Encapsulation of Low Temperature Ionic Liquids in Zeolites: A Vibrational Spectroscopy Study

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INTRODUCTION

The unique textural properties of zeolites have resulted in their extensive use in catalysis, gas separation technology and various other industrial applications. Ionic liquids (ILs) are salts having very low melting temperatures. Their thermal stability, non-volatility, non-flammability and their high ionic conductivity make them useful in different applications such as separation technology[1], catalysis[2], while they are used, lately, extensively in electrochemistry[3]. Since they exhibit low melting points they are used in a wide temperature range and they can be handled as liquids in ambient temperature. The encapsulation of ILs in zeolite powders might lead to the formation of new materials with enhanced catalytic or electrocatalytic properties. The aim of this work is to study the encapsulation of the H-3-methylimidazolium bis(trifluoromethanesulfonyl)imide (HMINTf2) (Scheme 1) in the NaY-FAU zeolite framework (Scheme 2).

![Scheme 1: The molecular structure of HMINTf2.](image1.png)

![Scheme 2: NaY-FAU zeolite framework.](image2.png)

EXPERIMENTAL PROCEDURE

> Ionic Liquid Encapsulation

The encapsulation of HMINTf2 was performed according to the following procedure:
1. NaY-FAU (SIGMA-ALDRICH Co.) crystals were degassed at ~270°C overnight,
2. HMINTf2 (SOLVIONIC S.A.) was mixed with the NaY powder inside a glass tube with a weight ratio HMINTf2/NaY = 250/1,
3. the tube was heated at 230°C for ~8 hrs,
4. Soxhlet extraction was performed for ~10 hrs to remove the excess ionic liquid.

Steps 1-3 were carried out in a glove bag.

> Characterization

HMINTf2/NaY composites were characterized using:
- X-ray diffraction (XRD), Bruker D8 Advanced
- Attenuated Total Reflectance/Fourier Transform-Infrared (ATR/FT-IR, VARIAN EXCALIBUR) and FT-Raman Spectroscopy (EQUINOX 55) to obtain information on the crystalline structure of the synthesized composites,
- Thermogravimetric Analysis (TGA, Q50 TA Instruments, Inc.) in order to evaluate the IL loading,
- N2 physisorption measurements (QUANTACHROME, AUTOSORB-1) to evaluate the textural properties of the composite.

RESULTS AND DISCUSSION

> XRD

![XRD spectra of NaY, HMINTf2 and the obtained composite after Soxhlet extraction.](image3.png)

CONCLUSIONS

- The zeolite is not destroyed by the entrapment procedure.
- The amount of HMINTf2 present in the composite after Soxhlet extraction is ~20%.
- The HMINTf2 molecules probably reside in the micropore volume of NaY:
  - (i) shift of X-ray diffraction peaks that indicate unit cell expansion,
  - (ii) changes of relative intensities of X-ray diffraction peaks,
  - (iii) reduction of specific surface area and micropore volume,
  - (iv) changes of the NTf2 conformation from trans (pure HMINTf2)[4,5].

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REFERENCES