In-Situ Characterization of Zeolite Synthesis Process

IPRO 302 – SPRING 2003

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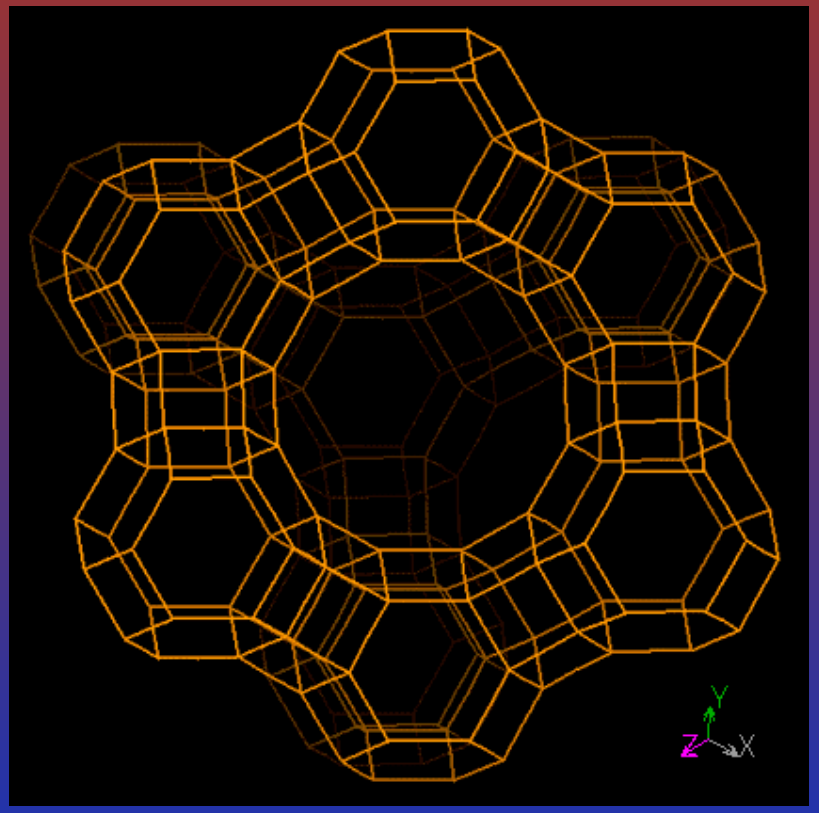
Presentation Overview

- Introduction to Zeolites
- Why is this project beneficial?
- Motor, Heater.
- Plastic, Damping.
- Possibilities.
- Acknowledgements.

Introduction to Zeolites

- Zeolites
  - Porous
  - Naturally occurring
  - Synthetic
  - Aluminosilicates.
- Rigid Pores Dimension
  - Small compounds to fit inside
  - Larger compounds cannot.



Structure of Faujasite (FAU)

Significance in this Project

- Zeolites characterization
  - Traditional post-synthesis,
  - ***In-situ*** studies – recent development
- Structural changes can be studied.
- Traditional *in-situ*
  - Single synthesis – inefficient
- Multiple syntheses
- Combinatorial analysis

Reaction

The IPRO Team must be able to produce a reaction in the lab before testing at the APS.

- Chosen synthesis : Preparation of Linde Type A (LTA)

