ZnO Nanoparticles: Synthesis Techniques, Properties and Applications

Tejendra Chandrakar[1] Sandhya Pillai[2]
[1,2]Department of Nanotechnology
[1,2]Christian College of Engineering and Technology, Kailash Nagar, Bhilai

Abstract— ZnO nanoparticles have been a subject of intensive studies in recent years due to their unique material properties and wide applications, in electronics, photonics, cosmetics, biosensors, rubber industry, pigments etc. ZnO is a direct wide band gap semiconductor having band gap energy of 3.37 eV and a large exciton binding energy of 60 meV at room temperature making it a prospective candidate for many applications such as UV lasers, light-emitting diodes, solar cells, nanogenerators, gas sensors, and photodetectors. Several novel and cost effective techniques have been employed by researchers in the synthesis of ZnO nanoparticles. The paper presents a review of the most important chemical, physical and biological techniques used in the synthesis of ZnO. The various useful properties of ZnO that has captured much attention owing to which it has found wide applications in different technological domains have also been discussed.

Key words: UV Visible Spectroscopy, Tauc's Plot, Absorption Spectra, XRD, SEM etc.

I. INTRODUCTION

Nanomaterials are of great interest because it shows unique optical magnetic, electrical and other properties. These properties have great impact on the potential impact in electronics field. Nanostructured materials are known to show various non-linear optical properties. Nanostructured semiconductors are used as window layers in solar cells. Nanosized metallic powders have been used for the production of gas tight materials, dense parts and porous coatings. In nanoparticles, high surface to volume ratio is a necessity but the agglomeration of small particle precipitated in the solution is the main concern in the absence of any stabilizer.

In addition, nanoparticles are generally stabilized by steric repulsion between particles due to the presence of surfactant, polymer molecules, or any organic molecules bound to the surface of nanoparticles. Sometimes, electrostatic repulsion also plays an important role in nanoparticles stabilization. With all the issues related to nanoparticle synthesis, there are various types of nanoparticles reported in the literature, e.g., metal nanoparticles, metal oxide nanoparticles, and polymer nanoparticles. Among all these, metal oxide nanoparticles are one of the most versatile materials, due to their unique optical mechanical and electrical properties.

Most preferably, among different metal oxide nanoparticles, Zinc oxide (ZnO) nanoparticles have their own importance due to their vast area of applications like gas sensor, chemical sensor, biosensor, superconductor, photocatalyst, optoelectronic device, cosmetics etc [1].Zinc Oxide is a II-VI semiconductor which is quite capable of exhibiting properties that are very useful in synthesing nanomaterial thin film and powder. Zinc Oxide is environmental friendly and easy to synthesize [2]. It is tolerant to large electric fields, high temperature and high power. It also has low resistivity and high transmittance in the visible solar region

Zinc Oxide is an interesting material not only because of its physical property but also due to a wide variety of morphologies. Some of the ZnO nanostructures exhibit various shape such as nanotube[3] nanowire/nanorod [4,5],nanobelt [6] nanocomb [7,8],nanoflower and nanosheet [9] and tetrapod [10] like structure.Due to their high catalytic activity, ZnO has a great advantage to be applied in catalytic reaction process[11]

Zinc oxide crystallizes in three different forms, namely hexagonal wurzite, cubic Zinc Blende, and rarely cubic rocksalt. The Wurzite structure is very stable at ambient atmosphere even upto a high temperature (800 ^{0}C). The hexagonal structure has a point group 6mm according to Hermann-Mauguin notation and the space group is P63mc or C6v4.

II. PROPERTIES OF ZINCOXIDE

The Various useful properties of ZnO has been recognized for a long time which has gained much attention in the last few years. ZnO is a material having a direct wide band gap of 3.44 eV at low temperature and 3.37 eV at room temperature which is comparable to the respective value of Wurtzite GaN which is 3.50 eV at low temperature and 3.44eV at room temperature[12].It has a high excitonic binding energy of (60 meV) which is much larger than that of GaN (25 meV)[13]. A few of its properties are:

A. *Electrical Properties:*

The photoresponse of ZnO consist of two parts: a rapid process of photogeneration and recombination of electron hole pair and a slow process attributed to the oxygen adsorption and photodesorption on the film surface as well as the grain boundaries. The presence of metal conductivity in film and lower wavelength shift of optical band gap shows the presence of relatively high carrier concentration. It can be caused by annealing. In ZnO the mobility depends on temperature, it will increase to a peak point slowly and decrease with increasing the temperature.

