ADEPT SYNTHESIS OF ZnO NANOPARTICLES USING DIRECT PRECIPITATION METHOD FOR PHOTOCATALYSIS

[a]Carolin Kiruba. E, Keruthika. K[b], **Ilavarasi. R, ** Naveen Rooba Doss. M

Nanotechnology Division, Department of Electronics and Communication Engineering, Periyar Maniammai Institute of Science and Technology, Thanjavur, Tamilnadu, India.

ABSTRACT:

Photo catalysis a green technology that convert photonic energy to utilitarian chemical energy with rapid rise of photo reactor flow. The design and development of novel semiconductor photo catalysis is emphasized; inspite of other photo catalyst notably titanium dioxide and molybdenum, ZnO has been the prominent photo catalyst due to lower cost and potentially immense photocatalytic activity, border energy gap of 3.2ev exhibit lower photocatalytic rate can be enhanced by modifying ZnO to nanoscale range. In this work the ZnO Nanoparticle were fabricated by direct precipitation method and obtained nanoparticle was characterized by UV-Vis Spectroscopy which confirmed ZnO NP presence at the wave length range of 235nm with calcinations temperature 450^0 C and SEM analysis witness the morphology of ZnO NP on its surface at1000X and 1,50,000X.

KEYWORDS: ZnO nanoparticles, direct precipitation, photo catalysis.

1. INTRODUCTION:

Zinc oxide is an N - type semiconducting metal oxide, it retains exemplary mechanical property, electrical property, biomedical systems, etc. Now a days, several type of inorganic metal oxides have been synthesized like TiO_2, CuO, ZnO etc. From these inorganic metal oxides, ZnO has much interest because they are inexpensive to produce, safe and can be prepared easily. The preparation expense of Zinc oxide is up to 75% lower than other inorganic metal oxide like TiO_2[1,2]. ZnO having tremendous semiconducting property with because of its direct large band gap width (3.37eV) and high exciton binding energy (60meV) [3,4]. In addition, zinc oxide has anti-inflammatory property and wound healing property along with good photocatalytic activity, UV filtration property [5].

It is used in cosmetic products like sunscreen lotion, and UV filter sunglasses etc. It has also been used for wide range of biomedical application like drug delivery, anti-bacterial, anti-cancer, etc. [1]. Various methods for synthesizing ZnO nanoparticle has been proposed, such as direct precipitation [6-9], thermal composition [10],

chemical vapor deposition [11], spray pyrolysis [12], sol gel [13,14], for nanocrystalline zinc oxide have been proposed, such as solution precipitation, microemulsion, non microemulsion, ultrasonic radiation precipitation method [9]. Among these methods, precipitation approach while compared to other traditional methods provide facile way for large scale production and low cost. In this precipitation method does not need expensive raw material and complicated equipment [6,8].

The direct precipitation method is simpler than the other precipitation methods like homogeneous precipitation, solution precipitation etc. In direct precipitation method, the raw materials used are cheaper. Many researchers have utilized several methods to prepare ZnO nanoparticles using direct precipitation method like adding surfactants or using ultra sound field. In this work direct precipitation method is engaged to synthesize nano sized ZnO particle using urea as one of the reactants.

India. Urea (CH_4N_2O) was a common fertilizer which is used for agriculture. According to methodology all synthetic compounds are dissolved with distilled water to prepare precursor solution.

2.EXPERIMENTAL SECTION:

2.1 MATERIALS:

Synthesis of Zinc Oxide nanoparticles by direct precipitation method, in this research we use zinc sulfate ($ZnSO_4$), Sodium hydroxide (NaOH) were purchased from Eswarr Scientific & Co in Tamil Nadu,

International Journal of Scientific Research in Engineering and Management (IJSREM)
Volume: 05 Issue: 05 | May - 2021 ISSN: 2582-3930

2.2 SYNTHESIS PROCESS:

Synthesis strategy including the following steps, originally the urea solution is added in to stirring tank containing the zinc sulfate solution under room temperature. Then the urea is gradually decomposed into CO_2 and NH_3 and the chemical reaction take place. At last white precipitation is formed after calcination, then ZnO nanoparticles will be obtained. Diffuse 0.6 grams of Zinc sulfate and 0.2 g of urea in 100 ml of water. Gradually shake or mix the solution until the solution become completely transparent. Again, add few drops (2-4) of sodium hydroxide to the above solution and adjust the pH to 12. The solution will change into a whitish shady precipitate. At that point filter the solution using filter paper, and keep it in hot air oven at 350^0 C until the water molecules removed completely.

