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PROJECT REVIEW 3

SUBJECT: Introduction to Nanoscience and Nanotechnology (ECE1006)

TITLE: Synthesis and Characterization of Zno Nanoparticle

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SLOT: E1

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ABSTRACT:

Fabrication of novel sensing devices using Nano structured semiconducting metal oxide (SMO) is gaining prominence in research in recent years due to its notable properties and reduced cost. Among various SMO like ZnO, SnO_2, TiO_2, In_2O_3 etc. ZnO is one of the most promising nanomaterial due to its unique properties and when synthesized in various size and morphology can be used for various applications. In this paper, we report the synthesis and characterization of well aligned ZnO (Zinc Oxide) nanoparticles.

OBJECTIVE:

The objective of this study is to synthesize zinc oxide nanostructures with the most practical ways by using sol-gel method and characterize the nanostructures. The obtained ZnO nanoparticles are homogenous and consistent in size which corresponds to the XRD result that exhibit good crystallinity. These particles are then characterized using XRD.

This report deals with characterization of the prepared zinc oxide using X-Ray Diffraction in order to determine whether this method is more feasible in terms of particle agglomeration, particle size, particle separation.

The XRD results show that the synthesized ZnO samples have wurtzite structure.

The synthesis method has potential for application in manufacturing units due to ease processing and more economical reagents. Zinc oxide can be called a multifunctional material thanks to its unique physical and chemical properties.

INTRODUCTION:

Understanding the mechanisms of the human body at the molecular and nanometer scale has improved tremendously, but developments in therapeutic options for treating severe and debilitating diseases such as cancer and autoimmunity have lagged by comparison.

In this regard, nanomedicine, which is the application of nanotechnol-ogy to medical problems, can offer new approaches in therapy. The application of nanotechnology in biology requires further studies for the development of new materials in the nanosize range. These materials have many potential applications in biological science and clinical medicine.
One of the better-known materials that have been widely used for medical applications is zinc oxide nanoparticles (ZnO-NPs). It is not too far from the truth to say that the ZnO is a magic material because of its wide area of applications and flexibility of preparation
in different morphologies with different properties. Reflecting the basic properties of ZnO, fine particles of the oxide have deodorizing and antibacterial action, and for that reason are added into various materials including cotton fabric, rubber, and food packaging.
ZnO is widely used to treat a variety of other skin conditions, in products such as baby powder and barrier creams to treat diaper rashes, and in calamine cream, antidandruff shampoos, and antiseptic ointments.

It is also a component in tape (called “zinc oxide
tape”) used by athletes as a bandage to prevent soft tissue damage during workouts.

Therefore, several new routes have been developed to synthesize ZnO-NPs. These particles are then characterized to know more about their properties and confirmation of a crystalline nanoparticle.

LITERATURE SURVEY:

Zinc oxide nanoparticles (ZnO NPs), as one of the most important metal oxide nanoparticles, are popularly employed in various fields due to their peculiar physical and chemical properties . ZnO NPs are firstly applied in the rubber industry as they can provide wearproof of the rubber composite, improve performance of high polymer in their toughness and intensity and antiaging, and other functions . Because of the strong UV absorption properties of ZnO, they are increasingly used in personal care products, such as cosmetics and sunscreen. In addition, ZnO NPs have superior antibacterial, antimicrobial, and excellent UV-blocking properties. Therefore, in the textile industry, the finished fabrics by adding ZnO NPs exhibited the attractive functions of ultraviolet and visible light resistance, antibacteria, and deodorant. Apart from the applications mentioned above, zinc oxide can also be used in other branches of industry, including concrete production, photocatalysis, electronics, electrotechnology industries, and so on.

X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of the incident rays with the sample

produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin \theta$). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2θangles, all possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material. Conversion of the diffraction peaks to d-spacings allows identification of the mineral because each mineral has a set of unique d-spacings. Typically, this is achieved by comparison of d-spacings with standard reference patterns.

