

UEC-4, -5 and -6 silicoaluminophosphates with 3D-structures from a 2D-organoaluminophosphate

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Abstract

Zeolitic structures can be synthesized in many ways, one of them is to use a layered precursor as source of T atoms (Si, Al, P, ...) in the presence or absence of organic structure directing agent (SDA). In this work the possibility of using a layered aluminophosphate, with N,N-dimethylbenzylamine as SDA, in combination with another organic directing agent to create three new zeotypic structures UEC-4, -5 and -6 was studied.

Key words: 2D-to-3D transformation, Silicoaluminophosphate, Zeotypes

Introduction

The possibility of combining two SDAs in the synthesis of zeotypes have been studied using one SDA from a layered aluminophosphate and another SDA added in the synthesis reactor¹.

The combination of N,N-dimethylbenzylamine from the AIPO-CJ70² with the addition of silica and tetraethylammonium, n-butylamine and hexamethylenimine in a hydrothermal process creates three new zeotypes silicoaluminophosphate with structures analogous to SAPO-5, -15 and -35, respectively, were named **Universidade Estadual de Campinas -4, -5 and -6**.

Results and Discussion

The powder XRD from UEC-4 (Figure 1a) shows a crystalline AFI, the XRD from UEC-5 (Figure 1b) shows only the crystalline phase from the SAPO-15 analogous, and the XRD from UEC-6 (Figure 1c) shows a LEV phase but with SAPO-15 contamination.

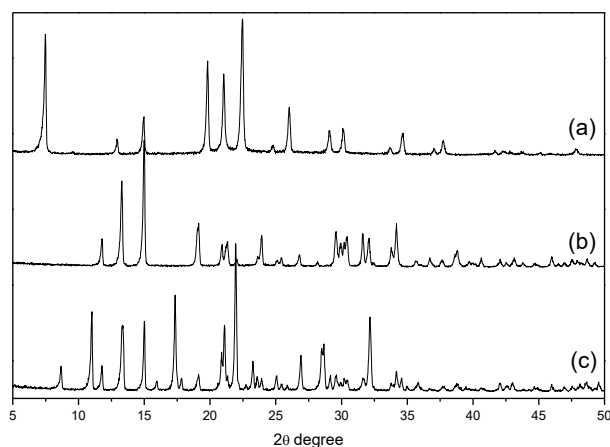


Figure 1. Experimental powder XRD from a) UEC-4, b) UEC-5 and c) UEC-6.

In the UEC-4 ¹³C MAS NMR, peaks from tetraethylammonium (7.5 and 53.0 ppm) and N,N-dimethylbenzylamine (41.5, 62.0 and 130.9 ppm) were observed. Their simultaneous presence means that both SDAs are working together to build the AFI-type structure of UEC-4. In the UEC-5 ¹³C MAS NMR, the n-butylammonium (11.3, 19.5, 36.3 and 46.4 ppm) added in the synthetic process causes the formation of the three-dimensional structure of SAPO-15, because of the pore size of the UEC-5 and the synthesis temperature, the N,N-dimethylbenzylamine molecules may be expelled from the

final structure or decompose during the hydrothermal process.

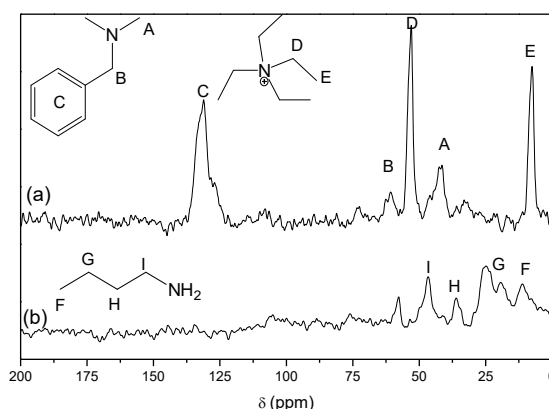


Figure 2. ¹³C CPMAS NMR from a) UEC-4 and b) UEC-5.

Scanning electron microscopy was used to monitor the transformation of the lamellar AIPO-CJ70 to a condensed morphology. The signals of the layered precursor are still evident on the surface of the final crystals, indicating a clear 2D-to-3D transformation.

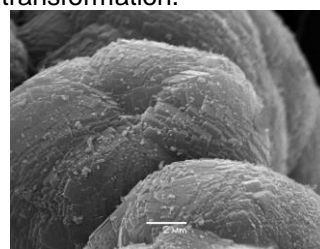


Image 3. SEM from UEC-4.

Conclusions

A new synthesis method to prepare AFI-type, SAPO-15 and LEV-type structures has been found, starting from a single source of Al and P in layered form. The 2D to 3D transformation is only possible because of the activation of the layered aluminophosphate by the addition of silica. The ¹³C NMR indicated that both N,N-dimethylbenzylamine and tetraethylammonium work together to build the UEC-4 structure, while the first decompose to form UEC-5.

Acknowledgement

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