3.FLOW CHART:

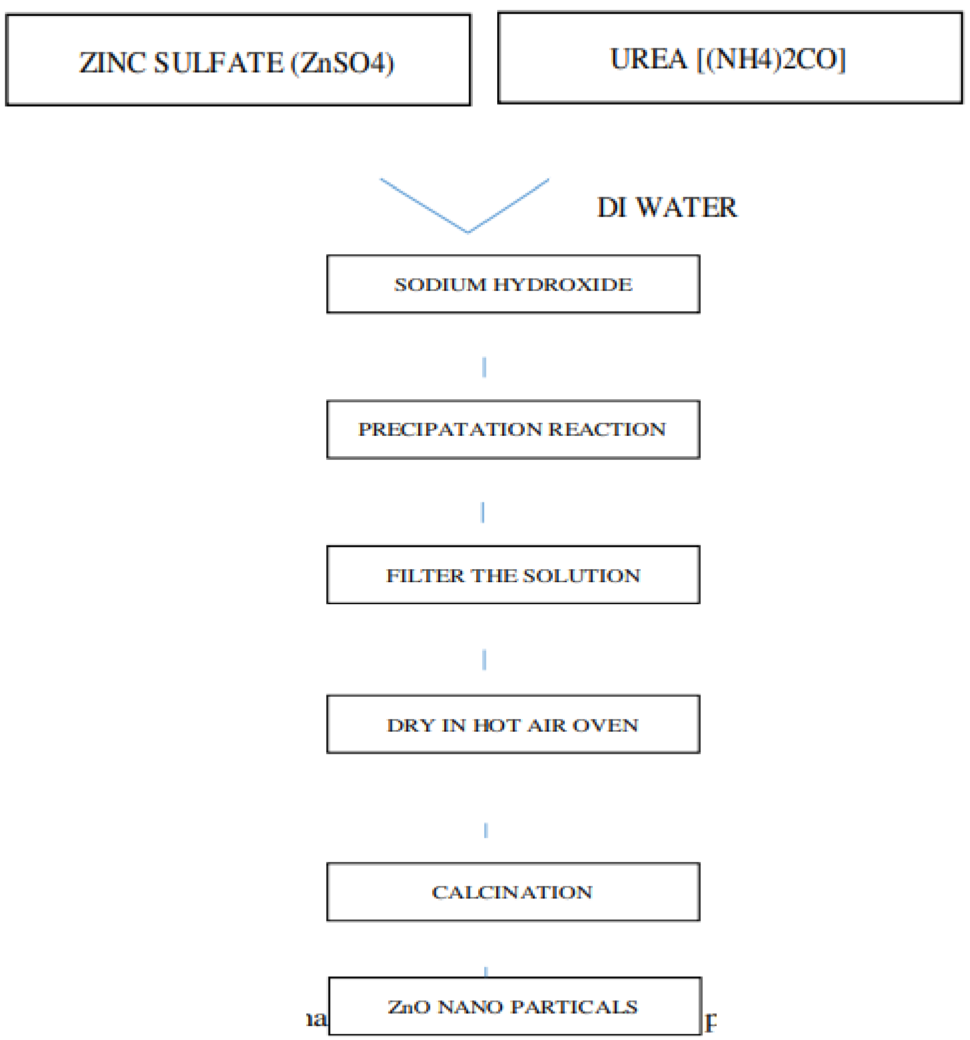


Fig 1: This flow chart represents the synthesis process of ZnO nano DI WATER

3. REACTION PROCESS:

Zinc sulfate will detonate zinc and urea provided oxygen to make zinc oxide (ZnO). This zinc sulfate is naturally salt and it included to the DI water and it separate's in to zinc (II) ion and sulfate ion. Urea is not a salt and simply get dissolved in the solution and float around. At a appropriate point small amount of sodium hydroxide is wrapped in the solution and hydroxyl groups in the solution shoots up. These