SYNTHESIS

CHEMICALS REQUIRED

- Zinc Acetate Dihydrate: [$Zn(O_2CCH_3)_2(H_2O)_2$][Zinc Acetate Dihydrate is used as a precursor]
- SodiumHydroxide :[NaOH]
- Ethanol: [C_2H_5OH] [Ethanol is used as a reagent]
- Distilled Wate:r [H_2O] [Distilled water is used as a solvent medium.

METHODOLOGY: SYNTHESIS [SOL GEL]

In order to prepare a sol, 2 g of Zinc Acetate Dihydrate and 8 g of Sodium Hydroxide are weighed using a weighing balance.

Then, 10 ml and 15 ml of distilled water are added to two measuring cylinders.
After that, 2 g of Zinc Acetate Dihydrate is dissolved in 15 ml of distilled water and 8 g of Sodium Hydroxide is dissolved in 10 ml of distilled water.

Both the solutions are stirred constantly for about five minutes each using a magnetic stirrer.
After constant stirring, the Sodium Hydroxide solution is poured into the Zinc Acetate Dihydrate solution facilitated with constant stirring by a magnetic stirrer for about five minutes.
Then, a burette is filled with 100 ml of Ethanol and titrated dropwise with the solution containing both Sodium Hydroxide solution and Zinc Acetate Dihydrate. After the reaction, white precipitate will be formed.

The solution is then centrifuged in a centrifugal machine for about 10 minutes at about 1500 RPM.
After centrifugation, the white precipitate thus formed is scraped off carefully and collected in a china dish.
The collected particles are put in an oven at about 100°C - 150°C for about 4-5 hours.

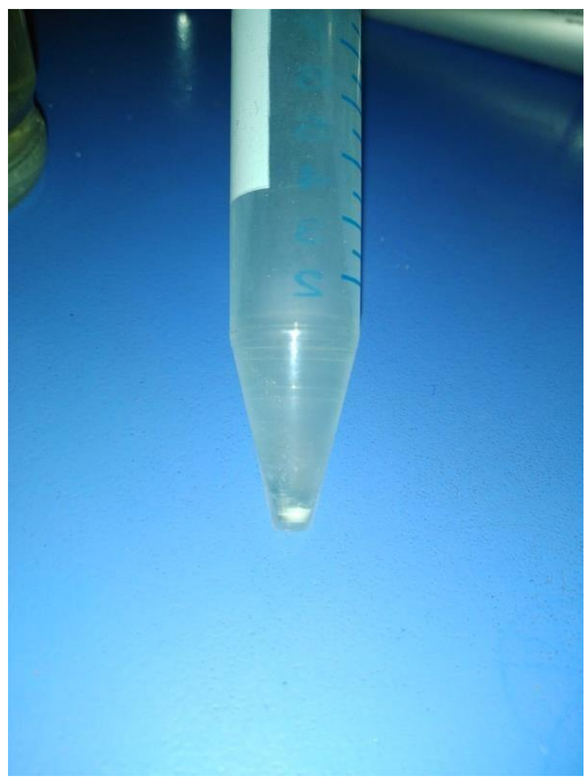
After heating, the final products are collected in a small tube and stored for further usage

The complete hydrolysis of Zinc Acetate with the aid of NaOH in an ethanolic solution should result in the formation of a ZnO colloid.

The final product is obtained as a result of the equilibrium between the hydrolysis and condensation reaction.
Due to the heating, Zinc Acetate within the solution undergoes hydrolysis forming acetate ions and zinc ions.
The abundance of electrons in the oxygen atoms makes the hydroxyl groups (-OH) of alcohol molecules bond with the zinc ions.

The overall chemical reaction to form ZnO nano-powder:
$Zn(O_2CCH_3)_2(H_2O)_2 + 2[NaOH]$ -- > ZnO +

$2NaCH_3COO + H_2O$ Nanoparticle is synthesized and characterized.



