



VITAL ROLE PLAYED BY NANOTECHNOLOGY IN CHEMISTRY

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Abstract

Inorganic nanostructures including metal oxides, ceramics and composites have gained a worldwide research attention in the past two decades and tremendous efforts have been made on their synthetic aspects, characterization and applications. Among the various classes of nanomaterials, nano metal oxides are particularly attractive from the viewpoint of scientific and technological applications. They display unique physicochemical properties due to their restricted size and thus have application potential in fields like catalysis, sensors and environmental remediation. Metals can form a large diversity of oxide compounds by employing various synthesis techniques. Metal oxide nanomaterials exhibit metallic, semiconductor, or insulator character due to the electronic structure difference. Semiconductors are materials whose valence and conduction bands are separated by an energy gap called as band-gap. When such materials absorb photons having energy equal or greater than its band-gap, it excites its electrons from the valence band to the conduction band, creating charge carriers.

Key Words: application, circuits, conduction, devices, electrons, insulator, properties

Introduction

Semiconductor metal oxides play a vital role in many areas of chemistry, physics, and material science. They are largely employed in a wide range of applications such as the fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, for coating against corrosion and as catalysts. The properties of metal oxides largely vary with the reduction of particle size in nano scale, which are either enhanced or completely display novel properties compared to their bulk materials. Thus, they are mainly prepared in the nanostructured form for improved properties and effectiveness. The term nanomaterials embody a large variety of materials in the realm of nanometers. These include ceramics, metals and metal oxide nanoparticles with nanometer-sized microstructures. By definition, a composite is considered to be any multiphase material that exhibits a significant proportion of the property of all constituent phases such that a better combination of properties is realized. Nanocomposite is a multiphase solid material in which at least one of the phases shows dimensions in the nanometre range, *i.e.* less than 100 nanometers. These include colloids, gels, porous media, copolymers and dendrimers. It is generally a solid combination of a bulk matrix and nano phase(s) material.

Some of the important classes of nanocomposites are as follows:

Ceramic-matrix Nanocomposites

In this type of composites the main part of the volume is occupied by a chemical compound from the group of nitrides, borides, oxides, silicides, *etc.* In most cases, ceramic-matrix nanocomposites include a metal as the second component. Such ceramic-metal combinations



were useful in improving their electrical, optical, magnetic, corrosion-resistance and other protective properties.

The concept of ceramic-matrix nanocomposites was also applied to thin films that are solid layers of a few nm to some tens of μm thickness deposited upon an underlying substrate that play an important role in the functionalization of technical surfaces.

Metal-matrix nanocomposites

Metal matrix nanocomposites are also referred as reinforced metal matrix composites. Boron nitride reinforced metal matrix composites, carbon nanotube metal matrix composites and carbon nitride metal matrix composites are the new research areas in metal matrix nanocomposites. Another kind of nanocomposite is the energetic nanocomposite, generally as a hybrid sol-gel with a silica base, which, when combined with metal oxides and nano-scale aluminum powder, can form superthermite materials.

Polymer-matrix Nanocomposites

In such type of nanocomposites a nano filler is added to a polymer and hence are also called as nanofilled polymer composites. If efficient dispersion of the filler is obtained in the polymer it can yield a high performance composite. An example of this would be reinforcing a polymer matrix by much stiffer nanoparticles of ceramics, clays or carbon nanotubes. In a recent study, polymeric nanocomposites were fabricated using various one-dimensional carbon nanostructures such as single- and multi-walled carbon nanotubes, as well as two-dimensional carbon and inorganic nanomaterials such as graphene platelets, graphene nanoribbon, single- and multi-walled graphene oxide nanoribbons, graphene oxide nanoplatelets and molybdenum disulphide nanoplatelets as reinforcing agents, to improve the mechanical properties of poly (propylene fumarate) nanocomposites, for bone tissue engineering applications. Oxide nanomaterials are used in a new class of ceramics. Advanced ceramics with their larger strength, higher operating temperatures, improved toughness and tailorable properties are used for applications such as chemical processing and environmental ceramics, engine components, computers and other electronic components, or cutting tools.