The total mobility is given by using this formula:-

$$\frac{1}{\mu} = \frac{1}{\mu_l} + \frac{1}{\mu_g} + \frac{1}{\mu_{ii}}$$

Where μ_g are scattering grain size, μ_l are scattering lattice at low temperature, μ_{ii} are ionized impurity scattering [14].

B. *Thermoelectric Property:*

ZnO is considered to be one of the most promising thermoelectric materials which has recently been given attention due to its stability and economy. TEG are based on p - and n - type semiconductor materials connected in series and parallel to deal with current and heat flow, respectively. The thermoelectric figure of merit, ZT = S2σT/κ where T is the temperature, S is the Seebeck coefficient, σ is the electrical conductivity, and κ is the thermal conductivity, is the measure of the potential efficiency of a thermoelectric material[15].

The thermoelectric power factor (PF) of ZnO is comparatively higher and can be used as alternatives to the conventional thermoelectric materials. The PF is given as ZT ¼(rS2T)/k, where r is defined as electrical conductivity. The doping mechanism can be adopted in ZnO to restructure the crystal architecture, which can possibly increase r. In such a way, the semiconductor behavior can be changed into a metallic one. The Al doping has been reported to increase the magnitude of r to the third order; however, high k results in a low thermoelectric performance. The thermoelectric properties can be improved by improving the electronic transport mechanism [16].

C. Structural Property:

The X Ray Diffraction spectrum of synthesized ZnO nanoparticles confirms the hexagonal wurtzite structure. Wurtzite structure of ZnO nanoparticles belongs to space group (C6V=P63mc) and have unit cell parameters a = b = 3.253 Å and c = 5.209 Å. The average size of the nanoparticles has been calculated using the Debye-Scherrer formula

$$D = \frac{0.9\lambda}{\beta\cos\theta}$$

Where, λ is the x-ray wavelength (1.504 nm), θ is the Bragg diffraction angle, and β is the full width at half maximum.

D. Optical Property:

ZnO absorbs the radiations in the UV range up to 361.75 nm and almost all the visible spectrum radiations are transmitted by ZnO nanoparticles. The absorbance curve can be plotted between Absorbance with respect to their respective wavelength. The band gap of ZnO nanoparticles was calculated by extrapolating the curve drawn between (hv) and $(ahv)^2$ Where v is the frequency and α is the optical absorption coefficient. The band gap energy obtained by extrapolating curve is found to be approximately 3.3eV[16].Higher transmittance is obtained in IR region. Reflectance decrease very rapidly with increase in wavelength.[17].

III. METHODS OF SYNTHESIZING NANOPARTICLES

There are some Physical, Chemical and Biological technique used in the synthesis of ZnO Nanoparticle

A. Physical Technique

1) High Energy Ball Mill

High energy ball milling is a non-equilibrium process. Amirkhanlou etal reported high energy ball milling processes as very efficient, cost-effective, and simple technique for the preparation of ZnO nanostructures[18].Powdered material placed inside a ball mill is subjected to high energy collision from balls [19]. Milling was performed in a horizontal oscillatory mill operated at 25Hz. The ball mill machine was operated at a rotation rate of 1200 rpm (rotation per minute).[20] Container is closed with tight lids. The mixture ratio of steel balls and ZnO powders was around 15:1 by weight percent. The milled materials were used directly with no added milling media[21]. Five balls were kept in each cell along with 10 g of the sample powder. If the container is more than one half occupied, effectiveness of milling is condensed. Weighty milling balls increase the impact energy on collision. Larger balls, used for milling produce smaller grain size and larger defects in the particles. The process may add some impurities from balls. [22] Temperature rise in the range of 100 to 1100 ^{0}C is expected to take place during the collisions. Lower temperature shows amorphous particle formation. The gases like oxygen, Nitrogen etc. can be the source of impurities as constantly new, active surfaces are generated. Cryo-cooling (low temperature cooling) is used sometimes to dissipate the generated heat. In addition they may rotate around some central axis and are called as 'planetary ball mill'. Some of the materials like Zn,Sn are made nano crystalline by this method.

By using this deposition technique, thin films of different metals are deposited on different surfaces. This technique involves condensation from the vapor phase. There are three main steps involved in this process.

