SEM-EDS: A Faster and Convenient Tool for Complete Elemental Profiling of Refinery Hydroprocessing Catalysts

Vijai S. Balachandran[1], Amit A. Deshmukh[2], Satyanarayana Reddy Akuri[2]
Vidyasagar Guggilla[2], Jose Nehamiah[2], Rajeshwer Dongara[2], Vishnupriya Bhaktavatsalam[1], and Philip Angevine[1]

[1.] Advanced Analytical Sciences, Reliance Industries Limited, Navi Mumbai 400701, India.
[2.] Fixed Bed Catalyst Processing & Scale-up, Reliance Industries Limited, Navi Mumbai, India.

Hyroprocessing in a refinery involves Hydrodemetallation (HDM), Hydrodesulfurization (HDS) and Hydrodenitrogenation (HDN) processes. HDM involves hydrotreating of the feed in a series of de-metallation catalyst in a fixed bed reactor to remove the metals, thereby, ensuring the proper quality of feed for downstream refinery units. HDM is a critical process that dictates the catalyst deactivation due to metal entrapment and coke formation. Complete and fast elemental mapping and distribution in the catalyst bed is imperative for understanding the performance and life cycle of the Hydrotreating reactor. Conventional elemental analyses techniques, in general, require longer sample preparation protocols and harsh wet chemical methods for quantitative & qualitative estimation of elements. Evaluation of lighter elements such as C, H, N, S and O require separate instrumental dependency. We have mapped the complete elemental profiling of Hydroprocessing catalysts using SEM-EDS efficiently for extrudates located at different bed layers. Concomitantly, SEM-EDS technique provided us the spatial distribution of entrapped impurities in each extrudate.

The samples were non-invasively fractured carefully for cross sectional elemental mapping and mounted perpendicular to the beam axis on a carbon coated adhesive. The surfaces were checked under optical microscope for any imperfections and only the neatly fractured samples were considered for elemental mapping. Images were acquired with SE and BSE modes and further subjected to EDS analyses using SDD array detector (EDAX-TEAM OCTANE PLUS). The homogeneity of the X-ray signals collected were ensured from the X-ray image generated on the sample and quantifications were done by eZAF method. Distribution of various metal impurities across the catalyst exturdates were analysed by line scanning measurements in addition to complete elemental mapping of the cross section.

Various samples from different levels of the beds were examined. The results of these studies depict the clear mapping on the extent of impurities trapped by the catalysts across the reactor height. The spatial distributions of metals on the Hydrotreating catalysts on the surface and across the cross-section using SEM-EDS provided valuable guidance to assess the life time and regeneration ability of spent catalysts or further to be proceeded for metal recovery. These results also significantly contribute in understanding the malfunctioning of upstream units by which the catalysts' performance and life cycle can be enhanced. The current study has enabled in improving the overall efficiency and economics of process, making SEM-EDS as one of the versatile tool for refinery applications.

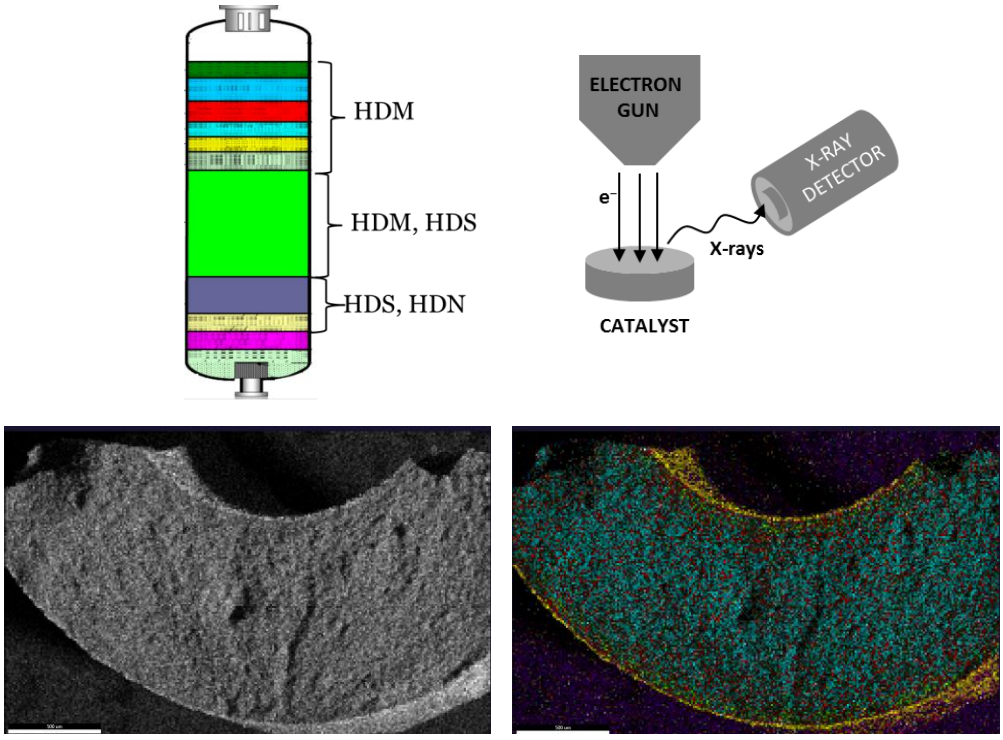

Figure 1. (TOP) Schematic representation of a Hydroprocessing reactor and the schematic of SEM-EDS. (BOTTOM) Cross sectional view of a typical HDM catalyst showing X-ray and false color elemental mapping.