Nanoalloys

Bulk nano-crystalline alloys have attracted much attention due to their interesting mechanical and magnetic properties when compared with their fully amorphous and fully crystalline counterparts. In material science, intermetallic compounds and alloys are prepared by mixing elements and the properties of such materials are studied. In certain examples, there is an improvement in specific properties after alloying. This is usually ascribed as synergistic effect of one element on the rest of composition. Myriad array of such alloys and compositions with different structures and properties have been prepared. Many of such alloys have found applications in catalysis, electronics and engineering. It is of great interest to fabricate materials with well defined, controllable properties and structures on the nanometer scale. The bimetallic and trimetallic nanoclusters are called as alloy nanoclusters or nanoalloys.

Photocatalysis

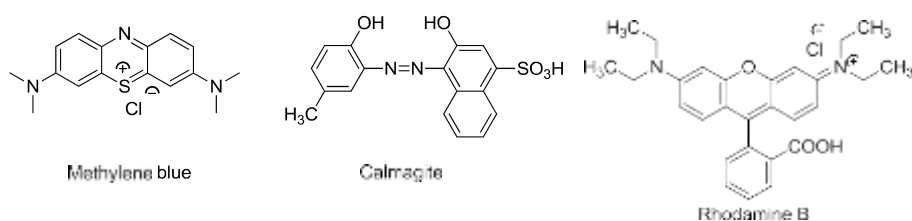
The term photocatalysis means a photochemical reaction accelerated by a catalyst. In this process light, *i.e.* a photon is absorbed by a catalyst directly or *via* a dye on the surface. The adsorbed photon excites an electron in the catalyst. The excited electron travels from the



valence band to the conduction band, leaving a hole in the valence band. The electrons and holes diffuse to the surface of the catalyst where they come in contact with the molecules of chemical adsorbed on it. The adsorbed chemical is the one on which we intend to have a photochemical reaction. The electrons can bring about the reduction of this chemical and the holes an oxidation. Such types of photo oxidations have a great ramification in the field of environmental cleansing. Dyes and other toxic chemicals can be broken down to benign or non-toxic molecules by such photo oxidation processes. The catalysts used for such processes are normally semiconductor materials. The elimination of contaminants from waste effluents has become a primary issue due to the consequences to human health. Worldwide environmental problems associated with the textile industry are mainly due to water pollution caused by the discharge of untreated effluent and toxic chemicals especially during processing. The major contaminant includes dye waste from industrial effluents. Dyes usually have a complex aromatic molecular structure, which makes them more stable and difficult to biodegrade. Many such organic dyes include Methylene blue, Rhodamine B, Malachite green, Calmagite, Methyl orange, *etc.* Methylene blue (MB) is a most commonly studied azo dye due to its complex aromatic structure and harmful intermediates. Methylene blue (tetramethylthionine chloride, $C_{16}H_{18}ClN_3S$) is a heterocyclic aromatic dye, a member of thiazine dyes used for a number of different staining procedures, which includes colouring paper, dyeing cottons, wools, silk, leather and coating for paper stock. Although methylene blue is not strongly hazardous, it can cause some harmful effects, such as heartbeat increase, vomiting, shock, cyanosis, jaundice, quadriplegia and tissue necrosis in humans.

Calmagite ($C_{17}H_{14}N_2O_5S$), is a synthetic azo-dye widely used in textile industries, cosmetic, paper, drug and food processing industries in large quantities. Effluent of this dye is hard to treat and made of harmful organic and inorganic chemicals that exhibit toxic and carcinogenic effects toward biological systems.

Rhodamine B ($C_{28}H_{31}ClN_2O_3$) is another basic dye, which is an important representative of xanthene dyes, has two molecular forms of cationic and Zwitterionic form. Cationic dyes are considered as toxic colorants and can cause harmful effects such as allergic dermatitis, skin irritation, mutations and cancer. It is widely used in the manufacturing of textile, printing, paper, pharmaceutical and food products, and because of its high solubility in water, it constitutes an important source of environmental pollution reaching the atmosphere and ground waters. To reduce the risk of environmental pollution from such effluents, it is extremely important to treat them before discharging. Photocatalytic degradation has enormous potential to control aqueous contaminants or pollutants [13-15]. Structures of these dyes are given below.



Brief outline of various synthetic methods

The properties of metal oxides can be altered based on many aspects. One of the most



important aspects is by altering the synthetic method. Research on the controlled formation of nanocrystalline materials has reached to an enormous extent in recent years. Modification in preparative strategies is done in empirical manner and rational approaches for the controlled formation of nanomaterials are available for a limited number of systems. This can be done by understanding the fundamental mechanism of nanocrystal formation.