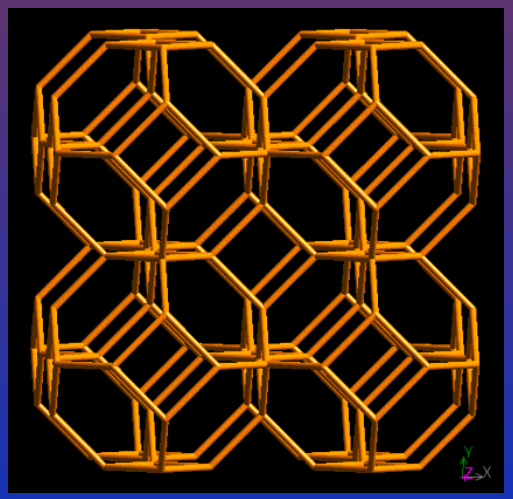
$NaO_2 \bullet Al_2O_3 \bullet 3\ H_2O$

+

$Na_2SiO_3 \bullet 5\ H_2O$

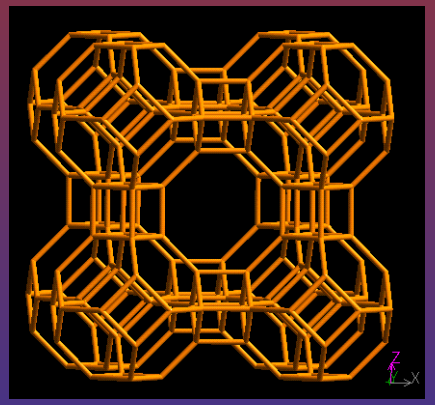
1 Hour

Sodalite



$Na_6Al_6Si_6O_{24}$

LTA

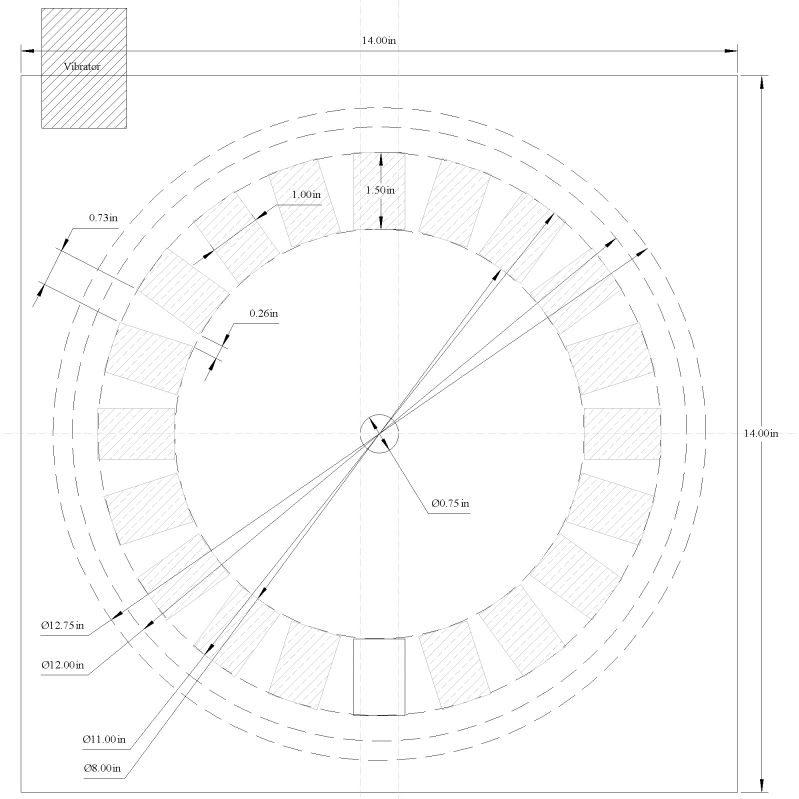


$Na_{12}[(AlO_2)_{12}(SiO_2)_{12}]$

$\bullet 27\ H_2O$