hydroxyl groups will respond with zinc (II) ion to make zinc hydroxide **$Zn(OH)_2$**. Add some drops of sodium hydroxide in the

solution until white precipitate is formed. After, the solution becomes white precipitate, it is exposed to the microwave. The zinc oxide nanoparticle is collected in white color fine powder form. And second, and more strangely, urea begins to decompose at higher temperatures giving out ammonia and carbon dioxide. In, this way, composed ammonia (weak base) diffuse back in to the water and make more hydroxyl groups.

Hydroxyl groups in the solution react with zinc (II) ions to make zinc hydroxide. Finally, in this previous process zinc hydroxyl are changed in to ZnO below thermal condition providing more cluster of zinc oxide crystals. By using microwave has used in previous process technique are used in given solution are large burst energy are increasing its temperature at very high rate. This outcomes in an enormous number of very small nanocrystals called cores which will self-assemble to make nanoparticles. However, if general warming is utilized, the decay of urea is moderately slow delivering small number of cores for longer durations. These will self-assemble and develop into bigger particles because of continues supply of growth materials. This is the reason behind why we can expect bigger particles of Zno if general heating is used [15 – 18].

$$(NH_4)_2CO \xrightarrow{\text{Heat}} CO_2+NH_3$$

$$NH_3+H2O \longleftrightarrow NH_3\cdot H_20$$

$$NH_3.H_2O \longleftrightarrow NH_4^{+}+OH^{-}$$

$$Zn^{2+}+2OH^{-} \longleftrightarrow Zn\ (OH)_2$$

$$Zn\ (OH)_2 \longrightarrow ZnO+H_2O^{-}$$

In visual observation, the color doesn't change were simply observed when chemicals were added. These white precipitations were captured by using a camera.

4.CHARACTERIZATION:

4.1 UV-VISIBLE SPECTROSCOPIC:

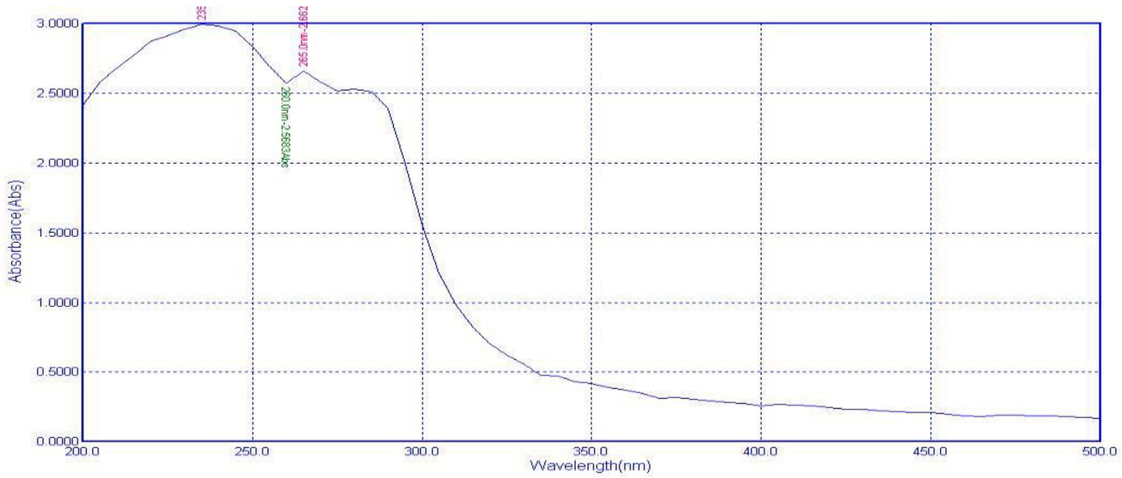


Fig 2: UV – Visible Spectra of ZnO nanoparticles is confirmed by showing the peak value at 235 nm.

