International Journal of Engineering, Science and Mathematics
Vol. 7 Issue1, January2018, ISSN: 2320-0294 Impact Factor: 6.765
Journal Homepage: http://www.ijesm.co.in, Email: ijesmj@gmail.com
Double-Blind Peer Reviewed Refereed Open Access International Journal - Included in the International Serial Directories Indexed & Listed at: Ulrich's Periodicals Directory ©, U.S.A., Open J-Gage as well as in Cabell's Directories of Publishing Opportunities, U.S.A

A review on Characterization techniques of Nanomaterials

Sathish Boddolla,
Assistant professor of Physics,
Sreyas Institute of Engineering & Technology
Nagole, Hyderabad, Telangana, India.

Satyanarayana thodeti
Assistant professor of Physics,
Sreyas Institute of Engineering &Technology
Nagole, Hyderabad, Telangana, India.

Absract:

The characterization of nano materials is important for understanding their properties and applications. This review describes the instruments and experimental set ups utilized for various measurements towards the characterization of the synthesized nanocrystals. The techniques adopted to characterize the nanoparticles are: X -ray diffraction(XRD),SEM,EDX,TEM,DC-Conductivity, Particle Size analyser(PSA), UV - Visible Spectroscopy, Thermo Gravimetric Analysis/Differential Thermal Analyzer (TG/DTA)

Keywords: Nanomaterials,XRD,SEM,EDX,TEM,DC,PSA,UV,TG/DTA

1.INTRODUCTION:

Nano scale science and technology is a young and burgeoning field that encompasses nearly every discipline of science and engineering [14]. With rapid advances in areas such as molecular electronics, synthetic bio molecular motors, DNA-based self-assembly, and manipulation of individual atoms via a scanning tunnelling microscope, nanotechnology has become the principal focus of a growing cadre of scientists and engineers and has captured the attention and imagination of the general public [3]. This field is defined primarily by a unit of length, the nanometre at which lies the ultimate control over the form and function of matter.The fundamental nanotechnology lies in the fact that properties of material change drametically when their size is reduced to the nanometer range, but measuring this nano dimension is not a very easy task. Although research is gioing on to synthesis nanostructured and nanophasic materials, characterizing these nanosized materials is also an emerging field posing lot of challenges to scientists and technologists. Thus nanotechnology has motivated the upsurge in research activities on the discovery and invention of sophisticated nano charcterization techniques to allow a better control of morphology, size and dimensions of materials in nano range [4]. The important characterization techniques used for nanotechnology reasearch in various applications have been discussed in this paper.

2. CHARACTERIZATION TECHNIQUES:

2.1 X-RAY DIFFRACTION (XRD):

X-ray diffraction (XRD) is a non-destructive type of analytical technique which provides valuable insight about the lattice structure of a crystalline substance like unit cell dimensions, bond angles, chemical composition and crystallographic structure of natural and manufactured materials [1]. XRD is based on the principle of constructive interference of x-rays and the sample concerned which should be crystalline. The x-rays which are generated by a CRT are filtered, collimated and then directed towards the sample. The interaction that follows produces constructive interference based on Bragg's law which relates wavelength of the incident radiations to the diffraction angle and lattice spacing.

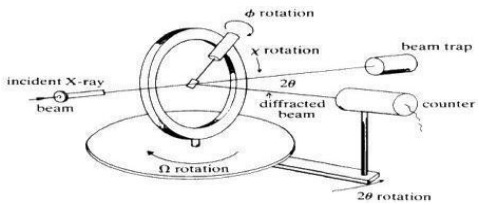

Figure.2.1. Schematic Diagram of a 4-Circle Diffract meter.

