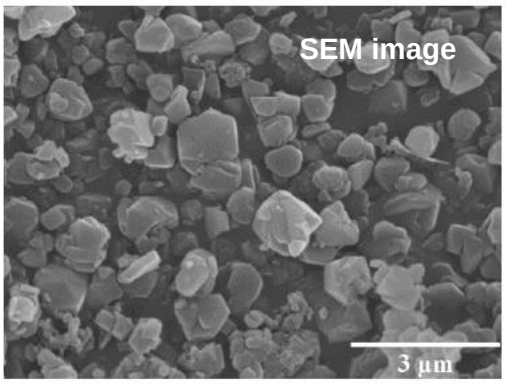
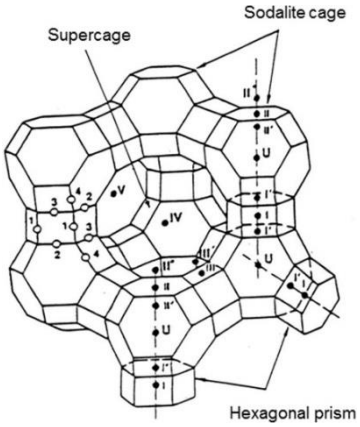
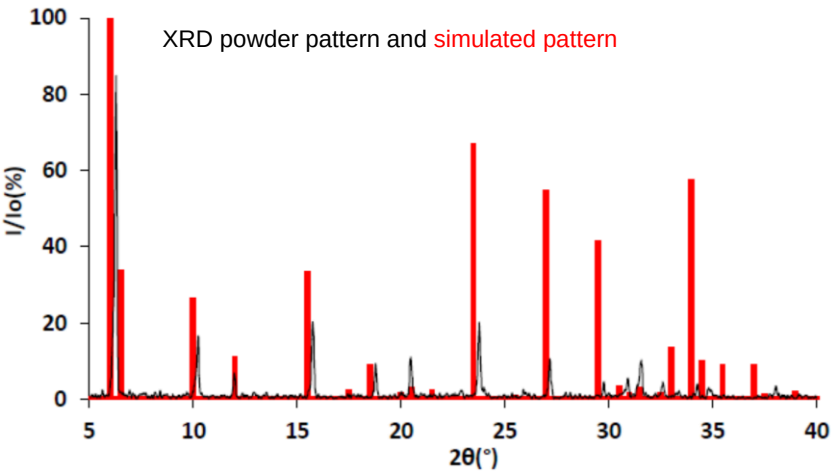
LEQ III

Heterogeneous Catalysis

SYNTHESIS AND CHARACTERIZATION OF Y ZEOLITE







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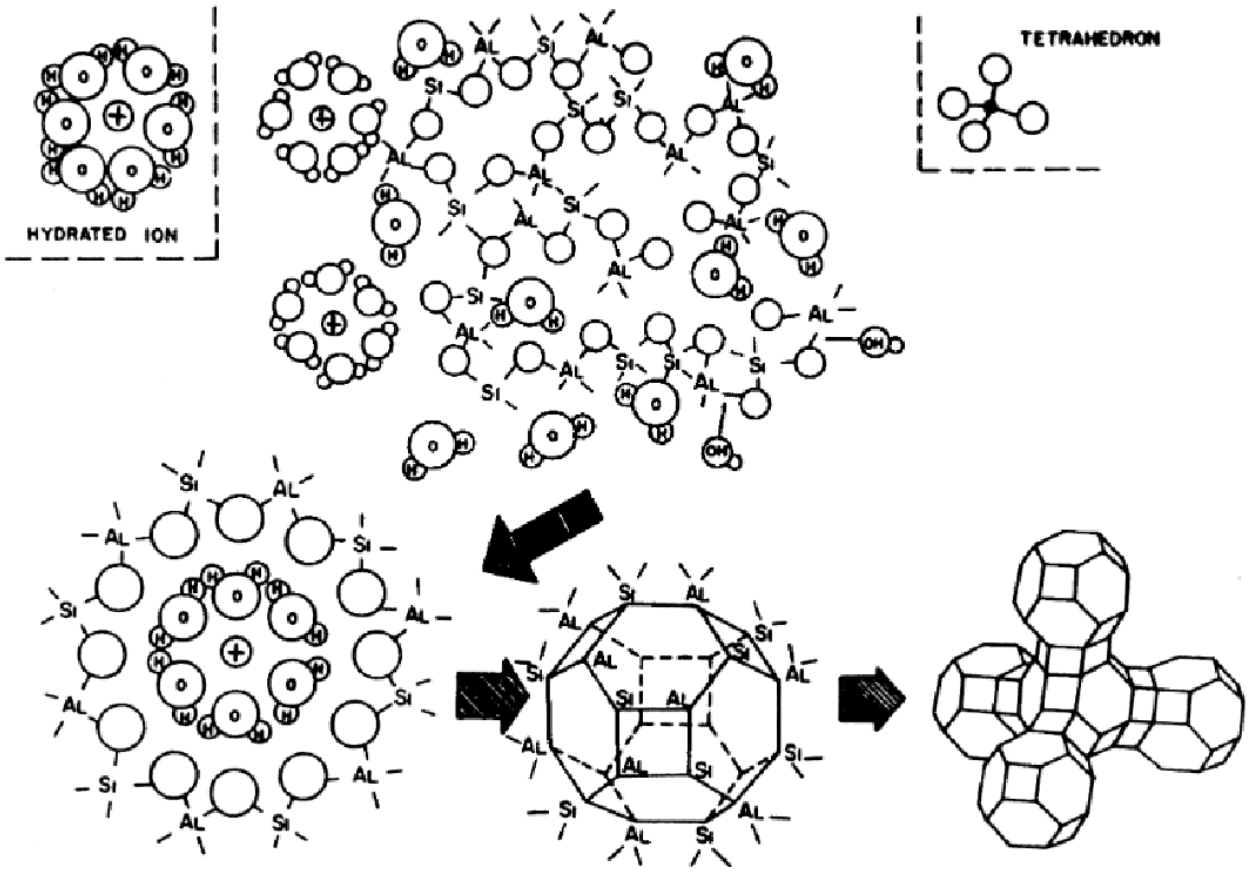
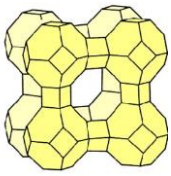


