Understanding solid catalysts at the atomic level*

The science of heterogeneous catalysis is a subject which is intimately intertwined with that of the fundamentals of surface science. Monitoring the atomic events occurring on catalyst surfaces therefore, is crucial for a full understanding of the working mechanism of a given catalytic reaction. This monitoring includes identification and characterization of the active centres, observation of reaction intermediates, activation processes of reactants as well as mobility of species at the catalyst surfaces. This demands probing of surfaces at the atomic level, where non-conventional powerful spectroscopic tools can play a major role. Though substantial progress has been made in this direction, a deeper insight into the atomic structure of catalyst surfaces is far more complex and still offers demanding challenges for further investigation. This motivated the Delhi Chapter of Indian Society of Analytical Scientists to organize a one-day seminar. The seminar was attended by over forty delegates and it spanned the whole range of research on spectroscopic methods of catalyst characterization.

The inaugural lecture was given by A. K. Bhatnagar (Director, R&D Centre, Indian Oil Corporation Limited, Faridabad) who explained the role of catalysts in getting more from less, especially in the petroleum refining industry. Discussing important issues for rational designing of catalysts, he presented a general view of the state of development, and application of several spectroscopic methods for the investigation of catalytic activities in zeolites and other nanostructured catalysts. He stated that re-engineering of molecules remains one of the greatest challenges before the refiners and that catalysis will play a central role in this context.

The keynote speaker for the event, B. Viswanathan (Indian Institute of Technology, Chennai) illustrated various aspects of surface heterogeneities. He emphasized the need for a much deeper knowledge of nature and action of atomic and molecular forces for a full understanding of the reaction mechanism of catalytic reactions. He also introduced the capabilities of X-ray photoelectron spectroscopy in investigating the local structure and chemistry of heterogeneous surfaces using several model systems. This technique determines the binding energy of an electron in an atom or molecule and can probe the top few nanometers of a sample from which chemical information can be obtained. He demonstrated that this method can help in understanding the relation between morphology and promotional effect of Co in $MoCo/Al_2O_3$ hydrodesulphurization catalysts.

The scientific programme consisted of six lectures in three different sessions. A. Datta (Indian Institute of Petroleum, Dehradun) provided an overview of application of several surface-sensitive techniques such as electron energy loss spectroscopy and low energy electron diffraction for characterizing the nature of adsorbates on surfaces at the submonolayer level and measuring the two-dimensional surface structure of catalysts, respectively. The lecture addressed new possibilities for obtaining atomic-scale information about active structures in refining catalysts using these methods. M. B. Patel (R&D Centre, Indian Oil Corporation Limited, Faridabad) demonstrated the use of X-ray diffraction combined with X-ray fluorescence techniques for structural elucidation of zeolytic and non-zeolytic components of a catalyst used in fluid catalytic cracking (FCC) process. He concentrated on advantages of the techniques in measuring the unit cell size, percentage crystallinity and Si/Al ratio of zeolites used in assembling FCC catalysts.

In the second session, a journey into the amazing world of atomic imaging was provided by Prasenjit Sen (Jawaharlal Nehru University, New Delhi), in his lecture on catalysis with atomic force microscope. This method has developed as one of the most powerful tools to determine the array and orientation of the adsorbed molecules on zeolite surfaces. Citing several case studies such as coking of selective oxidation catalysts and photoreduction of Ag^+ on individual TiO_2 particles, he described the working involved in bringing out the power and elegance of this technique. This lecture was followed by the presentation of A. S. Sarpal (R&D Centre, Indian Oil Corporation Limited, Faridabad) who demonstrated the key role of the use of solid state NMR method and its versatility for characterization of solid acid catalysts. He gave a general outlook on the methodology of CP-MAS(S) and 2D techniques together with examples of their application in elucidating the lattice structure, pore geometry, Bronsted acidity and movements of transition metals in zeolites.

In the third session, D. Srinivas (National Chemical Laboratory, Pune) described the role of electron paramagnetic resonance spectroscopy in heterogeneous catalysis. He highlighted important features of the technique and demonstrated that it can be used under ambient as well as catalytic conditions for identifying the active sites and reaction intermediates with very low abundance in the framework or on the surface of the catalysts. M. I. S. Sastry (R&D Centre, Indian Oil Corporation Limited, Faridabad) presented a detailed study on the usage of infrared and Raman spectroscopy in catalyst characterization. Both the techniques are useful for the assignment of Mo and W oxospecies dispersed in high surface area supports, which are used as catalysts for hydrodesulphurization reactions.

The deliberations provided the participants a feel for the current state-of-the-art of powerful spectroscopic techniques in understanding the behaviour of solid catalysts at the atomic scale.

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