X-ray powder diffraction (XRD) is rapid analytical technique primarily used for phase identification of the crystalline material and can provide information on unit cell dimension and atomic spacing. The X-ray are generated by cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed towards the sample. The interaction of the incident monochromatic rays with the sample produces constructive interference (and diffracted ray) when condition satisfy Bragg's Law $n\lambda = 2d \sin\theta$ This equation relates the wavelength (λ) of electro-magnetic radiation to the diffraction angle (θ) and the lattice spacing (d) in a crystalline sample by scanning the sample through arrangement of 2θ angles. All the possible diffraction directions of the lattice are attained due to the random orientation of the powdered materials

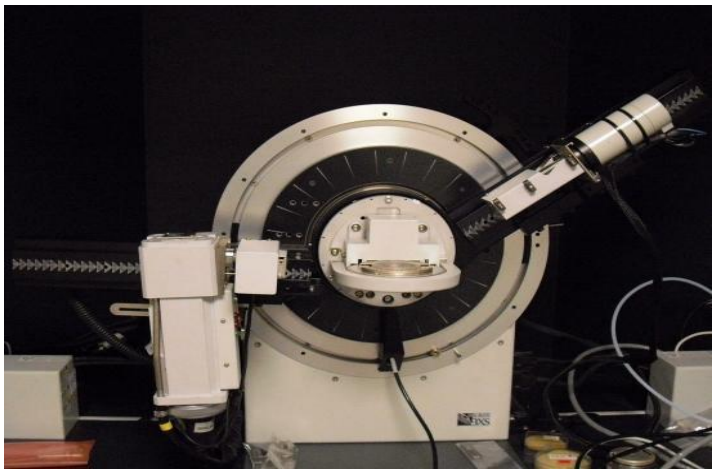

Figure 2.2.XRD-Equipment

2.2. SCANNING ELECTRON MICROSCOPY (SEM):

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons in a raster scan pattern [1], [2]. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. SEM can produce very high-resolution images of a sample surface, revealing details about less than 1 to 5 nm in size. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. Under vacuum, electrons generated by a source are accelerated in a field gradient. The beam passes through Electromagnetic Lenses, focusing onto the specimen. As result of this bombardment different types of electrons are emitted from the specimen. A detector catches the secondary electrons and an image of the sample surface is constructed by comparing the intensity of these secondary electrons to the scanning primary electron beam. Finally the image is displayed on a monitor.

Figure 2.3. SEM-Experiment set up.

In most of the applications, the data collected is over a pre selected area of the sample surface and following this, a 2D image is generated that shows the various spatial variations. Conventional SEMs with a magnification range of 20X-30000X with a spatial resolution of 50-100 nm can scan areas which vary from 1 cm to 5µm in width. SEMs also have the ability to analyze particular points as can be seen during EDX operations which help in determining the chemical composition of the sample concerned.

2.3. ENERGY DISPERSIVE X-RAY (EDX):

Energy dispersive X-ray spectroscopy (EDS or EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It is one of the variants of X-ray fluorescence spectroscopy which relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing X-rays emitted by the matter in response by hitting the charged particles [1], [2]. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing X-rays that are characteristic of an element's atomic structure to be identified uniquely from one another. To stimulate the emission of characteristic X-rays from a specimen,

a high-energy beam of charged particles such as electrons or protons or a beam of X-rays is focused onto the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted. This allows the elemental composition of the specimen to be measured.

2.4. TRANSMISSION ELECTRON MICROSCOPE (TEM):

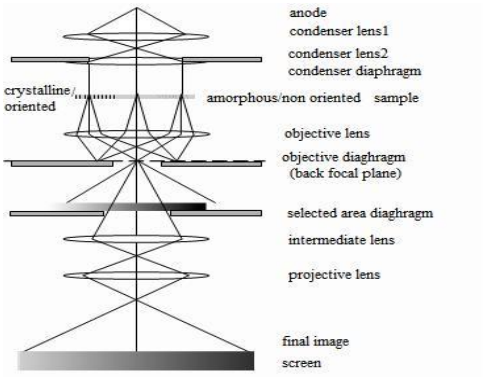

Figure.2.4.Ray diagram of TEM.