From UV- visible spectra examined the sample of ZnO particles was ready by scattering in ethanol. The stable absorption peak in fig2 represents to ZnO nanoparticle within the wavelength of 235 nm. This absorption peak corresponds to ZnO sample calcined at a temperature of 450^0 C. In between valence band to conduction band the electrons square measure transfer by this method is called electron transition and it form gap. This gap is point out as intrinsic band gap. Through this intrinsic band gap UV are absorb by using wavelength at 235nm by this we confirmed the presence of ZnO nano particle. The band gap (Eg) of ZnO nanoparticles was determined by utilizing the recipe

Eg=hc/λ.

Where:

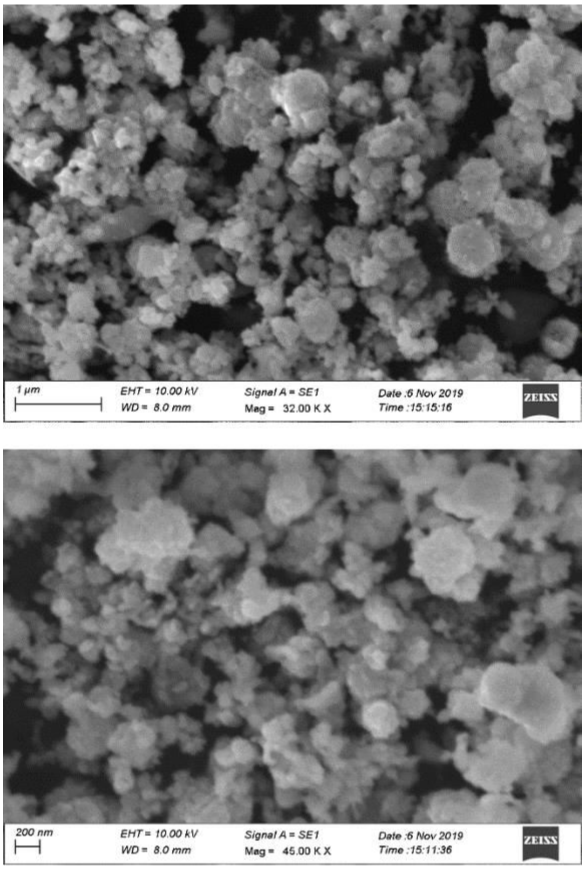
h=plank's steady;

c= speed of light and

λ= **frequency.** The relating band gap was seen as 3.27 eV

[19-21].

4.2 SEM ANALYSIS:



(a)

(b)

Fig 3: Electron photomicrographs of ZnO nanoparticles (a) 1000X magnification & (b) 150000 X magification

In SEM results evaluated the intensity of particle and arrangement of ZnO particles which is represented in fig 3 (b). We observed that the particles were agglomerated with small spherical shaped and none uniform structure. The arrangement of ZnO nanoparticle in fig 3 (a) shows high compactness. This formation leads to some nanopores inbetween the particles. This same work was did by Samira Bagheri et al using Zinc nitrate as precursor shows smoother and more aggregated of ZnO nanoparticles[22].

5.APPLICATION:

5.1. PHOTOCATALYSIS

ZnO nanoparticle is used for photocatalysis activity in various forms such as nano powders, scaffolds, nanowires, nanorods, etc. Some organic pollutants such as methylene blue, monocrotophos,

diphenylamine are treated for photocatalytic decomposition using radially oriented ZnO nanowires using poly-L**-lactide** nanofibers for

applying water purification[23]. The result shown here is reduction of pathogenic bacterial densities by the photocatalytically active ZnO nanorods in the presence of visible light implies potential *ex situ* application in water decontamination at ambient conditions under sunlight[24]. The photodegradation of methylene blue and methyl orange was done in the presence of paper filled with ZnO nanorods under visible light[25]. The result demonstrate that the photodegrading of organic contaminants in aqueous solution is done

by immersing ZnO thin film into that aqueous solution[26]. The enhanced result was found from the undoped ZnO nanoparticles for photocatalyst[27]. The result of ZnO nanoparticle annealed at 600^{0} C possess complete photodegradation of the DR – 23 dye[28]. The result shows that Co- doped ZnO nanoparticle performs higher photocatalyst than Mn- doped ZnO nanoparticle[29]. The result of the work is the Ag-ZnO nanoparticle provide super high photocatalyst than ZnO nanoparticle[30].