Source: Figure 2.3 from Synthesis of zeolite (ZSM-5 and Faujasite) and geopolymer from South African coal fly ash | Semantic Scholar



Synthesis of Y zeolite

(NaY)

Class #1

EXPERIMENTAL:

Materials:

- water bath with thermostat control

- stirring plate

- 100ml plastic cups

- plastic erlenmeyer 125 ml

- teflon rod and stir bar

- glass kitasato and buchner

Reagents:

- Metallic aluminium

- silicic acid, dried

- NaOH

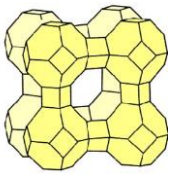
- $Al_2(SO4)_3$ To reduce the environmental impact of zeolite preparation, some groups will use recycled aluminum from tetrapack® packaging instead of pure aluminum.

Procedure:

I - Preparation of the nucleation solution

This solution involves the preparation of the following solutions:

Solution A:



Prepare a solution containing 15 g of NaOH in 35 g of H_2O.

Weigh 1.35 g of metallic aluminium and add to the previous solution.

The dissolution must be carried out carefully in the hotte because the reaction is violent and projections may occur.

Solution B:

Prepare a solution containing 3.9 g of NaOH and 8.75 g of H_2O.

Weigh 3.75 g of dry silicic acid and add to the previous solution.

Take a 1/4 (≅ 12.84 g) of the solution A corresponding and slowly add to solution B.

Transfer the mixture to a stoppered erlenmeyer and place under agitation in a water bath at 42 ± 2 °C. Leave to age for 90 minutes.

II – Preparation of the aluminosilicate gel

The gel involves the preparation of the following solutions:

Solution C Weigh 4.02 g $Al_2(SO_4)_3$ and add 15.63 g of H_2O

Solution D Weigh 3.75 g dry silicic acid.

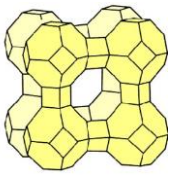
Slowly add solution D to solution C, stirring vigorously with a lab stick (teflon coated). Filter the resulting solution under vacuum and wash with iced water. Transfer to a glass and weigh the precipitate. Add water up to 22.5g.

III - Crystallization

Slowly add 3.75 g of nucleation solution (A + B) to the aluminosilicate gel (C + D). Transfer the formed gel into a polyethylene stopper and place in an oven at 92 ± 2 °C for 42 hours.

Note: In this synthesis glass material can not be used, which is replaced by polyethylene or polypropylene material. All solutions should be prepared in the hood using protective gloves.

After the crystallization step the preparation will be filtered and washed with hot water and



finally calcined at 450 °C in overnight. The calcined sample will be characterized by X-ray diffraction, XRD, (powder methods). The crystalline lots prepared by the different groups will be mixed (NaY2021) and the resulting product will be exchanged with ammonium nitrate solution to obtain HY (HY2021).

This step is not performed by students, it will be accomplished by the teacher.

Catalytic test

1-Butene isomerization

(Class #2)

The acidity of the prepared zeolite (in NaY and HY form) will be characterized by the 1-butene isomerization (A) reaction and using Hammett indicators (B).

Procedure:

A - Catalytic test for isomerization of butene-1

I-Weigh 250mg of zeolite (each group will only perform a catalytic test)

II-Fill the reactor in *pyrex* with the zeolite sample.

III-Heating the reactor to a temperature close to the reaction temperature under inert gas flow (N_2).

IV - Introduction of 1-butene in the reactor feed and start of reaction time counting.

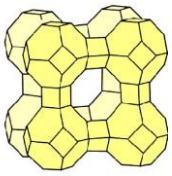
V - 7 min after the start of the reaction (this time interval is required to stabilize the feed composition to the reactor) start GC sampling of the reaction effluent. At each sampling, the reaction time and temperature in the reactor should be recorded.

VI- The analysis of the reaction effluent should be repeated at regular intervals, corresponding to the time of a GC analysis (± 14 min) until the area of the 1-butene peak remains constant.

B – Hammett acidity test

Analysis of the results

(class#3)



The zeolite samples designated NaY2018 and HY2018 will be sent to external laboratories to be characterized by techniques not available in the LEQIII laboratory: determination of specific area by BET method, X-ray diffraction (XRD), infrared spectroscopy (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and differential thermal analysis for zeolite after reaction (TG-DTA) samples. During the class (#3) the characterization data, above referred, will be analysed.

Report structure (pdf file**)**:

Cover: Title of work, student identification, date

Index

Summary: It should describe in a brief and brief way what has been done and the main results. Introduction: It should include bibliographic references to recent work.

Experimental Part: Zeolite synthesis; Catalytic tests (must include an assembly scheme used for catalytic tests).

Results and discussion

Annex: GC analysis of the reaction effluent (include a chromatogram with product identification); Conversion calculations and selectivities.

Bibliography The report must be sent, no later than 2 weeks after the last class, by email (apsoares@tecnico.ulisboa.pt) in pdf file.