Various synthetic approaches are available for the preparation of metal oxide based photocatalysts, and most popular are co-precipitation reactions, reduction synthesis, aqueous and non-aqueous sol-gel reactions, combustion method, decomposition method, hydrothermal and solvothermal approaches, *etc.* are adopted to prepare visible light active photocatalysts.

Co-precipitation is used for simultaneous precipitation of more than one component. Catalysts based on more than one component can be prepared easily by co-precipitation. The synthesis is generally carried out at room temperature and the properties of synthesized nanomaterial or nanoparticles are found to be dependent on initial reaction parameter such as pH values and reaction temperature. The precipitation process is used for preparation of bulk catalysts and support material such as Al_2O_3 , SiO_2 , TiO_2 , ZrO_2 , *etc.*

Hydrothermal method involves the thermal reaction (usually above $100\text{ }^\circ\text{C}$) of chemical/s in aqueous medium in a sealed vessel. Metal oxides with different shapes (flowers, rods, tubes, *etc.*) have been synthesized by using this method. It is a useful method for the preparation of hydroxides, oxyhydroxide and oxides. It can also be used for non-oxide metals (for instance nitrides, chalcogenides, *etc.*).

Solvothermal method is a chemical reaction in a closed system in the presence of a solvent (aqueous and non-aqueous solution) at a temperature higher than that of the boiling point of such a solvent. Solvothermal reactions have been mainly used for preparing micro or nanoparticles with different morphologies for applications such as sensors, separation and catalysis, molecular devices and spintronics.

Combustion synthesis is a low-cost method used for the production of various industrial materials. The process involves an exothermic reaction accompanied by evolution of heat and light. For any combustion synthesis, fuel and oxidizer are necessary ingredients. In this synthesis method, the metallic precursor gets decomposed by the heat generated by the violent reaction between the fuel the oxidizer. *The thermal decomposition method*, also called as thermolysis is used to produce tunable and highly crystalline products. It involves decomposition of some precursor in the presence of a surfactant dissolved in a high boiling point, non-reacting solvent. The reaction is usually endothermic in this process and heat is essential to break chemical bonds in the compound enduring decomposition.

Template/surface-mediated synthesis is generally based on the construction of the needed nanomaterial within the pores or channels of a nanoporous template. Various synthetic techniques such as electrochemical, sol-gel, chemical polymerization and chemical vapor deposition are the major strategies employed in this type of fabrication. Nanomaterial with desired morphologies such as fibrils, rods, and tubules can be synthesized by using different template materials. The method is mainly used to obtain highly monodispersed mesoporous



nanomaterials with uniform shape and a high specific surface area.

The sol-gel method is a homogeneous process wherein there is a continuous transformation of a solution into a hydrated solid precursor (hydrogel). The method involves hydrolysis and gelation of appropriate precursors in alcoholic solution. The advantages associated with sol-gel process include synthesis of nanosize materials of high purity and of uniform size, low reaction temperature, controlling the reaction process by stoichiometric addition of reagents, and synthesis of composite nano materials. These advantages have made the sol-gel method very popular for the synthesis of TiO₂-based photocatalysts.

Structural and surface properties

Mesoporous materials are materials having pore sizes in the range of 2–50 nm. Due to the combination of porous structure and high surface area these materials are expected to have improved catalytic properties, owing to which they have received much interest in the field of catalysis and nanomaterials. According to the IUPAC, a mesoporous material can be disordered or ordered in a mesostructure. In crystalline inorganic materials, mesoporous structure restricts the number of lattice units, and thus alters the properties of the material.

Optical Properties

The main mechanism of light absorption in pure semiconductors is direct inter-band electron transitions. This absorption is observed to be smaller in indirect semiconductors, *e.g.*, TiO₂, where the direct electron transitions between the band centers are prohibited by the crystal symmetry. Whereas direct semiconductor, possess comparatively higher absorption coefficient and most of the sunlight is absorbed within a small range beneath the surface.

