CRYSTAL STRUCTURE AND PHASE PURITY OF HYDROTHERMALLY SYNTHESIZED NAL ZEOLITE: AN X-RAY DIFFRACTION STUDY

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Abstract: ***This study investigates the crystal structure and phase purity of hydrothermally synthesized NaL zeolite using X-ray diffraction (XRD). The diffraction pattern was analyzed using monochromated CuKα radiation over the 2θ range of 3–60°. The observed reflections correspond to the characteristic LTL framework structure. The absence of additional diffraction maxima indicates a high phase purity of the synthesized material. Good agreement between the experimental and calculated diffraction patterns confirms the reliability of the applied profile analysis. The results demonstrate that the obtained NaL zeolite possesses a well-developed crystalline structure and a high degree of crystallinity, confirming the suitability of XRD for structural characterization of LTL-type zeolites.***

Keywords: ***NaL zeolite; LTL framework; X-ray diffraction; crystal structure; phase purity; crystallinity; hydrothermal synthesis.***

INTRODUCTION

Zeolites are crystalline microporous aluminosilicates widely used as adsorbents, molecular sieves, ion exchangers, and catalysts due to their well-defined pore systems, high thermal and chemical stability, and structural selectivity [1-3]. Among synthetic zeolites, NaL zeolite, belonging to the LTL framework type, is of particular interest because of its one-dimensional system of large twelve-membered-ring channels, which provides favorable conditions for molecular diffusion and selective adsorption [4-6].

The structural characteristics and phase purity of NaL zeolite strongly influence its functional properties. X-ray diffraction (XRD) is therefore an important method for identifying the crystalline phase, evaluating crystallinity, and detecting possible secondary phases. In this study, the crystal structure and phase purity of hydrothermally synthesized NaL zeolite were investigated by XRD, with particular attention to the characteristic reflections of the LTL framework and the agreement between experimental and calculated diffraction patterns.

METHODOLOGY AND MATERIALS

A hydrothermally synthesized NaL zeolite sample obtained from chemically pure reagents was used in this study. The synthesis procedure was described in detail in a previously published work [14]. Therefore, the present study focused on the structural characterization of the obtained material by X-ray diffraction.

X-ray diffraction analysis was performed using an automated powder diffractometer with monochromated CuKα radiation (λ = 1.5406 Å). The diffraction pattern was recorded over the 2θ range of 3–60° with a step size of 0.02°, at an accelerating voltage of 40 kV and an X-ray tube current of 15 mA.

The experimental diffraction data were processed using X'Pert HighScore Plus software. Profile analysis was applied to identify and separate individual diffraction reflections and to determine their principal parameters, including peak position (2θ), interplanar spacing (d), intensity, and FWHM. The experimental

pattern was compared with standard crystallographic data for LTL-type zeolites from the International Zeolite Association (IZA) database [4,12]. Phase purity was assessed from the positions and relative intensities of the characteristic reflections and the absence of additional peaks attributable to foreign crystalline phases.

RESULTS AND DISCUSSION

The X-ray diffraction pattern obtained for the hydrothermally synthesized NaL zeolite is presented in Figure 1. The diffraction pattern exhibits a series of well-defined and relatively intense reflections distributed mainly in the low- and medium-angle regions. The observed positions and relative intensities of the principal reflections are characteristic of the LTL framework structure, confirming the formation of the target NaL zeolite phase.

A characteristic feature of the obtained diffraction pattern is the presence of sharp and well-resolved diffraction maxima. The relatively high intensity and clear definition of the principal reflections indicate a high degree of crystallinity and a well-developed long-range order within the aluminosilicate framework. The experimental pattern is in good agreement with the characteristic diffraction features reported for LTL-type zeolites, confirming the structural identity of the synthesized material [4-6].

The profile-fitting results provide additional evidence for the reliability of the structural interpretation. As shown in Figure 1, the Observed pattern and Calculated pattern demonstrate a high degree of agreement over the investigated 2θ range. The calculated profile reproduces the principal experimental reflections with respect to their positions, shapes, and relative intensities. The Difference curve remains close to the baseline throughout most of the angular range, indicating only minor deviations between the experimental and calculated profiles. This agreement confirms that the selected profile-analysis model adequately describes the experimental diffraction data.

An important characteristic of the diffraction pattern is the absence of additional pronounced reflections that could be assigned to foreign crystalline phases. The

observed maxima can therefore be attributed predominantly to the LTL-type NaL structure. This result indicates a high degree of phase purity of the synthesized material. The absence of detectable secondary crystalline phases is particularly important because the formation of unwanted phases during hydrothermal synthesis can significantly affect the structural and functional properties of zeolitic materials.

