Facile and fast determination of Si/Al ratio of zeolites using FTIR spectroscopy technique

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ABSTRACT: A facile quantitative determination of the Si/Al ratio (SAR) in zeolite frameworks using IR spectroscopy is re- ported. From the linear regression analysis, the SAR for the medium-to-high Al content zeolites (1 < SAR < 5) correlates linearly to the corresponding intensity of the IR band at wavenumbers (950-1090 cm^{-1}). This model has been validated with 19 zeolites with different SAR and different framework topologies (EDI, ABW, EMT, MER, LTL, LTJ, MOR, FAU, SOD, HEU, BRE, OFF, GIS, *BEA, MFI). The quantitative determination of the SAR of zeolites by IR spectroscopy is shown to be an alternative technique to the conventional ICP-OES and XRF spectroscopy techniques.

1. Introduction

Zeolites are very important and useful industrial materials used in adsorption, separation and catalysis due to their various micropore sizes and unique framework properties [1–3]. Besides the structural proper- ties, the Al content in the framework or so-called Si/Al ratio (SAR) is of great importance and governs their applications [4,5].

In general, the most common analytical techniques for the identifi- cation of Al content in zeolites are X-ray fluorescence spectroscopy (XRF), atomic absorption spectroscopy (AAS), inductively coupled plasma atomic emission spectroscopy (ICP-OES), X-ray diffraction (XRD) and solid state Si nuclear magnetic resonance spectroscopy (Si NMR). However, these analytical tools face some limitations particularly in sample preparation workout, measurement time dura- tion, post-analytical interpretation treatment, etc. (Table 1). Therefore, the development of fast, simple and reliable method for quantitatively determining the SAR in the zeolite framework is highly desired.

Fourier-transform infrared spectroscopy (IR) is often used to study zeolites with emphasis on the functional groups, structural features, secondary building units (SBUs), pore openings and surface particular- ities. In the mid IR region (200–1300 cm), the vibrations associated with the framework structure are presented. In past few decades, several reports dealing with the use of IR spectroscopy for evaluation of zeolite particularities were published. The pioneer and iconic work was re- ported by Flanigen et al. [6]. Other studies presenting detailed and systematic spectroscopy studies on various aluminosilicate zeolites were also published [7–9]. They found out that the positions of certain IR bands at 970-1020 cm^{-1}, 670-725 cm^{-1}, 565-580 cm^{-1} and 360-385 cm^{-1} have a linearity with the Al content in faujasite type zeolites [7]. Since then, Lohse et al. reported on the use of the IR band at 748-837 cm^{-1} to calculate the SAR of cubic and hexagonal faujasite zeolites [8]. However, the validity of IR spectroscopy method for identifying the SAR is only limited to faujasites. Hence, the development of theoretical cal- culations using IR spectroscopy to determine the SAR in various zeolite frameworks is still not yet developed and remains unclear.

In this communication, we present the IR spectroscopy protocol to- wards the determination of the SAR of zeolites; this protocol was developed based on 19 zeolites subjected to comprehensive characterization with the classical chemical analyses (ICP and XRF) and NMR spectroscopy technique.

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