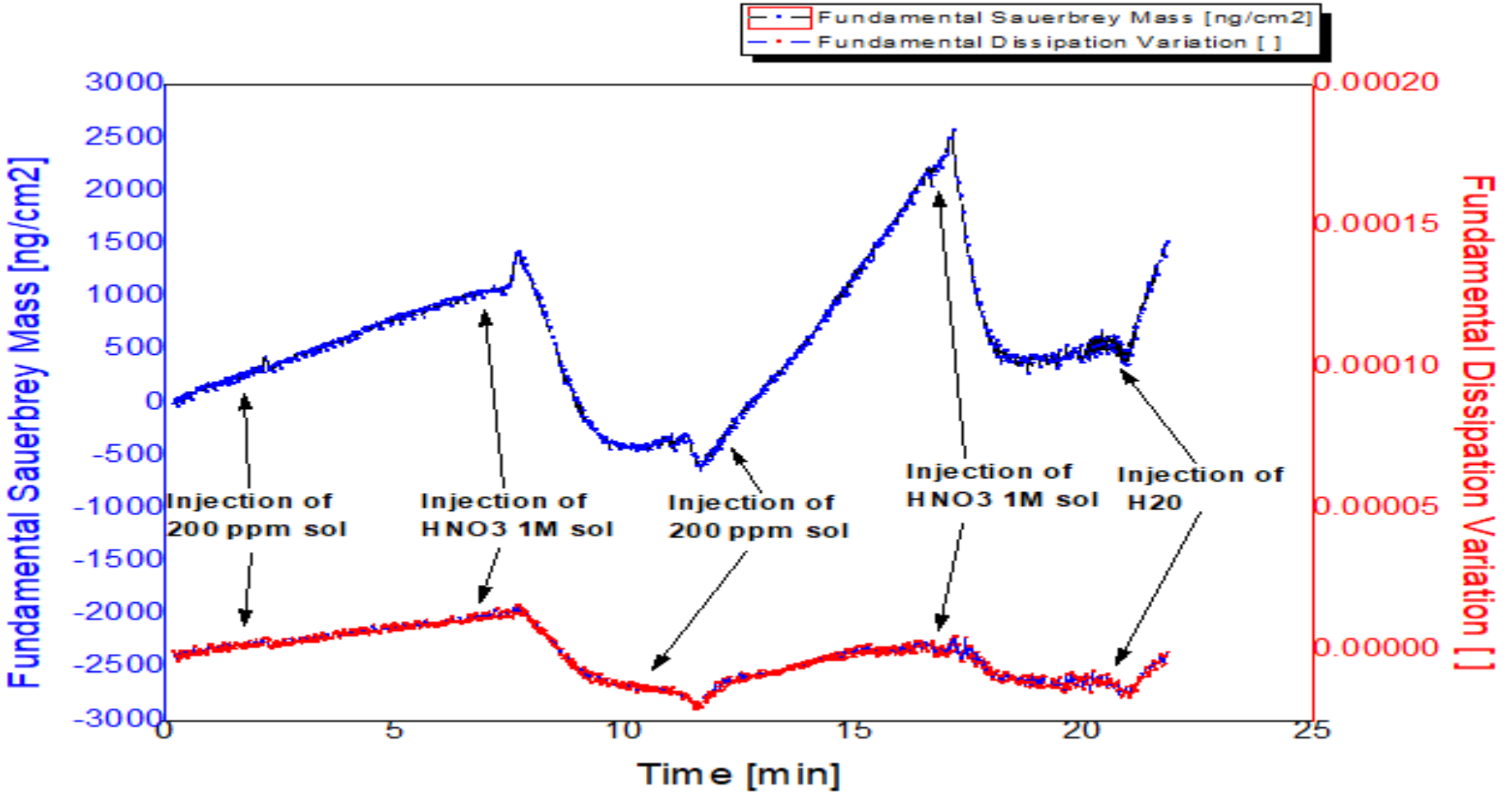
3. QCM-I Results



Results of the actual semester

- Research Progress

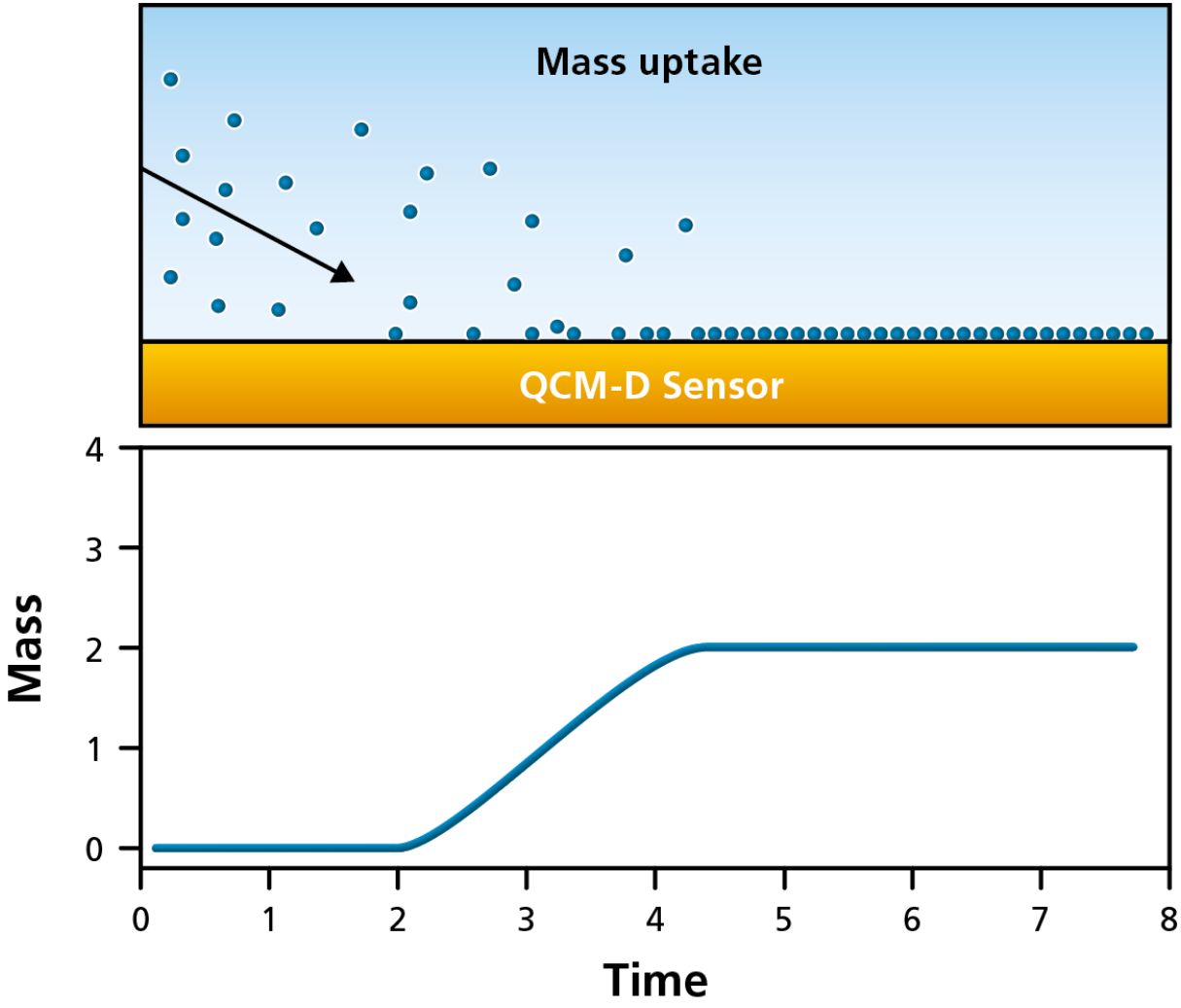
3. QCM-I Results



Results of the actual semester

- Research Progress

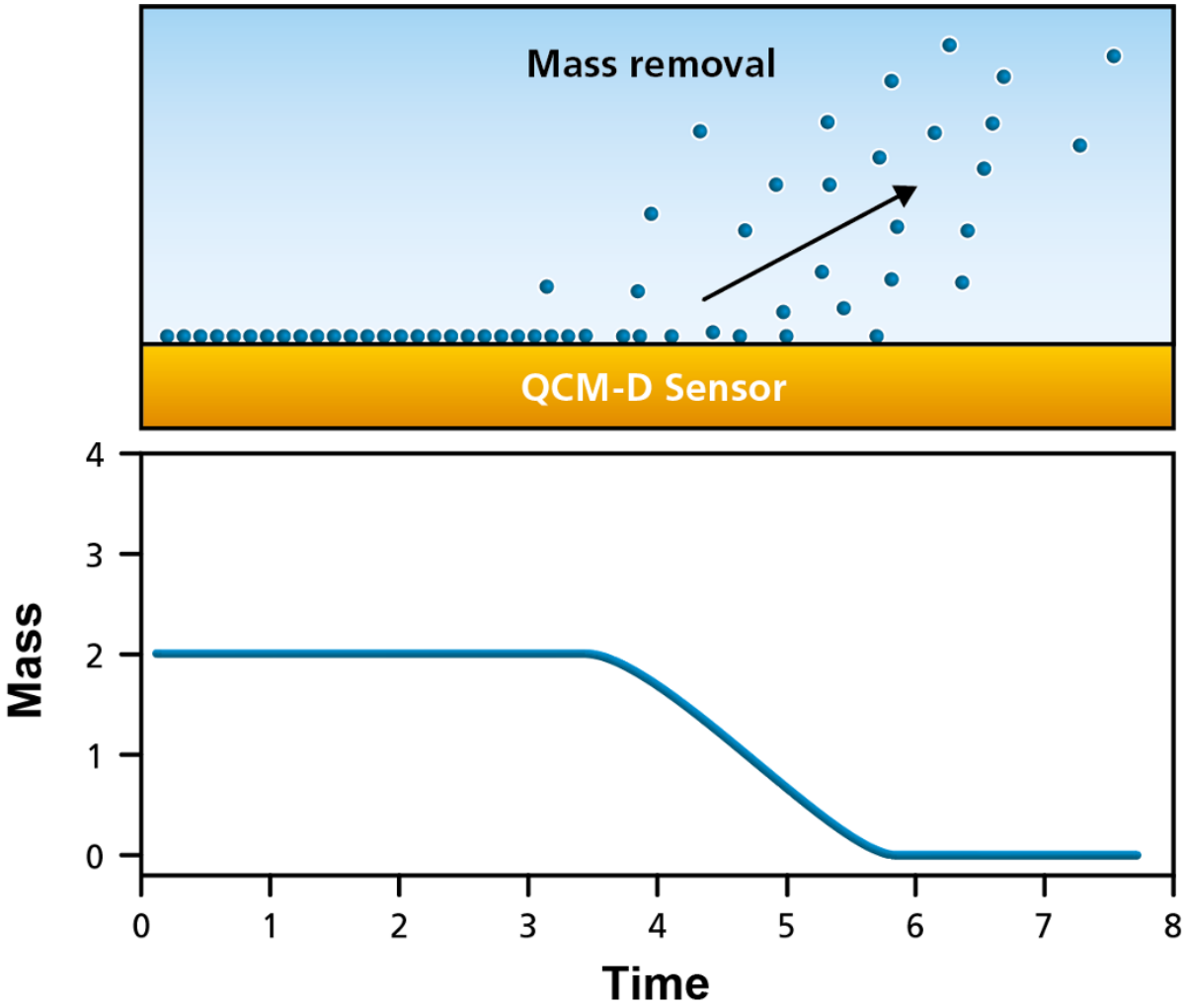
3. QCM-I Results



Results of the actual semester

- Research Progress

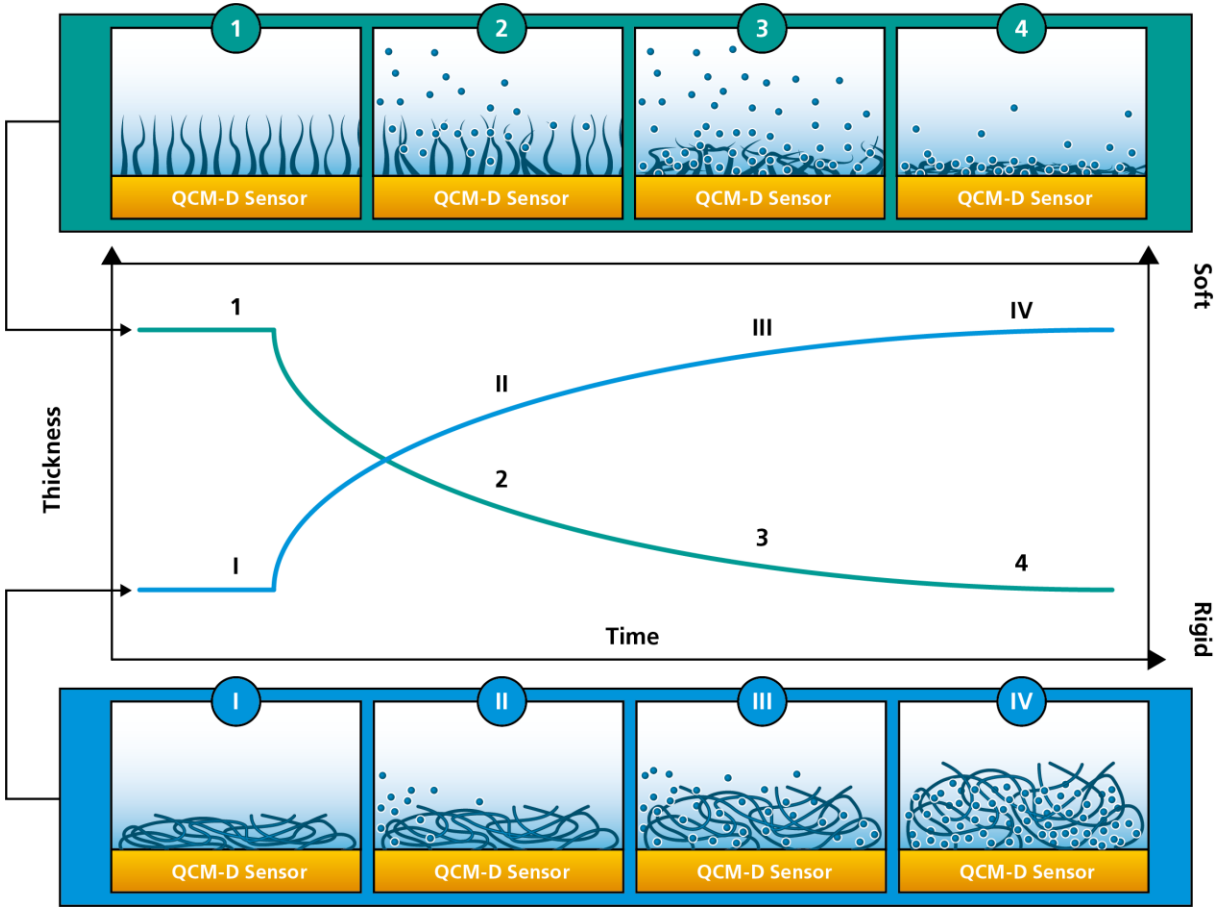
3. QCM-I Results



Results of the actual semester

- Research Progress

3. QCM-I Results



Results of the actual semester

- **Research advancement**

4. Research publication.

A transcript of a publication is underway and the plan is to submit it for publication in the next few weeks.

An abstract will be sent to take part in the Conferance: Sensors 2018, in Vienna.

Future tasks

- Continuation of characterization methods: AFM, ESEM, TGA, IR,
- Modification of sensor surface which enables us to obtain surface-modified electrodes for detecting heavy metals,
- Immobilization of polymers on sensing element surface and perform measurements using the QCM-I,
- Submission a publication and an abstract at a conference of sensors 2018.
- Two courses related to the topic of polymers will be taken in the third semester in order to strengthen the background ‘Cellulose chemistry and polymer chemistry 2’.

Conclusions

- Literature survey is well under way,
- New tool for detection of heavy-metals will be studied,
- These new tools offer new opportunities with many advantages,
- So we wish ourselves a lot of success for this topic to be one of the new trends in heavy metal sensing.


Thank You


KÖSZÖNÖM A FIGYELMET!