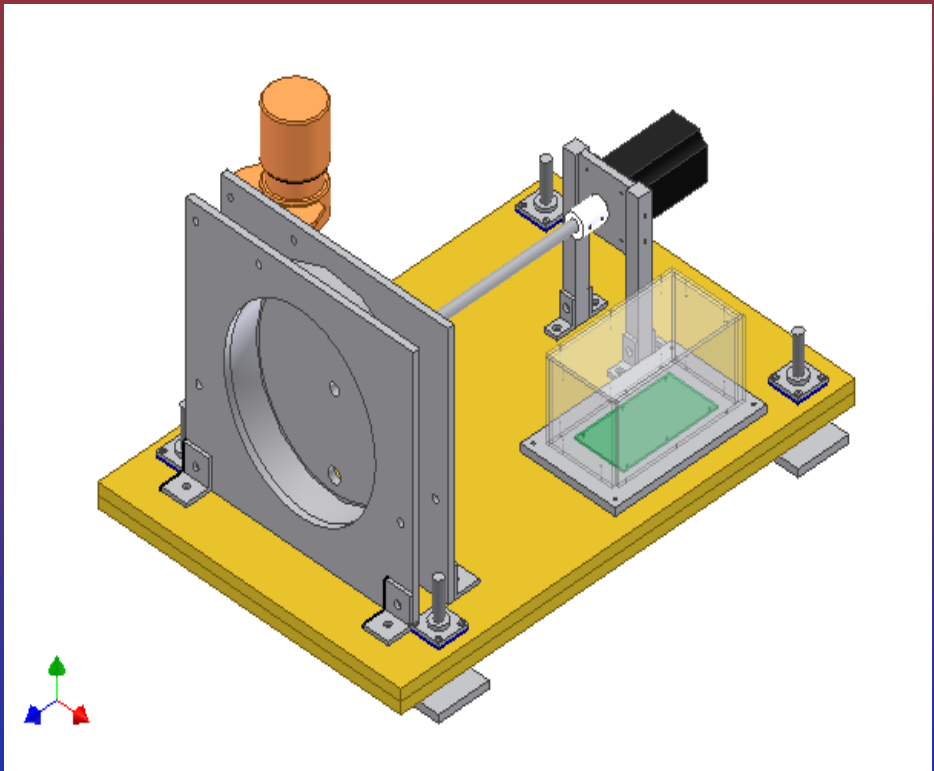
3 Hours

Before and After



- Last semester's design
- Only the cell housing was complete

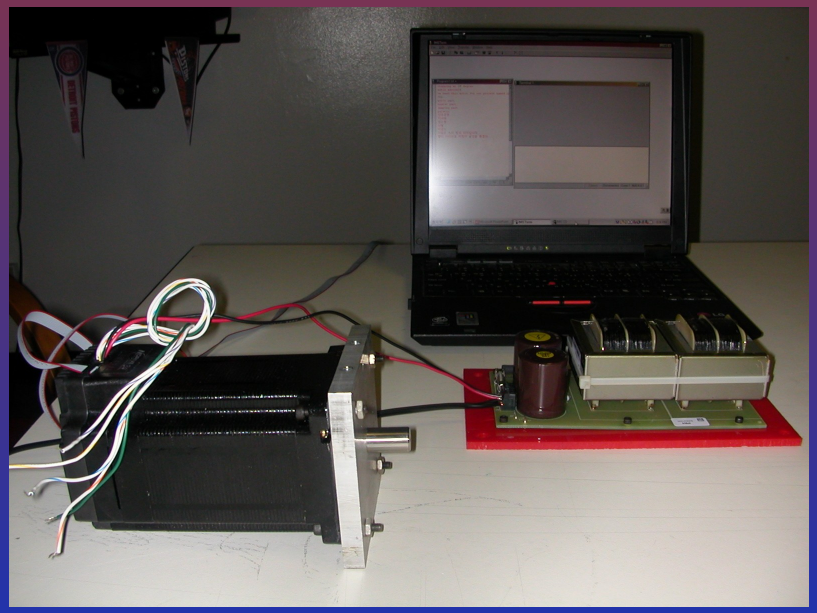
Before and After



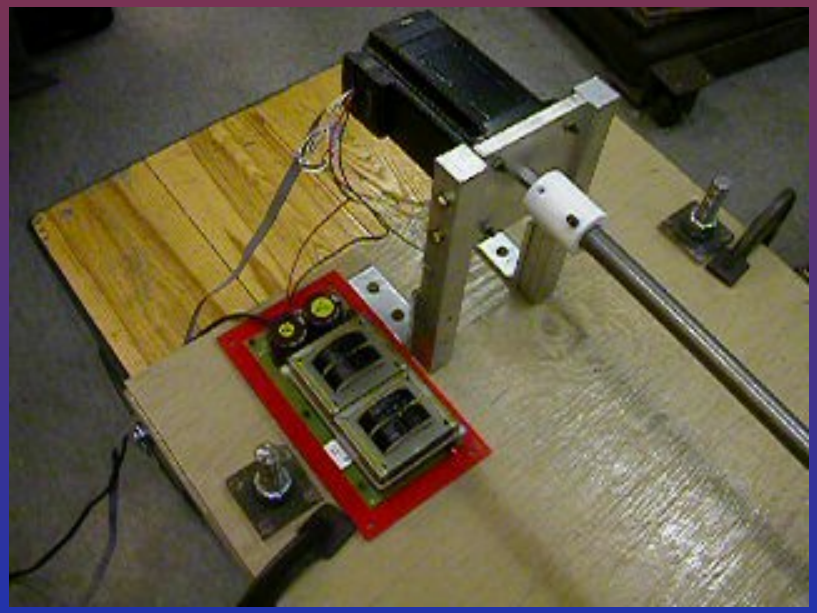
- Wheels for mobility
- Base for stability
- Adjustable height