A transmission electron microscope is constituted of: (1) two or three condenser lenses to focus the electron beam on the sample, (2) an objective lens to form the diffraction in the back focal plane and the image of the sample in the image plane, (3) some intermediate lenses to magnify the image or the diffraction pattern on the screen. If the sample is thin (< 200 nm) and constituted of light chemical elements, the image presents a very low contrast when it is focused. To obtain amplitude contrasted image, an objective diaphragm is inserted in the back focal plane to select the transmitted beam (and possibly few diffracted beam): the crystalline parts in Bragg orientation appear dark and the amorphous or not Bragg oriented parts appear bright. This imaging mode is called bright field mode (BF).In diffraction mode, other intermediate lens is inserted to image on the screen the diffraction pattern of the back focal plane. If the diffraction is constituted by many diffracting phases, each of them can be differentiated by selecting one of its diffracted beams with the objective diaphragm. To do that, the incident beam must be tilted so that the diffracted beam is put on the objective lens axis to avoid off- axis aberrations .This mode is called dark field mode DF. The BF and DF modes are used for imaging materials to nanometer scale. SAED and micro diffraction patterns of a crystal permit to obtain the symmetry of its lattice and calculate its interplanar distances (with the Bragg law). This is useful to confirm the identification of a phase, after assumptions generally based on the literature of the studied system and on chemical analyses.

2.5. DC CONDUCTIVITY:

Electrical resistivity (also known as resistivity, specific electrical resistance, or volume resistivity) quantifies how strongly a given material opposes the flow of electric current. A low resistivity indicates a material that readily allows the movement of electric charge [1]. Resistivity is commonly represented by the Greek letter ρ (rho).

The SI unit of electrical resistivity is the ohm·metre (Ω·m) although other units like ohm·centimetre (Ω·cm) are also in use. As an example, if a 1 m × 1 m × 1 m solid cube of material has sheet contacts on two opposite faces, and the resistance between these contacts is 1 Ω, then the resistivity of the material is 1 Ω·m.

Electrical conductivity or specific conductance is the reciprocal of electrical resistivity, and measures a material's ability to conduct an electric current. It is commonly represented by the Greek letter σ (sigma), Its SI unit is Siemens per meter (S/m) and CGSE unit is reciprocal second (s−1).

The temperature (T) and corresponding resistance(R) values are taken from the experiment and further the resistivity and conductivity is measured from the following formulas.

The electrical resistivity (*ρ)* is defined as:

$$\rho = RA/l$$

Where *R* is the electrical resistance of sample (measured in ohms, Ω)

A is the area of the specimen (measured in square meters, m^2).

l is the thickness of the pellet(measured in meters, m)

Conductivity (*σ)* is defined as the inverse of resistivity and it is defined as

$$\sigma = 1/\rho$$

Conductivity has SI units of Siemens per meter (S/m)

2.6. PARTICLE SIZE ANALYSER (PSA):

The technique of PSA is ideally suited for the determination of the size of particles in the nanometer size range. The Malvern Zetasizer Nano Series uses patented optics that provides exceptional levels of sensitivity and allows the determination of the size of samples that contain very small particles and/or particles that are present at very low concentrations [2]. In addition, the backscatter optics allows for the measurement of samples at much higher concentrations than is possible using conventional DLS instruments using a 90° detection angle

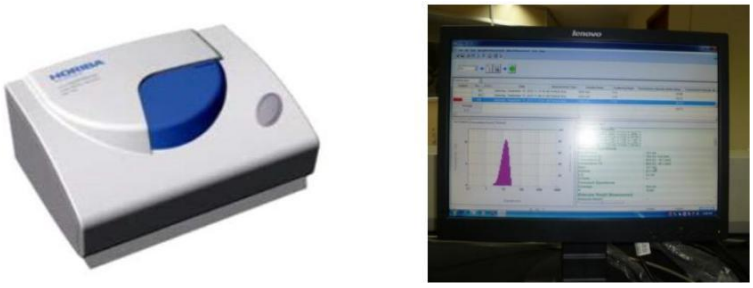

Figure 2.6.Particle size analyzer

Particle size is important parameters for the characterization of nanoparticles. The technique of dynamic light scattering is well suited to the measurement of the size of nanoparticles dispersions. Conventionally, measurement of very small and or poorly scattering particles or samples that are very dilute were difficult unless high powered lasers were used.