6.CONCLUSION:

In this work, the ZnO nanoparticles were austiciously synthesized by direct precipitation method using Zinc sulfate as zinc source and urea as precipitating agents in aqueous solution. In UV – visible analysis, the absorption peak range of the generated ZnO nanoparticle at wavelength 235 nm. The SEM analysis shows that the particles surface morphology was small spherical structured.

ACKNOWLEDGMENT:

I would like to thank Nanotechnology Division, Department of Electronics and Communication Engineering of Periyar Maniammai Institute of Science and Technology for providing necessary materials and supports for do this work.

REFERENCE:

1) Ong, Chin Boon, Law Yong Ng, and Abdul Wahab Mohammad.
2018. "A Review of ZnO Nanoparticles as Solar Photocatalysts: Synthesis, Mechanisms and Applications." Renewable and Sustainable Energy Reviews 81(March 2017): 536–51.

2) Agarwal, Happy, S. Venkat Kumar, and S. Rajeshkumar. 2017. "A Review on Green Synthesis of Zinc Oxide Nanoparticles – An Eco-Friendly Approach." Resource-Efficient Technologies 3(4): 406–13.

3) Sundrarajan, M., S. Ambika, and K. Bharathi. 2015. "Plant-Extract Mediated Synthesis of ZnO Nanoparticles Using
Pongamia Pinnata and Their Activity against Pathogenic Bacteria." Advanced Powder Technology 26(5): 1294–99.

4) Anbuvannan, M. et al. 2015. "Synthesis, Characterization and
Photocatalytic Activity of ZnO Nanoparticles Prepared by Biological Method." Spectrochimica Acta - Part A: Molecular and Biomolecular Spectroscopy 143: 304–8.

5) Gharoy Ahangar, Elham et al. 2015. "Preparation and Characterization of PVA/ZnO Nanocomposite." Journal of Food Processing and Preservation 39(6): 1442–51.

6) Moharram, A. H., S. A. Mansour, M. A. Hussein, and M. Rashad. 2014. "Direct Precipitation and Characterization of ZnO Nanoparticles." Journal of Nanomaterials 2014.

7) Wang, Yujun, Chunling Zhang, Siwei Bi, and Guangsheng Luo.
2010. "Preparation of ZnO Nanoparticles Using the Direct Precipitation Method in a Membrane Dispersion Micro-Structured Reactor." Powder Technology 202(1–3): 130–36.

8) Chen, Chang Chun, Ping Liu, and Chun Hua Lu. 2008. "Synthesis and Characterization of Nano-Sized ZnO Powders by Direct Precipitation Method." Chemical Engineering Journal 144(3): 509–13.

9) Siqingaowa, Zhaorigetu, Hongxia Yao, and Garidi. 2006. “Preparation and Characterization of Nanocrystalline ZnO by Direct Precipitation Method.” Frontiers of Chemistry in China 1(3): 277–80.

10) Yang, Yang, Huilan Chen, Bin Zhao, and Ximao Bao. 2004. “Size Control of ZnO Nanoparticles via Thermal Decomposition of Zinc Acetate Coated on Organic Additives.” Journal of Crystal Growth 263(1–4): 447–53.

11) Purica, M. et al. 2002. “Optical and Structural Investigation of ZnO Thin Films Prepared by Chemical Vapor Deposition (CVD).” Thin Solid Films 403–404: 485–88.

12) Ayouchi, R. et al. 2003. “Preparation and Characterization of Transparent ZnO Thin Films Obtained by Spray Pyrolysis.” Thin Solid Films 426(1–2): 68–77.