Magnetic Properties

Semiconductor materials can be readily transformed into a magnetic semiconductor material by adding some foreign impurities having magnetic properties. Since the magnetic element concentration is relatively low (dilute), the resultant material is termed as dilute magnetic semiconductor (DMS). These materials have gained much interest due to their semiconductor properties associated with magnetic properties. The ferromagnetism shown at room temperature on introducing small quantities of magnetic impurity into the non-magnetic material is generally termed as room temperature ferromagnetism (RTFM). Most of the late transition metals, mainly of the first transition series, are added as dopant to semiconductor metal oxides so as to prepare DMS materials and hence to show RTFM. Commonly reported systems include, Cu, Co, Ni, Fe doped TiO₂, ZnO, SnO₂, *etc.*

Brief introduction to characterization techniques

Physicochemical characterization is a major aspect to understand the detailed properties of a material. Some basic properties, such as crystallinity, crystallite size, nature of active sites, morphology, particle size, phase structure, optical properties, surface structure, textural properties, *etc.* are characteristic features of any materials to be studied. A brief introduction to some important characterisation techniques employed for the present research work is summarized here with their basic principles.

X-ray Diffraction Properties of TiO₂ Nanomaterials

XRD is an essential technique used in the determination of structural properties such as phase



identification and unit cell dimensions of the nanocrystalline material. It gives information about the crystal structure and the crystallinity, lattice parameter and crystallite size, which is estimated using Scherrer equation - $D = K\lambda / \beta \cos\theta$, where K is a dimensionless constant, 2θ is the diffraction angle, λ is the wavelength of the X-ray radiation, and β is the full width at half-maximum (FWHM) of the diffraction peak. Crystallite size is inversely related to the FWHM of an individual peak and is determined by measuring the broadening of a particular peak in a diffraction pattern associated with a particular planar reflection from within the crystal unit cell.

Diffraction patterns of the nanomaterials are recorded with an X-ray diffractometer using Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). By investigative diffraction pattern, the crystalline phase of nanomaterial, can be identified. It was discovered by Laue in 1912 to determine long range order of crystalline phases, in which crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice. It is now a commonly used technique for the study of crystal structures and atomic spacing. In this technique, the monochromatic beam of X-ray is made to incident on the analyte at various angles (θ). After the striking analyte it diffracts producing constructive diffraction patterns for certain angles corresponding to particular planes (h,k,l) to satisfy Bragg's diffraction condition, which is given by $2d\sin\theta = n\lambda$, where d is the interplanar distance between diffracting planes, θ is the angle of incidence of X-Ray, λ is the wavelength of the incident X-Ray and n is in an integer. The widths of the diffraction peaks are closely related to the size, size distribution, defects and strain in the nanocrystals.

Scanning electron microscopy (SEM)

This technique allows direct observation of topography and morphological features with the help of signals produced by the interaction of electrons with atoms in the sample. The shape and morphology of nanostructured materials can be preliminarily determined by SEM. It produces images of the sample by scanning the surface with a focused beam of electrons. When an incident electron beam interact with specimen surface, secondary electrons with energies typically smaller than 50 eV evolve from the surface, whose emission efficiency depends upon the surface geometry, surface chemical characteristics and bulk chemical composition.

Transmission electron microscopy (TEM)

The technique is used for atomic mapping of the crystal. It is a high spatial resolution structural and chemical characterization technique. In this a thin specimen is illuminated with uniform and high intensity electrons and the forward transmitted beam is analyzed. TEM can be used to characterize metal oxide nanomaterials to determine particle size, shape, crystallinity and inter-particle interaction.

High resolution-transmission electron microscopy (HR-TEM) is a further advancement of this technique which is used to provide atomic resolution images of catalytic active sites under controlled environments (<10 torr). Hansen and co-workers have further expanded such studies to a wide range of catalytic materials and reaction conditions.

Selected area electron diffraction (SAED) is a crystallographic technique performed inside TEM, in which angular distribution of scattering can be viewed in the form of diffraction patterns. It is used to identify crystal structures and examine crystal defects and is mostly



useful in polycrystalline specimens.

UV-Vis diffuse reflectance spectroscopy

UV-visible diffuse reflectance spectroscopy is one of the simplest techniques useful for studying optical and electronic properties of nanomaterials, which was discovered in 1970s and was mainly used for metal oxide catalytic materials. Weber extended these findings for quantitative determination of band-gap, *e.g.*, of a metal oxide. The basic operating principle of this spectroscopic technique is a measurement of light absorption due to electronic transitions in nanomaterials. It uses light in both the UV and visible region of the electromagnetic spectrum for electronic transition.