2.7. ULTRAVIOLET-VISIBLE SPECTROSCOPY:

Ultraviolet-visible spectroscopy or ultraviolet-visible spectrophotometer (UV- Vis) involves the spectroscopy of photons in the UV-Visible region. It uses light in the visible and adjacent near ultraviolet (UV) and near infrared (NIR) ranges. In this region of the electromagnetic spectrum, molecules undergo electronic transitions. UV-Vis Spectrophotometers are mainly used to measure transmission or absorption in liquids and transparent or opaque solids. It does so by sending a beam of light through the sample and then monitoring the remaining light in a detector. In the case of a UV-Vis spectrophotometer the light is in

the wavelength of 800 - 200nm, probing electronic transitions in the sample. It is hard to reach a lower wavelength than 200nm as oxygen starts to absorb light below that wavelength. When the light passes through the sample some of the molecules in the sample will absorb lights at various wavelengths of this spectrum, depending on their chemical bonds and structure. As a rule, energetically favored electron promotion will be from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO), and the resulting species is called an excited state. When sample molecules are exposed to light having an energy that matches a possible electronic transition within the molecule, some of the light energy will be absorbed as the electron is promoted to a higher energy orbital. A spectrophotometer records the wavelengths at which absorption occurs, together with the degree of absorption at each wavelength. The resulting spectrum is presented as a graph of absorbance versus wavelength.

Figure .2.7.UV-Visible double-beam spectrophotometer.

2.8. THERMO GRAVIMETRIC ANALYSIS/DIFFERENTIAL THERMAL ANALYZER (TG/DTA):

Thermo Gravimetric Analysis (TGA) is a thermal analysis technique which measures the weight change in a material as a function of temperature and time, in a controlled environment. This is very useful to investigate the thermal stability of a material, or to investigate its behaviour in different atmospheres (e.g. inert or oxidizing). It is suitable for use with all types of solid materials, including organic or inorganic materials [2]. Differential thermal analysis (DTA) is a calorimetric technique, recording the temperature and heat flow associated with thermal transitions in a material. This enables phase transitions to be determined (e.g. melting point, glass transition temperature, crystallization etc.). Thermo Gravimetric Analysis (TGA) is a type of testing performed on samples that determines changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in three measurements: weight, temperature, and temperature change. As many weight loss curves look similar, the weight loss curve may require transformation before results may be interpreted. A derivative weight loss curve can identify the point where weight loss is most apparent. Again, interpretation is limited without further modifications and de-convolution of the overlapping peaks may be required. To determine composition and purity one must take the mass of the substance in the mixture by using thermal gravimetric analysis. Thermal gravimetric analysis is the act of heating a mixture to a high enough temperature so that one of the components decomposes into a gas, which dissociates into the air. If the compounds in the mixture that remain are known, then the percentage by mass can be determined by taking the weight of what is left in the mixture and dividing it by the initial mass. Knowing the mass of the original mixture and the total mass of impurities liberating upon heating, the stoichiometric ratio can be used to calculate the percent mass of the substance in a sample. TGA is commonly employed in research and testing to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues. It is also often used to estimate the corrosion kinetics in high temperature oxidation. Simultaneous TGA-DTA/DSC measures both heat flow and weight changes (TGA) in a material as a function of temperature or time in a controlled atmosphere. The thermal analysis and weight loss of the sample are observed by S-II EXSTAR-6000, TG/DTA-6300 thermal analyzer as shown in figure 2.8

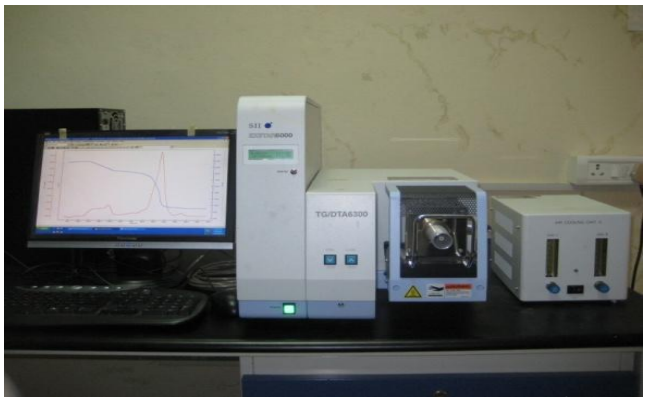

Figure 2.8.Thermo Gravimetric & Differential Thermal Analyzer.

Simultaneous measurement of these two material properties not only improves productivity but also simplifies interpretation of the results. The complementary information obtained allows differentiation between endothermic and exothermic events with no associated weight loss (e.g. melting and crystallization) and those that involve a weight loss (e.g. degradation).