1) By sublimation or evaporating of a material to the corresponding vapor phase.
2) Transportation of the material to the substrate from source.
3) Formation of the thin film and particle by nucleation and growth. The source can be evaporated by using electron beams, thermal energy, sputtering technique, cathode arc plasma.

2) ION Plating

Ion plating is also one physical deposition technique that uses electron beam evaporation. The material used in this type of coating is ionized and vaporized through the aid of an electric arc and then forced toward the target at high speed. This process is commonly performed within a vacuum chamber or an inert gas setting. It is also known as physical vapor deposition. Below Fig shows the experimental set up of ion plating [23].

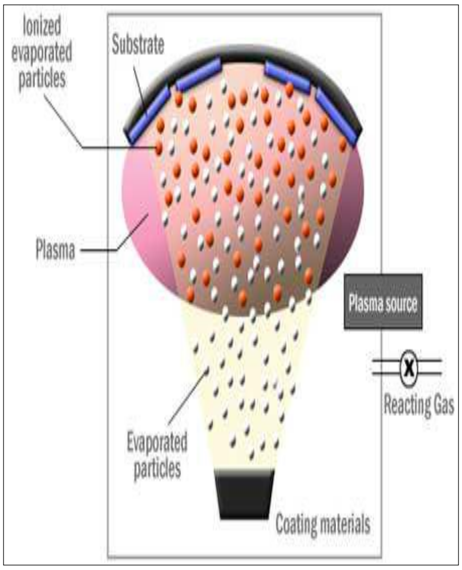

Fig. 1:

Materials that are to be subjected to ion plating are usually treated prior to plating in order to thoroughly clean them and eliminate far-off materials. This is also performed in the environment where plating is to be accomplished, a process called sputtering. Plating and sputtering can be two identical processes, but with the former, the target is typically bombarded with other materials ions, like argon gas, that can strip off all far-off particles rather than promoting a bond. When the target is prepared and treated for coating application, the process of ion plating commences. The coating material undergoes vaporization with the help of electric arc current that makes use of the high flow of current and low voltage. This is done in order to ionize all atoms that repel each other because of similar electric charges, as well as to vaporize the material used for coating. The vapor produced is propelled onto the target and delivered in opposite weak electrical charge to be a magnet for the ionized coating. This process makes ion vaporization possible and in turn results in the adhesion of coating to the surface [24].

3) Molecular Beam Epitaxy

MBE can be considered as a special case of evaporation for single crystal film growth, with highly controlled evaporation of variety of source in ultra-high vaccum of typically 10^{-10} torr.[25,26] MBE consist of real time structural and chemical characterization capability including X-Ray photoelectric Spectroscopy,Auger electron spectroscopy. In MBE the evaporated atom or molecule from one or more source do not interact with each other in the vapour phase under such a low pressure. Although some gaseous source are used in MBE, mostly molecular beam are generated by heating solid material placed in source cell. The source material are commonly raised to the desired temperature by resistive heating. The mean free path of atom or molecule far exceeds the distance between the source and the substrate inside the deposition chamber. The atom or molecule striking on the single crystal substrate result in the formation of desired epitaxial film.The main attribute of MBE include[27] :-

1) A low growth temperature that limit diffusion and maintan hyper abrupt interface, Which are very important in fabricating two dimensional nanostructure or multilayer structure such as quantum wells.
2) A Slow growth rate that insures a well-controlled two dimensional growth at a typical growth rate of 1 micrometre/hours. A very smooth surface and interface is achievable through controlling the growth at the monoatomic layer level.
3) A simple growth mechanism compared to other film growth techniques Insure better understanding due to ability of individual controlled evaporation of sources
4) A varity of in situ analysis capability provide invaluable information for the understanding and refinement of the process.

