

Chapter 2

Characterization Techniques

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This chapter deals with different characterization tools used in this work along with brief description of their basic principles.

2.1. X-ray Diffraction (XRD) analysis

X-ray Diffraction is a non destructive technique that provides information about the arrangement of atoms within a crystal, the crystallographic structure of the materials, thickness of thin film, estimate the size of NPs, etc. The technique is used to determine crystal structures, phase composition, crystallinity and can also be exploited for identification of semi-crystalline materials via Small Angle X-ray scattering (SAXS). As the spacing between the atomic arrays in a material is of the order of angstrom (\AA), a light of wavelength in the order of atomic distance is required to get an idea about these arrays. The wavelength of X-rays is in the order of 0.1-100 \AA . So, a diffraction pattern is produced when X-rays interact with a crystalline substance (irrespective of nature of crystallinity of materials). According to the Bragg's equation:¹

$$2d \sin\theta = n\lambda$$

Where, ' θ ' is the Bragg's angle, ' λ ' is the wavelength of X-ray; ' n ' is an integer notifying order of diffraction and ' d ' is the spacing between the atom planes in the lattice. Comparing this data with the standard JCPDS database, structure and planes are determined. XRD study was carried out using PW 1710, Philips, Holland using Co target with K_{α} radiation ($\lambda = 1.79 \text{\AA}$) or with Rigaku X-ray diffractometer, using Cu K_{α} radiation ($\lambda = 1.54 \text{\AA}$) or Co K_{α} radiation ($\lambda = 1.79 \text{\AA}$).

2.2. Scanning Electron Microscopy (SEM) analysis

Scanning electron microscope (SEM) reveals topographical information of the materials. Secondary electrons (SE), back-scattered electrons (BSE), characteristic X-rays and specimen current are the usual types of signals produced by a SEM. These electrons are collected by a source and digitally processed to form image. This technique is extensively used for study of morphology, particle distribution and surface roughness. In our work, the morphology of the samples was studied using Carl Zeiss and JSM-6390LV SEM instrument.

2.3. Field Emission Scanning Electron Microscopy (FESEM) analysis

A field-emission cathode in the electron gun of a SEM produces narrow probing beams with high electron energy, resulting in both spatial resolution and low sample charging. This will make less damage to the sample compared to same fact happened in SEM analysis. Basically, it is somewhat different from normal SEM by the electron source, where, a field emission cathode in the electron gun of a SEM is used instead of traditional thermionic source. In this work, FESEM was carried out on a Carl Zeiss Supra 40 instrument.

2.4. Transmission Electron Microscopy (TEM) analysis

Transmission electron microscope (TEM) is a very useful tool for imaging the nanomaterials, where high voltage electrons are accelerated to pass through a sample and create a image of the same. The dispersed samples in acetone or ethanol had been drop casted on carbon coated copper grid and properly placed under the electron beam. TEM images were recorded here using Phillips CM 200 and JEOL, Model: JEM 2100.

2.5. Energy Dispersive X-ray (EDX) analysis

This is an excellent tool for characterization of materials and it gives an insight of around 2μ depths with elemental information. This is usually connected to an electron microscope (SEM/FESEM/TEM) and as the electron beam penetrates through the material, resulting formation of a hole by ejection of an electron. Subsequently, an electron from the higher energy shell fall down with the release of the extra energy in form of X-ray. These spectral lines are highly element specific and those are analyzed to get the information about composition of the material.

2.6. Fourier Transform -Infra Red (FT-IR) analysis

FT-IR is an effective tool for identification of functional groups based on vibrational spectroscopy of the bonds. The sample of interest is first mixed with KBr and compressed into a pellet. The spectra were recorded on a Perkin Elmer FT-IR and Nicolet (Impact 410) FT-IR spectrophotometer analyzer.

2.7. Brunauer–Emmett–Teller (BET) surface area analysis

Brunauer–Emmett–Teller (BET) theory² aims to explain adsorption of gas molecules on a solid surface and provides the basis for the measurement of the specific surface area of a material. The BET theory refers to multi layer adsorption and generally, non-corrosive gases like nitrogen, argon, carbon dioxide etc. are used as adsorbates to determine the surface area of specific samples. In our case, it was measured on Beckman Coulter Instruments in the presence of nitrogen gas.

2.8. Atomic Absorption Spectroscopy (AAS) analysis

Atomic absorption spectroscopy (AAS) is an analytical technique for the quantitative identification of chemical elements using the absorption of light of specific wavelength. In analytical chemistry, the technique is used for determination of the concentration of a particular element (the analyte) in a sample. AAS can be exploited to determine over 70 different elements in solution or in solid samples used in chemistry, pharmacology and toxicology research. We have used Atomic Absorption Spectrophotometer (Model AA 7000, Lab India Pvt. Ltd, India and Perkin Elmer, AAnalyst 200) to carry out the elemental analysis of the samples.

2.9. Nuclear Magnetic Resonance (NMR) Spectroscopy analysis

Nuclear magnetic resonance (NMR) spectroscopy is a research technique that exploits the magnetic properties of certain atomic nuclei. It is a phenomenon in which nuclei in a magnetic field absorb and emit electromagnetic radiation. It determines the physical and chemical properties of atoms or the molecules in which they are contained. It can provide detailed information about the structure, reaction state and chemical environment of molecules. The intramolecular magnetic field around an atom in a molecule affects the resonance frequency and provides opportunity to access the details of the electronic structure of a molecule. In our case, ¹H and ¹³C NMR spectra were recorded in both 300 MHz NMR spectrophotometer (Bruker, ADVANCE) and 400 MHz spectrophotometer (JEOL, JNM ECS) in CDCl₃ using tetramethylsilane (TMS) as the internal standard and coupling constants are expressed in Hertz. NMR of some of the compounds had been also recorded with 600 MHz NMR spectrophotometer (Make: Bruker, Model: D8- Advance).

References:

1. Cullity, B.D. *Elements of X-ray diffraction*. Addison Wesley Pub. Co. Inc., Reading Massachusetts, 1978.
2. Brunauer, S., et al. *J. Am. Chem. Soc.* **60** (2), 309--319, 1938.