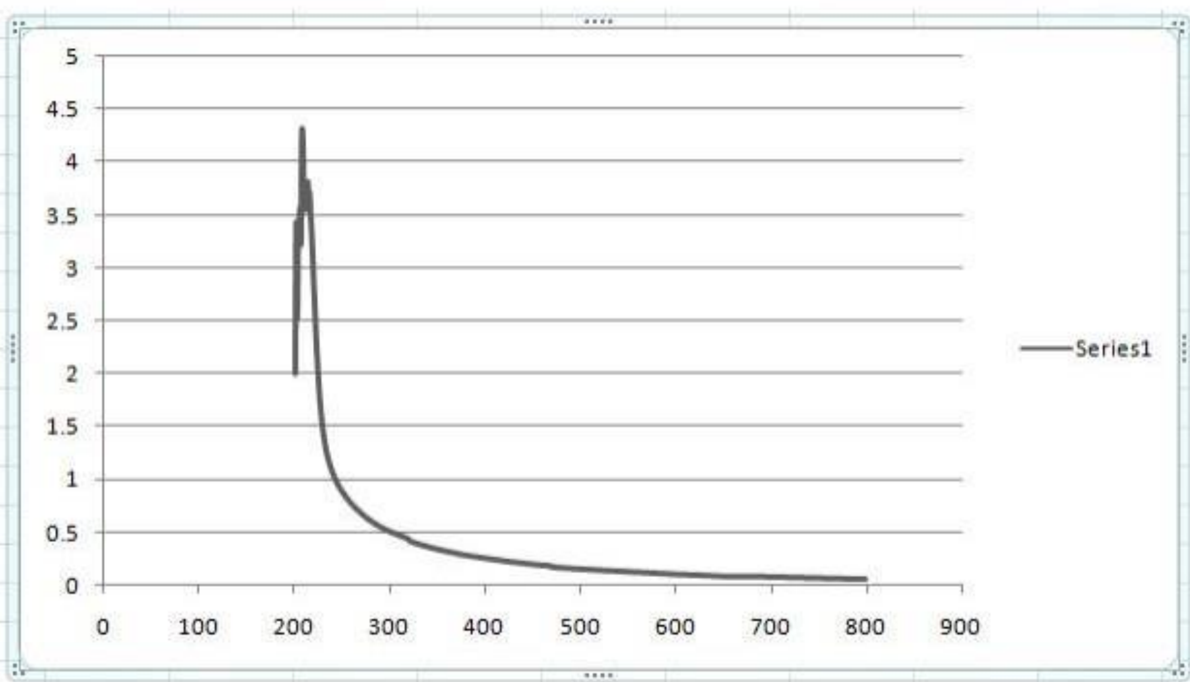
CHARACTERIZATION

METHODOLOGY: CHARACTERIZATION [UV]

UV spectrometer range is 200nm to 800nm.
The plot of absorbance vs wavelength is drawn.
UV spectroscopy is based on the principle of **Lambert's Law** and **Beer'sLaw**. It measures the intensity of light passing through a sample.

Fraction of light energy is absorbed by the molecules and the electrons jump to higher energy levels.
The Spectrometer records the degree of absorption, and a graph is plotted between absorbance and wavelength.
Applications of UV spectroscopy involves Detection of impurities,quantitative and Qualitative analysis of the compounds.

Graph



Values

Display	Absorbance	
Correction	Reference	
Slit	4 nm	
Lamp change at	320 nm	
Meas. mode	Spectral Scan	
Range [nm]	200 - 800	
Delta lambda [nm]	1	
Speed [nm/s]	50	

200	2.0043
201	3.4374
202	2.5313
203	3.2774
204	3.254
205	3.5771
206	3.2492
207	4.3136
208	4.2031
209	3.6928
210	3.6602
211	3.5581
212	3.6342
213	3.823
214	3.701
215	3.719

METHODOLOGY: CHARACTERIZATION [XRD]