B. Chemical Technique

1) Sol Gel Method

As the name suggests sol-gel involves two types of materials or components, ‘sol’ and ‘gel’. Sol – gel are known since the time when M. Ebelman synthesized them in 1848. However it is only last one or two decades that considerable interest in it, both in scientific and industrial field, has been generated due to realization of the several advantages one gets as compared to some other techniques. This method also enables to modify the surface of ZnO by using some organic compounds and, as a consequence, changing the properties and extending its domain of applications. Suwanboon and e.tal [28] prepared nanocrystalline ZnO powder by sol-gel method from zinc acetate dihydrate $Zn(CH_3COO)_2\ 2H_2O$, polyvinylpyrrolidone (PVP) and NaOH. [28] The precipitate obtained was dried at 60 ºC and calcined at 600 ºC in air for 1 h.The XRD characterization indicates a wurtize or hexagonal structure with small average crystallites of about 45 nm. The morphology was modified from platelet-like to rod shape when adding PVP into solution. The sol-gel method was also used to obtain zinc oxide nanoparticles with good antibacterial activity [29]. The raw materials used for the synthesis were zinc acetate, oxalic acid and water. The white gel precipitate obtained was heated first at 87 ºC for 5 h, and then at 600 ºC for 2 h. It is also possible to synthesize nanoparticles, nanorods, nanotubes etc.using sol-gel technique.[30] Sols are solid particles in a liquid see in below fig They are thus a subclass of colloids. Gels are nothing but a continuous network of particles with pores filled with liquid (or polymers containing liquid).Sol-gel process involve formation of ‘sol’ and then connecting the sol particles (or some subunits capable of forming a porous network) to form a network. By drying the liquid, it is possible to obtain powders, thin films or even monolithic solid. Sol-gel method is particularly useful to synthesize ceramics or metal oxides although sulphides, borides and nitrides also are possible.

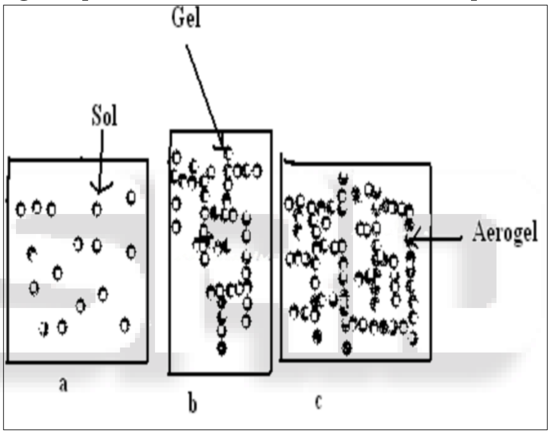

Fig. 2:

2) Hydrothermal Method

Hydrothermal Method is one of the chemical method for the synthesis of Nano particle. The hydrothermal method is a simple and environmentally friendly technique, which takes place in an autoclave at a programmable temperature and reaction time[31] . For Preparation of ZnO Zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$, 98%) and sodium hydroxide (NaOH, 97 %) were used as the starting chemicals. To prepare ZnO nano crystals, Zinc nitrate hexahydrate and NaOH were taken in required quantities and were dissolved in de-ionized water. An aqueous solution of 0.5 mol/L $Zn(NO_3)_2.6H_2O$ was mixed with the appropriate amount of 1 and 5 mol/L NaOH solution under magnetic continuous stirring to obtain the mole ratio of $Zn(NO_3)_2.6H_2O$:NaOH of 1:2 and 1:10. The final pH of the mixed solutions was highly basic with pH of 14. The mixture was put into a Teflon-lined-stainless steel autoclave unit for hydrothermal reaction at 150 ˚C for 7h. After hydrothermal reaction, the reactor was naturally cooled to room temperature. The obtained product was filtered, washed with de-ionized water till the pH of the final solution was 7.0. Finally the as-prepared sample was calcined at 600˚ C in a programmable muffle furnace at a rate of 2˚ C / min for 1 hr.[32].

3) Chemical Bath Deposition

Chemical Bath deposition method is simple and cost effective technique for the synthesis of Nanocrystalline thin film. A Kathalingam & e.tal [33] used chemicals such as zinc acetate and ethylenediamine as the starting material. A hot plate with a magnetic stirrer was used to heat and stir the bath solution.Glass plates were used as the substrate. Bare soda lime glass and SnO2 coated glass plates were also used as substrates, in order to study how the choice of substrate material affects the deposition of ZnO thin films. All the substrates were cleaned with acetone, and then washed with triple distilled water. The zinc plate was first scrubbed with emery paper before washing. The film deposition was performed at various temperatures, with and without stirring the bath solution. The bath solutions were prepared with zinc acetate and ethylenediamine, both their concentrations varying between 0.25 M and 1 M. A mixture of solution with the desired density of zinc acetate and ethylenediamine was placed in a 100 cm3 beaker and the solution was kept in a constant temperature bath. One side of the surface of each substrate, in particular of the zinc plate, was cov-ered with insulating tape before being dipped into the solution. The pH of the bath was varied between 8 and 11 with the use of NaOH solution. The reaction mixture was maintained as constant at a desired temperature for deposition with continuous stir-ring. The substrates were removed from the deposition solution at various time inter-vals, washed with distilled water and dried, and then these deposits were subject to various tests.[33]