III. CONCLUSION:

The basic principles of characterization techniques, such as X -ray diffraction(XRD),SEM,EDX,TEM,DC-Conductivity, Particle Size analyser(PSA), UV - Visible Spectroscopy, Thermo Gravimetric Analysis/Differential Thermal Analyzer (TG/DTA) are discussed. Nanoparticle characterization parameters include:surface area and porosity,solubility,particle size distribution, aggregation, hydrated surface analysis,zeta potential,wettebility,adsorption potential,shape and size of interactive surface.

IV. REFERENCES:

[1]. Synthesis and Characterization of Pure and Indium Doped Sno2 Nanoparticles By Sol-Gel Methods Satyanarayana Thodeti1, M.V.Ramana Reddy2, J.Siva Kumar 3- ISSN 2229-5518

[2].Synthesis And Characterization of Zno Nanostructures by Oxidation Technique Satyanarayana Thodeti1, Hima Bindu Bantikatla2, Y.Kiran Kumar3, B.Sathish4-ISSN 2319-8314

[3] Lue, Juh Tzeng, (2007), "Physical properties of nanomaterials", *Encyclopedia of nanosci. & nanotech*., Vol. 10, pp 1-46.

[4]. Nanoparticles: Properties, applications and toxicities Ibrahim Khan a, *, Khalid Saeed b , Idrees Khan c- Arabian Journal of Chemistry (2017)

[5]. synthesis of anatase nanoparticles with graphene domains. ACS Appl. Mater. Interfaces. 6, 10638–10648. http://dx.doi.org/10.1021/ am502322y

[6]. Nagarajan, P.K., Subramani, J., Suyambazhahan, S., Sathyamurthy, R., 2014. Nanofluids for solar collector applications: a review. Energy Procedia 61, 2416–2434. http://dx.doi.org/10.1016/ j.egypro.2014.12.017.

[7]. Novel development of nanoparticles to bimetallic nanoparticles and their composites: A review Gaurav Sharma a,⇑ , Amit Kumar , Shweta Sharma , Mu. Naushad ,⇑ , Ram Prakash Dwivedi , Zeid A. ALOthman, Genene Tessema Mola -Journal of King Saud University – Science direct.

[8]. the fascinating world of nanoparticle research Florian J. Heiligtag and Markus Niederberger*-Materials Today Volume 16, Numbers 7/8 July/August 2013

[9]. M.Faraday, Philos.Trans.R Soc. Lond. 147(1857)

[10]. A. Henglein, Chem. Rev. 89 (1989) 1861.

[11]. A.P. Alivisatos, J. Phys. Chem. 100 (1996) 13226.

[12]. C. Burda, et al. Chem. Rev. 105 (2005) 1025.

[13].Metal Nanoparticles: synthesis, Characterization and applications by Daniel L Fedlhem, Colby A.Foss.

[14].Textbook: Nanoparticles:Synthesis characterization and applications by R.S.Chaughule, R.V.Ramanujan.

[15].Textbook: An Introduction to Nanoparicles and Nanotechnology by Maria Benemekki.

[16].Methods Of Preparation Of Nanoparticles – A Review- Namita Rajput ISSN: 22311963

[17]. Properties, Application and Synthesis Methods of Nano-Molybdenum Powder-Pengfa Feng, Weicheng Cao- ISSN Online: 2327-6053.

[18] Pal, Sovan Lal, Pal, Utpal, Manna, P.K.,Mohanta, G.P. & Manavalan, R. , (2011) "Nanoparticle : An Overview Of Preparation And Characterization", *J. Of Pharamaceutical Sci.,* Vol. 1(6), Pp228-234.
[19] A Review of Methods for Synthesis Of Al Nanoparticles

[20] Boldyrev, V.V. & Tkacova, K., *Journal of Materials Synthesis and Processing* 2000, *8*, 121-132.
[21] Preparation of Nanomaterials-NPTEL- Dr. Pallab Ghosh
[22]. Text Book of Nanoscience and Nanotechnology by T.Prdeep-2012-Mcgraw Hill-ISBN: 9781259007323