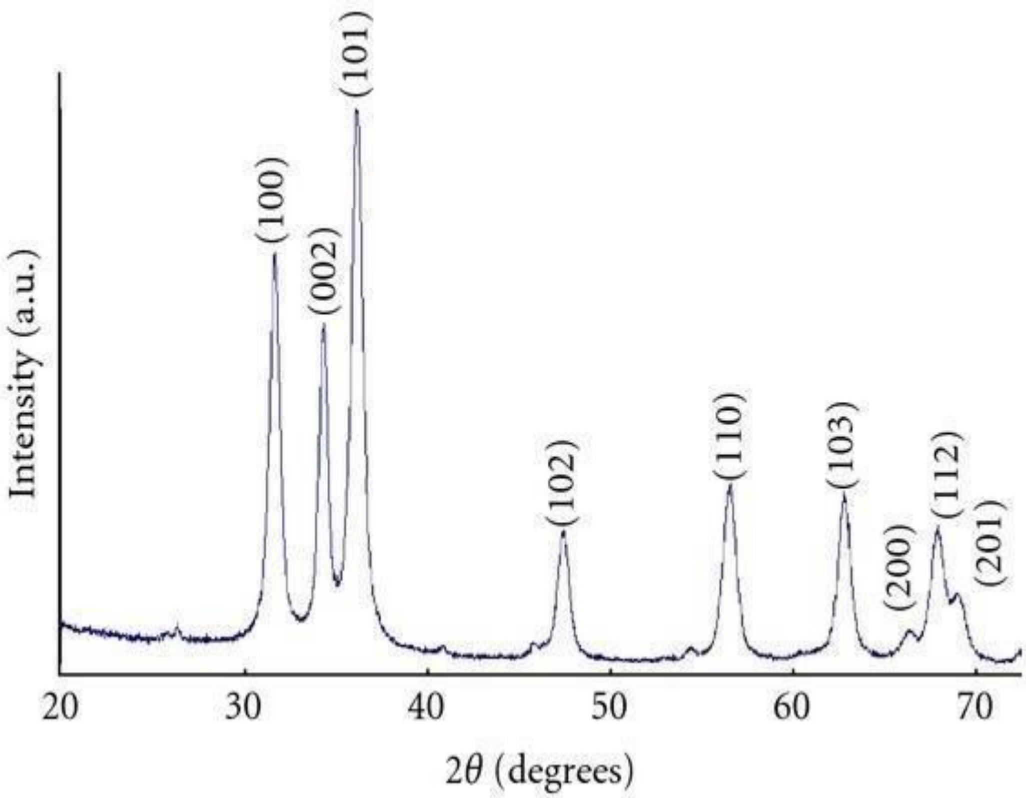
1. Zinc Oxide nanostructure was synthesized by using sol-gel method. Powder XRD measurements were performed using the X-ray diffractometer (Shimadzu XD-3A) in the diffraction angle ranges $20 \leq 2 \leq 80$ with monochromatic CuK[redacted] radiation □ (= 1.5418 ˚ A) source.

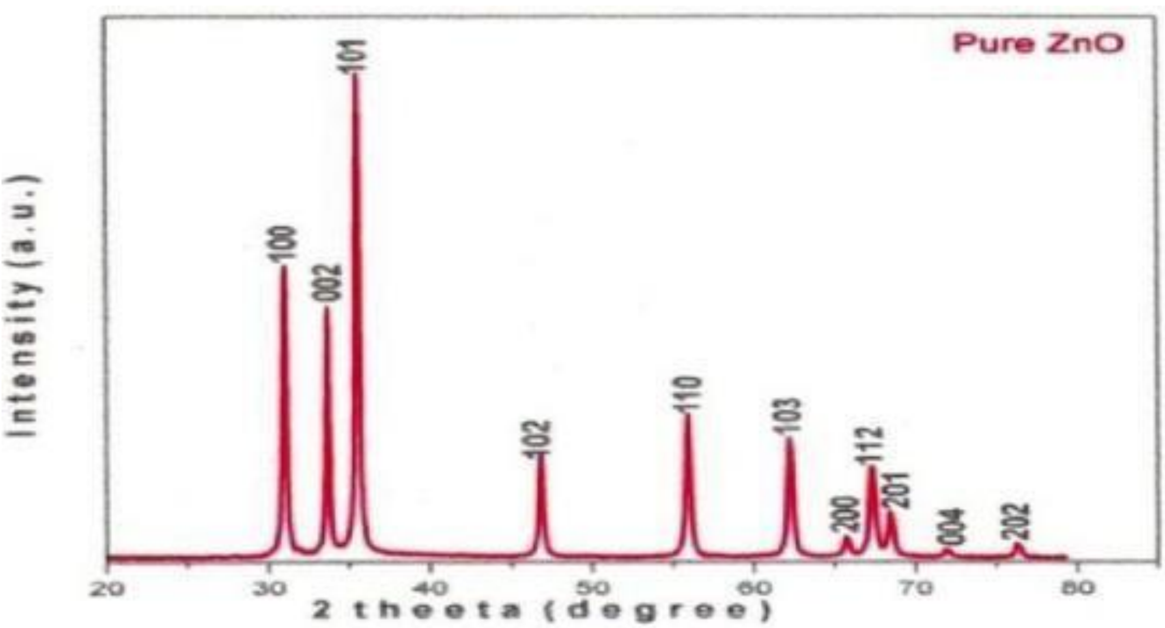
2. A definite line broadening of the XRD peaks indicates that the prepared material consist of particles in nanoscale range. From this XRD patterns analysis, we determined peak intensity, position and width, full-width at half-maximum (FWHM) data.

