Caractérisation in situ de zéolithes par couplage LIBS - spectroscopie infranrouge:vers la quantification de l'hydrogène

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Abstract

Hydrogen storage is a major issue for the energy transition. One of the possible ways to achieve such storage efficiently is to exploit the properties of zeolites. Storage capacities must be verifiable using validated techniques. In this thesis, we tested the consistency of the results on this storage capacity obtained by infrared spectroscopy (IR) and by LIBS (Laser-Induced Breakdown Spectroscopy). Rather than directly testing H2 storage, which presents significant experimental difficulties, we have instead chosen to store hydrogen in zeolites by adsorbing hydrogenated molecules such as pyridine (C5H5N), ammonia (NH3) and water (H2O). LIBS is a low-destructive characterization technique based on the properties of a thermal plasma produced by laser pulse. IR spectroscopy is a widely used technique for the characterization of surface sites in zeolites. Experimentally, we have developed a LIBS-IR platform for coupling the LIBS and IR spectroscopy in situ. Using IR spectroscopy, we quantified the Lewis and Bronsted sites of the zeolites MFI (Mobil-FIve) and FAU (FAUjascites). With the picosecond LIBS, we measured the Si/Al ratio of unactivated FAU zeolite. With the nanosecond LIBS, we measured the Si/Al ratios of the unactivated, activated and/or pyridine, ammonia or water zeolite FAU. Taking into account the uncertainties, the results obtained are consistent with the results given by the NMR and the manufacturer (UOP). With LIBS and IR spectroscopy, we measured the H/Al ratios and total H concentrations in FAU zeolite activated and/or charged in hydrogenated molecules. Taking into account uncertainties, the H/Al ratios and H concentrations determined by IR spectroscopy and LIBS are close for activated FAU zeolite and pyridine-charged activated FAU zeolite. For the FAU zeolite charged with ammonia, it seems that not all forms of ammonia are quantified by IR spectroscopy. For the water-charged FAU zeolite, it seems that the plasma formed by LIBS desorbs the water of the pellet that may be adsorbed by the walls of the cell.