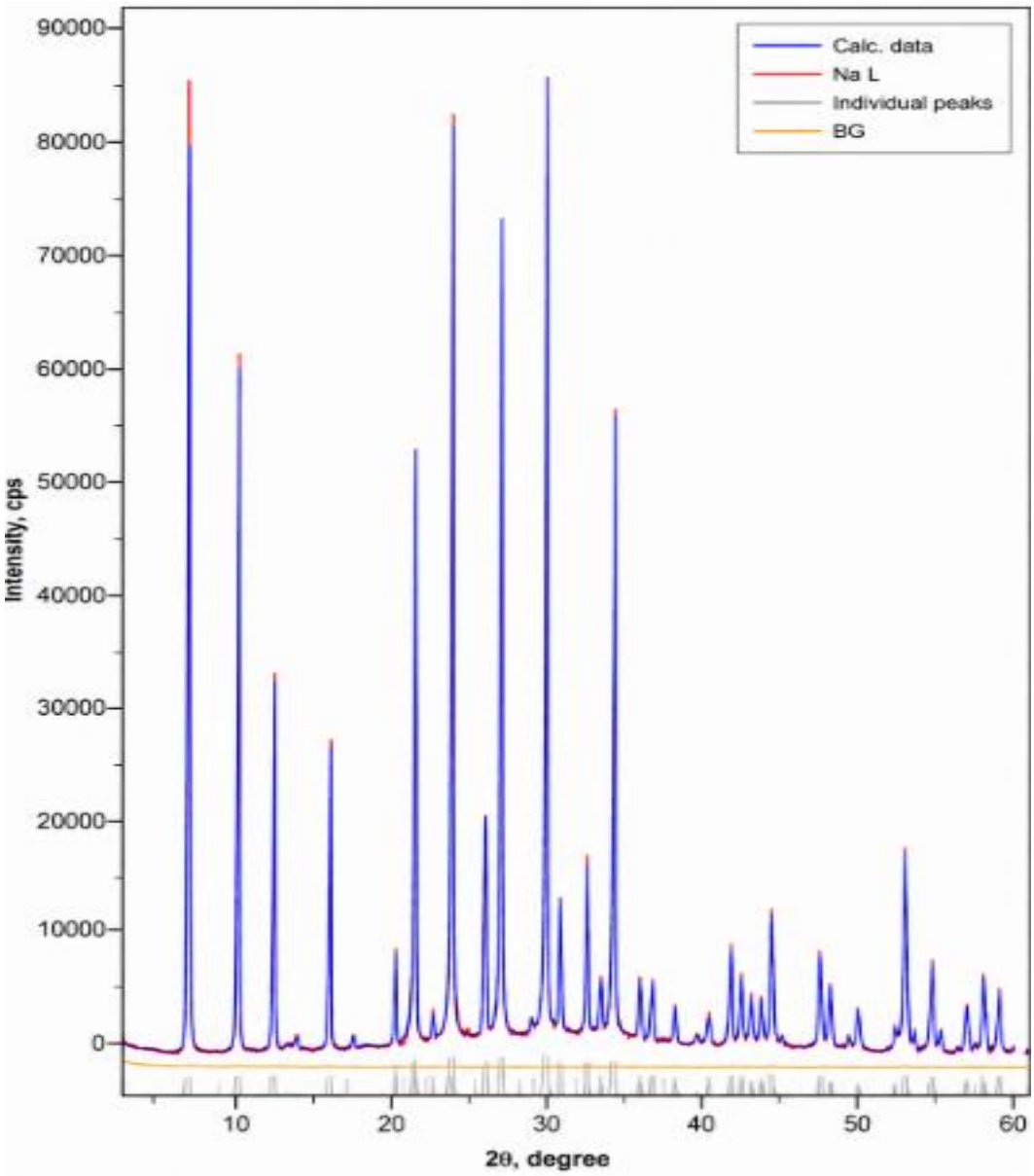


Figure 1. X-ray diffraction pattern of hydrothermally synthesized NaL zeolite with the results of profile analysis.

The diffraction pattern also demonstrates that several reflections are located relatively close to one another, particularly in the medium-angle region. Under conventional qualitative XRD analysis, such reflections may partially overlap and consequently make the interpretation of individual peaks less reliable. The application of profile analysis allows these contributions to be mathematically resolved and provides a more detailed representation of the experimental diffraction pattern. This approach is therefore useful not only for phase identification but also for obtaining quantitative characteristics of individual reflections.

Overall, the XRD results demonstrate that the hydrothermal synthesis produced a well-crystallized NaL zeolite with an LTL-type framework and high phase purity. The close agreement between the experimental and calculated diffraction profiles further confirms the reliability of the structural analysis. These findings demonstrate that XRD combined with profile fitting is an effective approach for characterizing the structural quality of synthetic NaL zeolite and provide a basis for further investigation of its adsorption, catalytic, and ion-exchange properties.

CONCLUSION

The X-ray diffraction study confirmed the successful formation of a highly crystalline NaL zeolite with an LTL-type framework structure. The experimental diffraction pattern contained the characteristic reflections of the LTL framework, while no additional pronounced diffraction maxima attributable to foreign crystalline phases were detected. This indicates a high degree of phase purity of the synthesized material.

The good agreement between the experimental and calculated diffraction patterns, together with the low deviations observed in the difference curve, demonstrates the reliability of the applied profile-fitting procedure. The well-defined

diffraction maxima also indicate a high degree of structural ordering and a well-developed crystalline framework.

Overall, the results demonstrate that X-ray diffraction combined with profile analysis is an effective approach for evaluating the structural quality and phase purity of hydrothermally synthesized NaL zeolite. The obtained structural characteristics provide a reliable basis for further investigation of the functional properties of LTL-type zeolites, particularly their adsorption, catalytic, and ion-exchange behavior.

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