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FTIR BAND ASSIGNMENT AND IDENTIFICATION OF SURFACE CONTAMINATION IN NaP ZEOLITE: A DETAILED SPECTROSCOPIC ANALYSIS

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Abstract: *This study presents a detailed band-by-band analysis of the Fourier-transform infrared (FTIR) spectrum of hydrothermally synthesized NaP-type zeolite (GIS framework type), obtained using pure chemical reagents. The analysis was performed to assign each absorption band in the 4000-400 cm^{-1} range to specific structural vibrations of the aluminosilicate framework or to surface contamination. The framework-related bands-at 3444, 1638, 991, 700, 608 and 455 cm^{-1} were assigned to hydroxyl, adsorbed-water and Si-O(Al) tetrahedral vibrations, while the bands at 2923, 2854, 1448 and 1405 cm^{-1} were identified as a single set of CH_2 vibrations arising from trace organic contamination rather than from the zeolite structure itself. The results demonstrate the usefulness of detailed FTIR band assignment for distinguishing genuine framework features from surface artefacts in synthetic zeolite characterization.*

Keywords: *NaP zeolite; GIS framework; FTIR spectroscopy; band assignment;*

surface contamination; aluminosilicate framework.

INTRODUCTION

Fourier-transform infrared spectroscopy (FTIR) is a widely used technique for characterizing the structure of zeolites and for identifying structural features that influence their functional properties [1, 2]. While the overall spectral pattern of a zeolite confirms its general phase, detailed band-by-band assignment provides additional information on the nature of the aluminosilicate framework and can reveal the presence of extraneous, non-structural species on the sample surface [3, 4].

For NaP zeolite (GIS framework type), reliable distinction between framework-related vibrations and bands arising from surface contamination is particularly important, since several bands in the mid-infrared region can, in principle, be attributed to more than one origin. Careful comparison of band positions, relative intensities and characteristic band groupings is therefore required to assign each feature correctly [3-5].

The aim of this study was to perform a detailed assignment of the FTIR bands of hydrothermally synthesized NaP zeolite, with particular emphasis on distinguishing the bands corresponding to the tetrahedral aluminosilicate framework from those attributable to trace organic contamination on the sample surface.

METHODOLOGY AND MATERIALS

A hydrothermally synthesized NaP zeolite sample, obtained from pure chemical reagents (sodium silicate and sodium aluminate) at a molar ratio of SiO_2:Al_2O_3:Na_2O = 2.0:1.0:3.0, was used as the investigated material. The synthesis procedure-including gel formation and hydrothermal crystallization at 100 °C for 48 hours-was described in detail in a previous study [6]. The present work focused on the detailed analysis of the FTIR band positions and their structural assignment.

Infrared spectra were recorded directly on the powdered sample by the attenuated total

reflectance (ATR) technique, without KBr pellet preparation, on a Bruker spectrometer in the 4000-400 cm^{-1} wavenumber range in transmittance mode. The recorded spectrum was used to determine the position of each absorption band, which was then assigned to a specific type of vibration on the basis of literature data for aluminosilicate zeolites.

RESULTS AND DISCUSSION

The detailed band assignment of the FTIR spectrum of the synthesized NaP zeolite is summarized in Table 1. The broad, intense band with a maximum at 3444 cm^{-1} is due to the O-H stretching vibrations of both structural silanol groups (Si-OH) and water molecules coordinated with the exchangeable Na^+ cations located within the zeolite channels. The considerable width of the band indicates the presence of hydrogen bonds of varying strength, typical of zeolitic materials with a well-developed porous structure [7, 8].

A particularly informative group of bands is observed at 2923, 2854, 1448 and 1405 cm^{-1}. Rather than being treated as isolated features, these four bands were analyzed jointly and assigned to a single source: the asymmetric and symmetric stretching vibrations of C-H bonds (2923 and 2854 cm^{-1}) together with the corresponding bending (scissoring) vibrations of CH_2 groups (1448 and 1405 cm^{-1}). The joint occurrence and consistent relative intensities of all four bands indicate trace organic contamination on the sample surface rather than a structural feature of the zeolite itself, since such vibrations are not characteristic of a pure NaP aluminosilicate framework.

Table 1. Assignment of the main FTIR bands of the synthesized NaP zeolite.

Band, cm^{-1}	**Assignment**
3444	ν(O-H) of silanol groups and adsorbed water
2923, 2854	ν(C-H) asym./sym. - trace organic contamination
1638	δ(H-O-H) of adsorbed water
1448, 1405	δ(CH_2) - trace organic contamination

Band, cm^{-1}	Assignment
991	ν asym. Si-O(Al) framework (most intense)
700	Vibrations of secondary building units (double rings)
608	δ(Si-O-Si / Si-O-Al)
455	δ(T-O-T), T = Si, Al

The band at 1638 cm^{-1} corresponds to the bending vibration of adsorbed water molecules (H-O-H) located within the pores and on the surface of the crystallites, consistent with the hydrated formula $NaAlSi_2O_6 \cdot H_2O$.

The most diagnostically significant band, at 991 cm^{-1}, represents the asymmetric stretching vibration of Si-O(Al) bonds within the tetrahedral framework. Its high intensity and comparatively narrow shape indicate the formation of an ordered crystalline framework, while its position near 990-1000 cm^{-1}, rather than in the 1050-1090 cm^{-1} region characteristic of zeolites with a higher Si/Al ratio, is consistent with the relatively low SiO_2/Al_2O_3 modulus used in the synthesis (2.43).

The bands at 700 and 608 cm^{-1} were assigned to vibrations of secondary building units (double rings) and bending vibrations of Si-O-Si / Si-O-Al bonds, reflecting the manner in which the tetrahedra are linked within the framework, while the band at 455 cm^{-1} was attributed to the bending vibration of T-O-T bonds, characteristic of intra-tetrahedral vibrations irrespective of the zeolite framework type.

Overall, the detailed band assignment demonstrates that six of the ten identified bands (3444, 1638, 991, 700, 608 and 455 cm^{-1}) can be confidently attributed to the aluminosilicate framework and adsorbed water, whereas the remaining four bands (2923, 2854, 1448 and 1405 cm^{-1}) are best explained as a single, internally consistent set of vibrations arising from trace surface contamination. This distinction is important for the correct structural interpretation of FTIR spectra of synthetic zeolites and helps to avoid over-interpretation of non-framework spectral features.

CONCLUSION

Detailed band-by-band assignment of the FTIR spectrum of hydrothermally synthesized NaP zeolite made it possible to distinguish framework-related vibrations from bands arising from trace surface contamination. The bands at 3444, 1638, 991, 700, 608 and 455 cm^{-1} were assigned to hydroxyl groups, adsorbed water and the tetrahedral aluminosilicate framework, confirming the formation of an ordered crystalline structure typical of the GIS framework type.

The bands at 2923, 2854, 1448 and 1405 cm^{-1} were shown to represent a single, internally consistent set of CH_2 vibrations attributable to trace organic contamination rather than to the zeolite structure. This approach to detailed FTIR band assignment provides a more reliable basis for the structural characterization of synthetic zeolites than a purely qualitative overview of the spectrum.

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