Motor

- Connect to computer

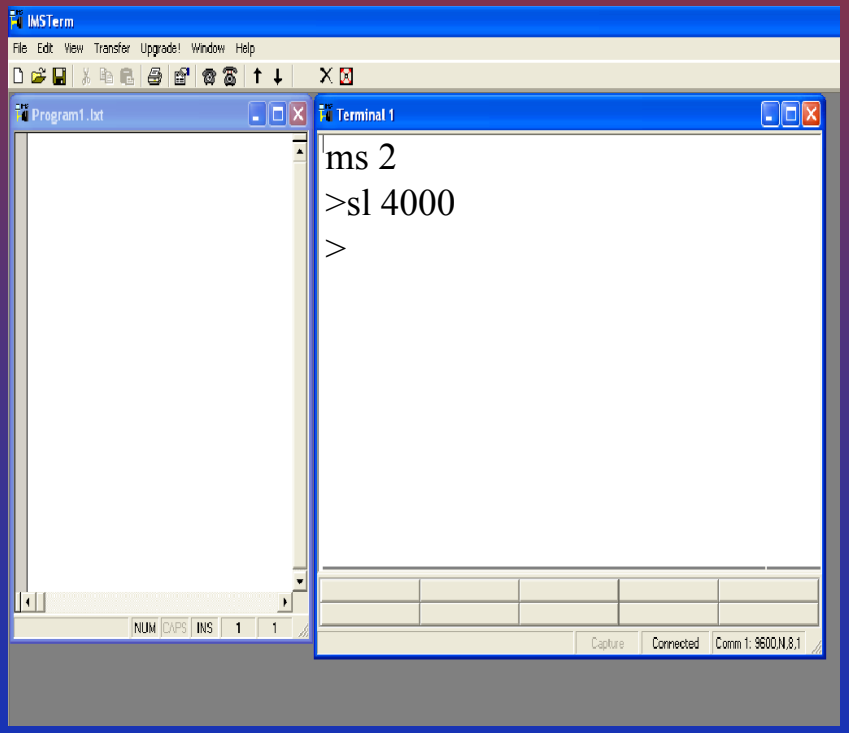


- Connect to the shaft



Motor

- Stepping at 18 degrees

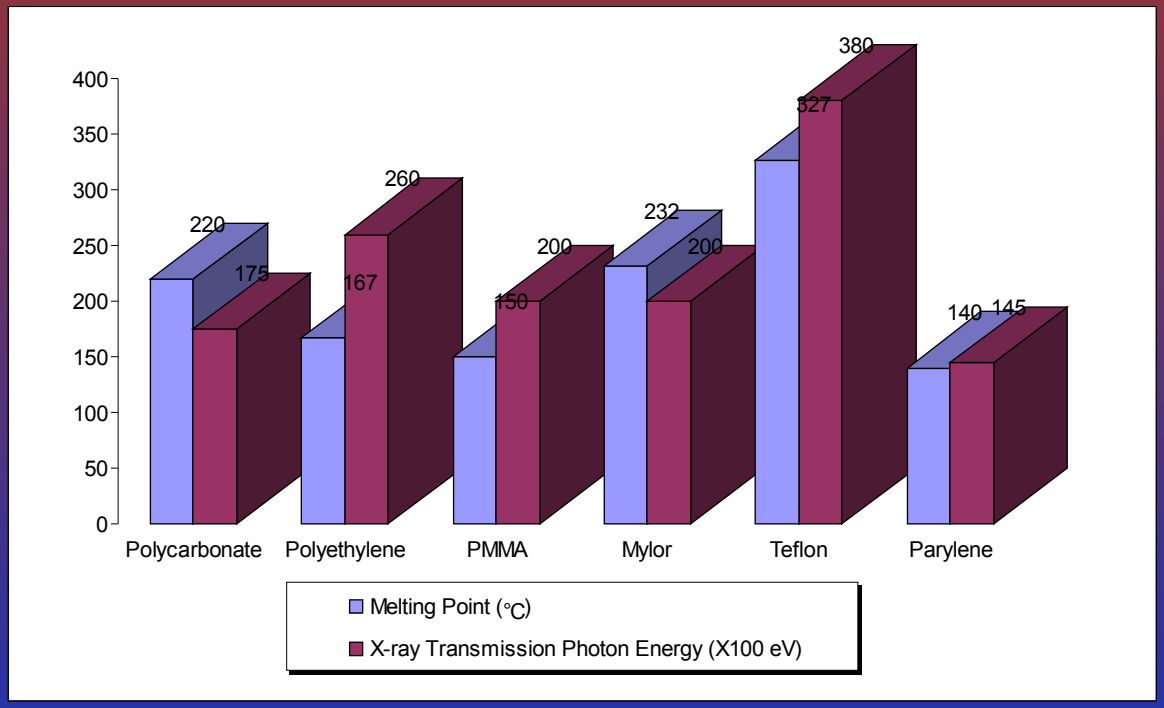


Microstep Resolution Settings (MS)	
MS= (Microsteps/Step)	Steps/Rev
Binary Microstep Resolution Settings	
2	400
4	800
8	1,600
16	3,200
32	6,400
64	12,800
128	25,600
256	51,200

Decimal Microstep Resolution Settings	
5	1,000
10	2,000
25	5,000
50	10,000
125	25,000
250	50,000

Table 2.6: Microstep Resolution Settings

Plastic



Finalist

- **Plastic: Polycarbonate**
- **Formula: $C_{16}H_{14}O_3$**
- **Melting Point: 220℃**

Shaker

- Products required to be in suspension for characterization to occur.
- Shaker model: Vibco #SCR-100

Heater

- Control temperature.
- Radiant heat.
- Operate under hydrothermal conditions.

Heater

- Tubular Electric Heater.
- Temperature Controller.
- Solid State Relays.
- Fine Tip Temperature Probe.

Base

- Wooden base.
- Wide-base steel legs.
- Motor suspended so that x-ray has space to pass through.

Hydrothermal conditions

- Required to counter pressure that develops in the teflon pouches.
- Use of steam at 200 C.
- Safety Concerns.

Damping

- Shaker causes reaction cell to vibrate.
- Translational movement may disrupt x-ray beam and prevent good results.
- Different damping methods: Mass Loading, Structural Reinforcement, Extensional Damping.
- Rubber layer attached to base legs.
- Motor side wrapped Visco-elastic Damping Foil.

Approach

- Confirm technique

- Teflon pouches
- Mixing
- Conformity

Approach

- Test cell at the Advanced Photon Source

Possibilities

- Quick analysis
  - Traditional –
    - 20 samples = 3 hours prep + 10 hours analysis
  - *In situ* –
    - 20 samples = 3 hours prep + 0 hours analysis

Possibilities

- Better understanding of zeolite formation
- *In situ* analysis more common in industry
- Creating "customized zeolites" for a client
  - Environmental cleanup
  - Petroleum hydrocarbon "cracking"

Acknowledgements

- Thomas Torres
- Intelligent Motion Systems
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- Saadia Tabussum
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Questions?