Diffuse reflectance spectroscopy (DRS) is a technique closely related to UV-Vis spectroscopy in which valence electron is excited to an empty orbital and the relative change in the amount of light being reflected off from a source is measured. It is used to study the band gap of a semiconductor material which is calculated by using equation - $E = h \cdot C / \lambda$, where, h = Planck's constant = 6.626×10^{-34} Joules sec, C = Speed of light = 3.0×10^8 meter/sec and λ = Cut off wavelength

Fourier transforms infrared spectroscopy (FT-IR)

Infrared is a common vibrational spectroscopy technique useful for characterizing structural properties such as vibrational frequencies of molecules and phonons for identification of functional groups of the sample. It also provides information about chemisorbed molecules on oxide surfaces. However, it is lacking the fundamental molecular structural information about the metal oxide catalytic active sites on surfaces. Thus, it is used to study bonding modes of inorganic and organic materials. It is based on the measurement of transmitted IR light through a sample.

In this technique, the sample is illuminated with IR radiation which leads to excitation of vibrational modes in the chemically bonded functional groups. For nanoparticles, IR spectrum measures the phonon mode. Interaction between the surface molecules or ions with nanoparticles usually causes a change in the vibrational or phonon frequencies. Hence the changes in frequencies measured in IR spectrum can give information about the interaction between the nanoparticles and surface molecules. The necessary condition to spectrum to appear is vibrations amongst bonded atoms should produce the change in the permanent dipole moment of the molecule. It is considered that the more polar bond gives intense signals.

Thermal Analysis [Thermo-Gravimetric/Differential Thermal Analysis (TGA/DTA)]

Thermo-gravimetric analysis (TGA) studies change in mass of a sample with an imposed change in the temperature. The changes that occur on heating include melting, phase transition, sublimation, and decomposition. TGA measures mass changes in a material as a function of temperature (or time) under a controlled atmosphere. Its principal uses include measurement of thermal stability and composition of a material. TGA is most useful to study for dehydration, decomposition, desorption, and oxidation processes. In Differential Thermal Analysis (DTA), the temperature of a sample is compared with that of an inert reference material during a programmed change of temperature. Changes in the sample, either exothermic or endothermic, can be detected relative to the inert reference. Thus, a DTA



curve provides data on the transformations that have occurred, such as glass transitions, crystallization, melting and sublimation. The area under a DTA peak is the enthalpy change and is not affected by the heat capacity of the sample.

Brunauer-Emmett-Teller (BET) surface area

The specific surface area of a material is evaluated by this analysis by nitrogen multilayer adsorption measured as a function of relative pressure. This technique covers the external area and pore area evaluations to determine the total specific surface area in m^2/g of macroporous and mesoporous materials, along with pore volume and pore size distributions that characterise porosity of a material. Standard BET analysis is conducted at the boiling point of liquid nitrogen (77 K).

Barrett-Joyner-Halenda (BJH) analysis can also be employed to determine pore area and specific pore volume using adsorption and desorption techniques. This technique characterises pore size distribution independent of external area due to particle size of the sample.

Vibrating Sample Magnetometry (VSM)

It is a technique used to measure the magnetic properties of a material. It is based on Faraday's law of induction in which the vibrating component causes a change in the magnetic field of the sample, which generates an electric field in a coil. The sample is placed within suitably placed sensing coil and held at desirable angle and is mechanically vibrated. The magnetic field appears around the sample and magnetization of the sample is analysed as the vibration begins. This generates hysteresis loop which shows the nature of magnetization and is a characteristic of a material.

X-ray photoelectron spectroscopy (XPS)

XPS is a surface sensitive quantitative spectroscopic technique, essentially used for measuring elemental composition, empirical formula, chemical state and electronic state of the element within the material. It is mainly used to study the binding energy of core electron in an atom. XPS is based on the principle of measurement of the kinetic energy of photoelectrons generated when the sample is illuminated with soft X-ray radiation in the ultrahigh vacuum.

The energy of the photoelectrons thus leaving the sample is determined and this gives a spectrum for intensity as a function of binding energy. The binding energies of the peaks are characteristic of each element. Hence, XPS can also provide chemical binding information. For catalytic study, it was first developed by Siegbahn in the late 1950s and 1960s, to allow determination of the surface region of catalytic materials.

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