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Mössbauer Study of Hydrothermal Transformation of Natural Clinoptilolite into Y and P_1 Zeolites

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The hydrothermal transformation of Na–clinoptilolite to Na–Y and Na–P_1 zeolites has been studied by Mössbauer spectroscopy of well-characterized solid intermediate phases (XRD, IR, ^{29}Si MAS NMR, and thermodielectric analysis). The evolution of the quadrupole splitting of high spin Fe^{3+} during the amorphization and crystallization processes which characterize the hydrothermal transformations indicates that octahedral sites are structure sensitive, while the tetrahedral ones fundamentally depend on the Si/Al ratio. Isomer shift values depend on Si/Al ratio in both cases. © 1991 Academic Press, Inc.

Introduction

Natural raw materials used for the hydrothermal syntheses of molecular sieves include clays (*1, 2*), volcanic glass (*3–5*), and natural zeolites (*6–8*). These processes are really promising because of the availability and low cost of the used reagents. The control of the iron content in the raw materials as well as in the reaction products is of capital importance for the practical application of these materials, due to the fact that the presence of iron could be undesirable in the obtained product and the knowledge of its location makes it easier to manage the process. Iron can also be considered as a Mössbauer probe in zeolite reactions and it can be used as an analytical tool for several studies (*9*).

The information obtained about the state of iron in natural zeolites from Mössbauer and related experiments (*10*) makes possible the use of Mössbauer spectroscopy to follow the kinetics of the hydrothermal syntheses when these minerals are used as raw materials.

A recent paper (*11*) reported the kinetics of recrystallization of Na–clinoptilolite to Na–Y and Na–P_1 zeolites by hydrothermal treatment with NaOH. In the present paper, using Fe^{3+} as a useful Mössbauer probe in well-characterized samples, we show the applicability of Mössbauer spectrometry as a complementary method of study of zeolite synthesis in cases where iron is included in the synthesis gel.

Experimental

Mössbauer spectra were obtained in a Wissell constant acceleration spectrometer in transmission geometry, with ^{57}Co in an Rh source. Iron surface density was less than 15 mg/cm^2. REM computer program (*12*), based on Gauss–Newton methods,

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