High Throughput Analysis of Small Quantity of Catalyst by WDXRF

Magalie Fontaine, Yannis Toe, Charles-Philippe Lienemann, Laboratoire AAS-ICP-FX, Direction Physique et Analyse, IFP News Energy, F-69360 Solaize, tel: 33-(0)4.3770.2726, fax: 33-(0)4.3770.2745, email: charles.lienemann@ifpen.fr

Justin Strangeways, FLSmidth A/S, Vigerslev Allé 77, DK-2500 Valby, tel: 45-36181000, fax: 45-36301820

Direct analysis of solid sample is an important challenge in elemental analysis. Many industry are facing this and various analytical solution are proposed. The use of Inductively Coupled Plasma coupled to optical (ICP-OES) or mass spectrometry (ICP/MS) detection is routinely used, but the mineralization of the solid is time and reagent consuming as the digestion step is required for solution analysis. Direct laser ablation before ICP or Laser Induced Breakdown Spectroscopy (LIBS) is also proposed, but routine analysis is not commonly used within the industry.

Both Energy Dispersive (EDXRF) and Wavelength Dispersive X-ray fluorescence (WDXRF) are used for the characterization of catalyst. Different approaches are possible with direct analysis on raw powder issued from the crushing of the initial catalyst or analysis of a pressed pellet made with the catalyst. The fusion of the catalyst is also possible to reduce matrix interferences and the choice of the preparation step is most of the time determined by the element that are measured or the available amount of the catalyst.

The market is proposing since many years automatic system able to do the preparation of sample (fusion, pressed pellet), but these system were mainly present in the cement or mineral industry only.

In our case, the analytical methods should be able to measure some difficult element (Na, S, Cl, Pt) with a limiting amount of catalyst (< 1 g). The loss of ignition should also be taken into account and more than 15 samples per days should be analyzed. The challenge was also defined by the fact that a small amount of catalyst was available with the need to fuse refractory and volatile element within the same run.

The proposed solution is based on an automatic fusion machine (weighing, fusing and cleaning) coupled to a WDXRF instrument with a robot as an interface. The challenge was to incorporate an oven and a desiccator as an initial step to get dry samples before the automatic weighing of the catalyst. Different fusion program were developed to keep volatile element (Cl, S) and allow the analysis of light element (Na, Al and Si) with the same approach.

A prototype of the machine was developed and successfully installed within a lab dedicated to high throughput synthesis and analysis of catalyst. This prototype and the validation step will be presented during the conference.