3.The diffraction peaks located at 31.84°, 34.52°, 36.33°, 47.63°, 56.71°, 62.96°, 68.13°, and 69.18° have been keenly indexed as hexagonal wurtzite phase of ZnO [28, 29] with lattice constants ==0.324 nm and =0.521 nm (JPCDS card number: 36-1451) [30], and further it also confirms the synthesized nanopowder was free of impurities as it does not contain any characteristics XRD peaks other than ZnO peaks.

4. The synthesized ZnO nanoparticle diameter was calculated using Debye-Scherrer formula [31]=0.89$\lambda\beta$cosθ,(1)where 0.89 is Scherrer's constant, λ is the wavelength of X-rays, θ is the Bragg diffraction angle, and β is the full width at half-maximum (FWHM) of the diffraction peak corresponding to plane $\langle 101 \rangle$.

5. The average particle size of the sample was found to be 16.21 nm which is derived from the FWHM of more intense peak corresponding to 101 plane located at 36.33° using Scherrer's formula.


(100)
(002)
(101)
(102)
(110)
(103)
(200)
(112)
(201)
Intensity (a.u.)
20
30
40
50
60
70
2θ (degrees)


Pure ZnO
100
002
101
102
110
103
200
112
201
004
202
Intensity (a.u.)
20
30
40
50
60
70
80
2 theeta (degree)

S No.	d=λ/2sinθ (Observed)	d=λ/2sinθ (Reported)	I/Io*100 % (Observed)	I/Io*100 % (Reported)
1	2.4779	2.4786	100	100
2	2.8165	2.8179	59.8	57.8
3	2.60485	2.6049	83.33	44.2
4	1.62572	1.6269	24.40	32.4
5	1.47777	1.4784	23.31	27.6
6	1.37912	1.3799	16.62	24.3
7	1.91202	1.9128	20.77	22.9
8	1.35934	1.3601	7.55	11.4

- In X-ray diffraction, some prominent peaks were considered and corresponding d-values were compared with the standard values. X-ray diffraction shows that metal oxide is pure ZnO having hexagonal structure.

- The broadening of the X-ray diffraction lines, as seen in the other figure reflects the nano-particle nature of the sample. Sharpness of the peaks shows good crystal growth of the oxide particles

RESULTS AND ANALYSIS:

ZnO nanoparticles have been prepared using wet chemical synthesis method and were characterized by XRD. ZnO nanoparticles have been prepared using wet chemical synthesis method and were characterized by XRD. These ZnO nanoparticles can be used in different industrial applications, namely, luminescent material for fluorescent tubes, active medium for lasers, sensors, and so forth.

The XRD results show that the ZnO-NPs were formed in a hexagonal structure with crystalline size of 33 ±2 nm.

APPLICATIONS

1. Zinc oxide is used in the manufacture of rubber and cigarettes (used as a filter).
2. Popularly known calamine lotion is made out of zinc oxide powder. It is also used in a host of other creams and ointments that are used to treat skin diseases.
3. As an additive in the manufacture of concrete.
4. Ceramic industry has a number of uses for zinc oxide powder.
5. It is also used as an additive in food products such as breakfast cereals.
6. Various paints use zinc oxide as a coating agents

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