13) Qian, Dong et al. 2004. “Comment on ‘Catalysis and Temperature Dependence on the Formation of ZnO Nanoparticles and of Zinc Acetate Derivatives Prepared by the Sol-Gel Route.’” Journal of Physical Chemistry B 108(39): 15434–37.

14) Song, Rui, Ying Liu, and Linghao He. 2008. “Synthesis and Characterization of Mercaptoacetic Acid-Modified ZnO Nanoparticles.” Solid State Sciences 10(11): 1563–67.

15) Comparelli, Roberto, et al. "UV-induced photocatalytic degradation of azo dyes by organic-capped ZnO nanocrystals immobilized onto substrates." Applied Catalysis B: Environmental 60.1-2 (2005): 1-11.

16) Kumar, S. Girish, and KSR Koteswara Rao. "Zinc oxide based photocatalysis: tailoring surface-bulk structure and related interfacial charge carrier dynamics for better environmental applications." Rsc Advances 5.5 (2015): 3306-3351.

17) Mittal, Neeraj. Synthesis and Photocatalytic Studies of ZnO Nanoparticles. Diss. 2010.

18) Amer, Hussam Ahmad Abdullah. ZnO nano-particle catalysts in contaminant degradation processes with solar light naked and supported systems. Diss. 2012.

19) Ghorbani, Hamid Reza, et al. "Synthesis of ZnO nanoparticles by precipitation method." Oriental Journal of Chemistry 31.2 (2015): 1219-1221.

20) Hoffmann, Michael R., et al. "Environmental applications of semiconductor photocatalysis." Chemical reviews 95.1 (1995): 69-96.

21) Yu, Jiaguo, and Xiaoxiao Yu. "Hydrothermal synthesis and photocatalytic activity of zinc oxide hollow spheres." Environmental science & technology 42.13 (2008): 4902-4907.

22) Bagheri, Samira, K. G. Chandrappa, and SB Abd Hamid. "Facile synthesis of nano-sized ZnO by direct precipitation method." Der Pharma Chemica 5.3 (2013): 265-270.

23) A. Sugunan, V. K. Guduru, A. Uheida, M. S. Toprak, and M. Muhammed, "Radially oriented ZnO nanowires on flexible poly-L-lactide nanofibers for continuous-flow photocatalytic water purification," *Journal of the American Ceramic Society*, vol. 93, no. 11, pp. 3740–3744, 2010.

24) A. Sapkota, A. J. Anceno, S. Baruah, O. V. Shipin, and J. Dutta, "Zinc oxide nanorod mediated visible light photoinactivation of model microbes in water," *Nanotechnology*, vol. 22, no. 21, Article ID 215703, 2011.

25) Baruah, S., Jaisai, M., Imani, R., Nazhad, M.M. and Dutta, J., 2010. "Photocatalytic paper using zinc oxide nanorods." *Science and Technology of Advanced Materials*.

26) Hariharan, C. "Photocatalytic degradation of organic contaminants in water by ZnO nanoparticles: Revisited." *Applied Catalysis A: General* 304 (2006): 55-61.

27) Kaur, Japinder, and Sonal Singhal. "Facile synthesis of ZnO and transition metal doped ZnO nanoparticles for the photocatalytic degradation of Methyl Orange." *Ceramics international* 40.5 (2014): 7417-7424.

28) Umar, Ahmad, et al. "Effect of annealing temperature on the properties and photocatalytic efficiencies of ZnO nanoparticles." *Journal of Alloys and Compounds* 648 (2015): 46-52.

29) Saleh, Rosari, and Nadia Febiana Djaja. "Transition-metal-doped ZnO nanoparticles: synthesis, characterization and photocatalytic activity under UV light." *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 130 (2014): 581-590.

30) Liu, Yutong, et al. "Novel and efficient synthesis of Ag-ZnO nanoparticles for the sunlight-induced photocatalytic degradation." *Applied surface science* 476 (2019): 632-640.