Fig. 3:

4) Chemical Precipitation Method

This method is widely used to obtain zinc oxide with repeatable properties. The method consists of a spontaneous reduction of an aqueous solution of a zincsalt (ZnSO4, Zn(NO3)2, Zn(CH3COO)2) with a reducing agent, followed by precipitation of the precursor of ZnO from the solution and the thermal treatment [34]. This process is controlled by different parameters such as solution concentration, pH, reaction temperature, time of precipitation and washing medium [35]. Zinc nitrate as the precursor, KOH as a precipitating agent to synthesize ZnO nanoparticles. ZnO nanoparticles were synthesized by direct precipitation method using zinc nitrate and KOH as precursors. In this work, the aqueous solution (0.2M) of zinc nitrate (Zn(NO3)2.6H2O) and the solution (0.4 M) of KOH were prepared with deionized water, respectively. The KOH solution was slowly added into zinc nitrate solution at room temperature under vigorous stirring, which resulted in the formation of a white suspension. The white product was centrifuged at 5000 rpm for 20 min and washed three times with distilled water, and washed with absolute alcohol at last. The obtained product was calcined at 500 °C in air atmosphere for 3 hr.[36] The precipitation method is an unsophisticated,high quality, relatively low cost method with high manufacture yield.

C. Biological Method

1) Synthesis using Micro-Organism

Under the microscope the microorganisms like fungi, bacteria, yeast etc. are detected. In processing of curds, chesses, bread, vaccines, alcohol etc. some bacteria are quite used. Some are responsible for spoiling food causes disease & some are harmful. There is interaction between the microorganisms with the metals & comes with contact with them through their cells & nanoparticles are formed. In Fig. (a) prokaryotic & (b) eukaryotic cells were illustrated respectively. The cell metal interactions are quite complex in general due to complexity of cells themselves. It is, however, well known that certain microorganisms are capable of separating method ions. This is widely used to either recover precious metals or detoxify water.[37] However, use of microorganisms in deliberate synthesis of semiconductor, metal or insulator nanoferriteparticles is relatively new area and will be discussed now with some examples.

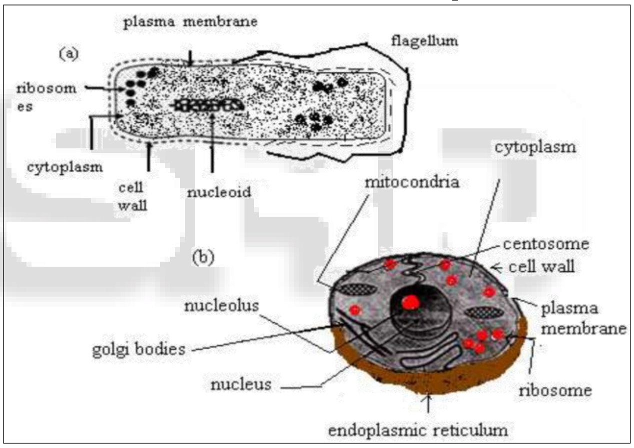

Fig. 4:

2) Synthesis using Plant Extract

Using the plants for production of nano-particles is an organism rather new and heading to really green chemistry that technologists are looking for. However, compared to use of microoranization to produce nanoparticles, use of plant extracts is relatively less investigated. There are few examples which suggest that plant extracts can be used in nanoparticles synthesis. For example it has been reported that live alfalfa plants are found to produce gold nanoparticles from solids. [38] Leaves of geranium plant (pelargonium graveolens) have also been used to synthesize nanoparticles of gold. It should be mentioned here that there is also a plant associated fungus which can produce compounds such as taxol and gibberellins. There is an exchange of intergenic genetics between fungus and plant. However, the nanoparticles produced by fungus and leave have quite different shapes and size distributions. Nanoparticles obtained using Colletortichum sp. fungus related to geranium plant has a wide distribution of size and particles are mostly spherical. On the other hand geranium leaves produce rod and disk shaped nanoparticles. This approach is an environment-friendly, cost-effective, biocompatible, safe, green approach. Green synthesis includes synthesis through plants, bacteria, fungi, algae etc. They allow large scale production of ZnO NPs free of additional impurities.

