Journal of Advances in Applied Sciences and Technology

ISSN : 2393-8188 (print)
2393-8196 (online)
www.milliyasrcollege.org.journal.php

STRUCTURAL STUDIES OF NATROLITE BY SOLID STATE HIGH RESOLUTION ^{29}Si NMR SPECTROSCOPY

H.V. Bakshi

ACS College, Shankarnagar, Tq. Biloli, Dist. Nanded.
hvbakshi@rediffmail.com

ABSTRACT:

The high resolution magic angle spinning ^{29}Si and ^{27}Al spectra of Natrolite have been studied. NMR spectroscopy allows us to establish the structure of Natrolite. Using the spectra Si / Al ratio of Natrolite is obtained.

KEYWORDS: Zeolite, Natrolite, NMR, Crystal structure.

1. INTRODUCTION

Zeolites are hydrated aluminiosilicates of alkali and alkaline earth metals. The framework structure of Zeolites consists of inter crystalline channels and voids. Water molecules occluded in the channels are loosely bound to extra framework cations, such as Na, K, Ca etc. The structural characteristics of group five zeolites were established by Pauling [1] and Taylor [2] followed by several workers [3-7]. Natrolite belongs to group V zeolites which are known to be fibrous zeolites. The characteristics of fibrous habit of zeolite crystals is parallel to c axis.

NMR allows us to obtain direct information about the crystal structure of zeolite. In NMR when a certain spinning nuclei in strong magnetic field when irradiated by second weaker field perpendicular to it, gives characteristics absorption of energy. The ordering of Si and Al atoms in zeolite framework is also very important for the spatial distribution of non-framework cations and the chemical property of zeolites. ^{29}Si NMR spectroscopy of the framework silicon atoms is a very useful tool for structural studies of solid silicates and aluminosilicates [8-10]. It was of interest to study ^{29}Si MAS, ^{27}Al MAS spectra of Natrolite. With the NMR spectra it was aimed to calculate the Si/Al ratio.

2. RESULTS AND DISCUSSION

2.1 EXPERIMENTAL

In the present study samples of natural Natrolite were obtained from different parts of Maharashtra. Two varieties of Natrolite were selected for NMR study and designated as A and B. The crystals were cleaned, crushed and sieved to get 106 µm size crystals. The powdered samples were washed repeatedly and dried.

2.2 NMR MEASUREMENTS

All ^{29}Si MAS NMR spectra were recorded in 47 KG field at 59.627 MHz with pulse width 2 µs and repetition time 2s. Reference is taken to be TEOS.

For ^{27}Al MAS NMR spectra is recorded at 78.205 MHz. The pulse width is 1 µs with repetition time 200ms.

2.3 ASSIGNMENT OF NMR SPECTRA

We have assigned NMR spectra to show degree of silicon substitution by aluminium in the second co-ordination sphere of Si atoms. The ^{29}Si MAS NMR spectrum of Natrolite (sample A) is as shown in fig. 1.

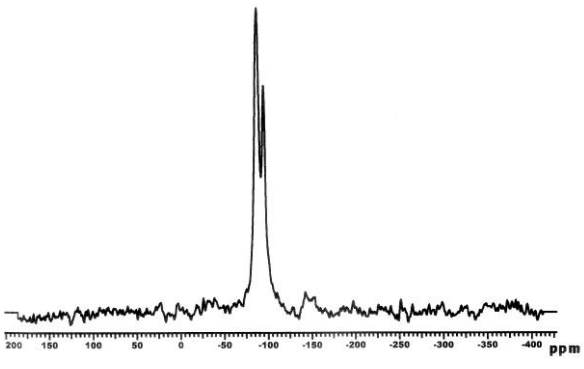

Fig. 1 ^{29}Si MAS NMR spectrum of Natrolite (sample A)

The ^{29}Si spectrum of sample A consists of two lines at -97.2 ppm and -92.8 ppm can be assigned to

Si(2Al) and Si(3Al) units respectively. In sample B the values 96 ppm and -83.9 ppm can also be assigned to Si(2Al) and Si(3Al) units. Si/Al ratio obtained in sample A and sample B is shown in Table 1.

Table 1: ^{29}Si MAS NMR isotropic chemical shifts of Natrolite

Zeolite	Si/Al	Si(4Al)	Si(3Al)	Si(2Al)	Si(1Al)	Si(0Al)
Sample A	1.8	-	-92.8	-97.2	-	-
Sample B	1.9	-	-83.9	-96	-	-

3. CONCLUSIONS

1) ^{29}Si spectra is sensitive tool to obtain direct information about crystal structure of Natrolite.
2) It provides qualitative information about Zeolite structure especially about Si/Al ordering.

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