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Precise analysis of elements in silica powders by LA-ICP-MS

Istvan Halasz and Runbo Li
PQ Corporation, USA

Silica is important component of many heterogeneous catalysts. Catalysis affects ~90% of all chemical products. For elemental analysis by AA or ICP these silica-based catalysts are dissolved by HF and other acids. HF is exceptionally dangerous because of its ability to diffuse through the skin where the fluoride ion binds calcium with subsequent disruption of electrical activity. Therefore, it is desirable to minimize its use to reduce risk and eliminate a lengthy and tedious dissolution process. We speculated that coupling laser ablation (LA) to an ICP-MS could fulfill this desire. However, LA cannot be used for powder samples and LA-ICP-MS is known to be much less accurate than liquid phase measurements, owing to inhomogeneous ablated particle size and inadequate analysis parameters. Here, we report the development of a LA-ICP-MS method for accurately analyzing powdered silica supported catalysts. We melt the powders with a mixture of Li₂B₄O₇ - LiBO₂ into a homogeneous solid bead, vaporize the surface with a laser, and then apply a small cyclone before the ICP. Moreover, we optimized the analysis conditions by using statistical experimental design of 11 parameters. Using three commercial zeolite catalysts having Si/Al ratios 2.6, 40, and 140, we show that different parameters significantly affect the accuracy of measuring their Al contents. The relative standard deviation, RSD, remains <5% over the entire concentration range tested, sometimes even <0.5%, which is better than that obtained by the HF dissolution technique.

Biography

Istvan Halasz obtained his PhD in Hungary from the Hungarian Hydrocarbon Institute, where he developed and scaled-up efficient processes for pharmaceutical, fine chemical and petrochemical industries. Later, at the Hungarian Academy of Sciences and at USA Universities, he studied zeolite catalysis, oxide superconductor synthesis, and catalytic fume abatement for automobile exhausts. In the past 16 years, he has investigated the properties of silicates at PQ R&D. He chaired the Philadelphia Catalysis Club; is current President of North-East Corridor Zeolite Association (NECZA); edited a book on catalysis, and authored 110+ book chapters and papers, 7 patents, and 80+ conference presentations. Runbo Li obtained her PhD in Analytical Chemistry from Drexel University in USA. In her thesis, she studied different methods for preparing samples for analysis by MALDI TOFMS and applied these methods to quantify proteins. At PQ R&D, she has focused on analytical method development and characterization research related to silicates, glass beads, amorphous silica gel and zeolites. She has published 18 papers.

istvan.halasz@pqcorp.com

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