IV. Application of zinc oxide

Zinc Oxide has been extensively used in many technological application such as gas sensor, Chemical Sensor, FET, Photodiode, Cosmetics, in textile

A. Gas Sensor, Chemical Sensor

Conductive metal oxide semiconductor thin films are the most promising devices among all solid state chemical sensors due to their low cost, low power consumption, online operation and high compatibility with microelectronic processing. The fundamental sensing mechanism of metal oxide based gas sensor relies on a change in electrical conductivity due to the process of interaction between complex surface Such as O^-, O_2^-, H^-, OH^- reactive gas species and chemicals and gas molecules to be detected. Although a large number of different oxides have been investigated for their gas sensing property, commercially available gas sensors are mainly made of ZnO in the form of nanocrystalline thin film.[39]

B. Field Effect transistor

Early studies of ZnO FET have focus only on their device performance and photo detection[40,41].The interface roughness play a prominent role in the electronic transport for transistor also and the electronic capabilities of ZnO nanowire FET strongly depend on their shape and size. The ZnO nanowire FET shows good properties such as good transparency to visible light, excellent uniformity and high mobility compared with traditional amorphous material. The temperature dependence of the stable device of ZnO nanowire FET range from 323k to 363k [42].

C. Photodiode

The Zinc Oxide nanostructure are synthesized and fabricated as photodiodes also. There are number of scientific and industrial application of photodiode at nanoscale[43,44].The experimental analysis of Zinc Oxide reported that the nanostructure of ZnO heterojunction photodiode consisting of p-Si and n-ZnO nanowire core and shell structure. The coating consisting of an n-type ZnO layer and a p-type silicon nanowire is used in photodiode applications. These Photodiode present enhanced ultraviolet and visible responsivities compared to other planar thin film photodiode[45,46].

D. Cosmetic Industry

Owing to good antifungal activity, zinc oxide is widely used in production of various raw materials used in medicine such as disinfectant agents and for Cosmetics applications [47].ZnO nano particles absorb UV radiation and can be used in sun protective creams [48].Even if there have been made experimental and clinical studies about using vitamin E in new formulations such cosmetics and sun care products, there is still a lack of enough indications if it has a clinical benefit [49]. So, there are many research works that had studied skin tissue regeneration. In recent years, it have been used medicinal plants to induce regeneration of the skin. An example is aloe-vera, due to its therapeutic properties, such anti-inflammatory, antibacterial and the ability to improve the collagen to promote the skin repair [50].

E. Textile Industry

The applications of nanoparticles in textile industry become attractive because provide high durability for treated fabrics due to large surface area and high surface energy that ensure better affinity for fabrics and leads to an increase of the desired textile functions [51].Many researches shows that the ZnO nanoparticles used in finishing processes improve UV absorption characteristics to the treated materials but also add two functions: an antibacterial activity and self-cleaning property [52].Research regarding the use of zinc oxide in matrix of films of chitosan showed a better antibacterial activity since the decrease of molecular weight of chitosan increases the antibacterial activity. This phenomenon was explained as the decrease of chitosan molecular weight in the composite, which improves the movement of the chains in the solution by decreasing the viscosity [53].

F. Rubber Industry

Zinc Oxide has utilized increasing number of many optical physical and Chemical property over the past one century. Zinc Oxide is known as additive in rubber industry, acting as activator for sulphur vulcanization, by increasing the efficiency of the cross-linking system. It proved the most effective activator to speed up the rate of cure with the new accelerator. Tires carry high loading of zinc oxide for heat conductivity as well as reinforcement since heat buildup is critical at higher operating speed compared with their solid counter rubber counterpart. It is useful in the preservation of plantation latex as it react with the enzymes responsible for the decomposition.[54]

V. Conclusion

Zinc Oxide is an evergreen multifunctional material which will give a various application in different field because of its many interesting property. It has the capacity to be obtained a variety of structure which allow be used as a new material in different domain. This review paper describe the various important and cost effective Physical, Chemical and Biological technique for synthesizing ZnO nano material. This paper presents various properties for its recent applications and after sometime for its innovative application. It covers a wide range of applications. The use of such materials can provide, among other things, more durable ceramics, transparent solar filters blocking infrared and ultraviolet radiation, and catalysts. These materials are also useful in biomedical research and in the diagnosis and treatment of diseases. They can be used to deliver medicines directly to diseased cells, in a way that avoids adverse effects. These are the various property which shows that ZnO proved it a useful and multifunctional material

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