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CHEMI-THERMAL METHOD OF SYNTHESIS OF ZnO NANOMATERIAL AND ITS CHARACTERIZATION

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ABSTRACT

Present study reports of synthesis of better yield ZnO nanomaterial by chemi-thermal route and its characterization. Characterization of zinc oxide powder was analyzed by using UV-Visible Spectroscopy, SEM and EDX. The absorption spectrum was found to be at 369nm and its band energy about 3.9 eV. SEM results showed aggregated pebble like morphology with average size about 100 nm at high magnification. EDX results showed the presence of major amount of Zn^{2+} and O^{2-} elements with respect to ZnO molecule by both weight and atomic percentage. Therefore, this method can be used as an alternative for other chemical synthesis method aimed at various applications.

KEYWORDS: Chemi-thermal, Sodium lauryl sulphate, Simple and robust method, ZnO nanomaterial.

The unique properties of nanomaterials such as quantum size effect, surface to volume ratio, modifications in surface morphology etc., attracted researchers to work on nanomaterial fabrication for applications in different areas [1,2]. Although there are numerous types of nanomaterials available, nanomaterial of metal oxides continuously made good progress in the field of nanotechnology because metal element has high compatibility with oxygen and develop stable compound which shows several geometrical forms leads to change in band gap and binding energy of particular structure [3]. Due to energy change and nanodimensional metal oxide materials acquire superior electronic properties and high density on edge surface sites respectively [4] compare to their bulk counterparts. Amongst all other metal oxides, zinc oxide nanomaterial attracts a greater attention because of its physical, chemical and biological properties. Fundamentally, Zinc oxide is a heterogeneous semiconductor n-type compound found in two main stable forms viz. wurtizite with hexagonal crystalline structure and zinc-blende with cubic crystalline structure where, anionic zinc atoms covalently bonded (Sp3 Hybridization) with four cationic oxygen atoms (tetrahedron) and eventually formed tetrahedral structure (Figure 1.) [5]. Further research showed ionic bonding in ZnO added an extra energy to increase the band gap up to 3.37eV at room temperature with quite large exciton binding energy of 60 meV[4,6]. Depending on requirements and applications, ZnO nanomaterial can be synthesized by physical and/or chemical methods. Physical methods are usually top down approach of synthesis includes thermal reduction [7-8] thermal evaporation [9] laser ablation [10] UV irradiation [11] etc., gives high yield of pure material since, no other chemical additives involved in it. Compared to physical method, chemical synthesis includes hydrothermal [12-15] sol-gel method [16-18] microwave irradiation [19-21] solvothermal method [22-24] Electro-chemical method [25] etc., are cost effective, simple and robust, but gives material with some impurities.

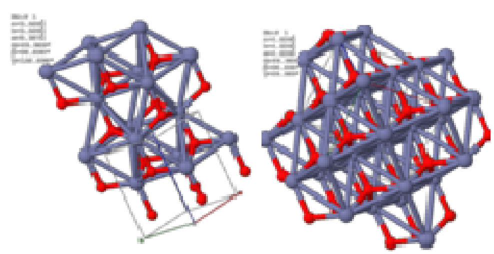

Figure 1: Crystal structure of ZnO wurtizite [Left] ZnO-zinc blende [Right] [5]

Generally, ZnO nanostructures shows improved chemical, physical, mechanical, electrical properties, biological activity [11,26-32] and hence in present study proposed the chemi-thermal method of synthesis of ZnO nanomaterials for better yield.

MATERIALS AND METHODS

Chemicals

All the bulk chemicals, solvents used in this study were analytical grade and purchased from suppliers including SRL chemicals and Qualigenes India.

Synthesis of ZnO nanomaterial

100 mM of $Zn(CH_3COO)_2$ were dissolved in 50mL of DI water by continuous steering on magnetic stirrer at 210^0C for 30 minutes. Drop wise addition of

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100ml mixture of 500 mM of NaOH and 10 mM Sodium Lauryl Sulfate (as a capping agent) was carried out with the help of burette into the reaction mixture at 210^0C.The resultant mixture was washed tree times with DI water and then with absolute ethanol followed by one last wash with DI water at 6000 rpm for 15 minutes in centrifuge. The supernatant after each wash were discarded and dried those pallets at 60^0 C followed by calcination at 550^0 C in muffle furnace for 6 hours. The resultant powder was used for further characterization.

Characterization of ZnO nanomaterial

The structural, morphological features and elemental composition of resultant powder were characterized by UV-Vis spectroscopy, SEM and EDX respectively for identifying various crystalline structures present in the powders as well as their morphological and optical information.

RESULTS AND DISCUSSION

Synthesis of ZnO nanomaterial:

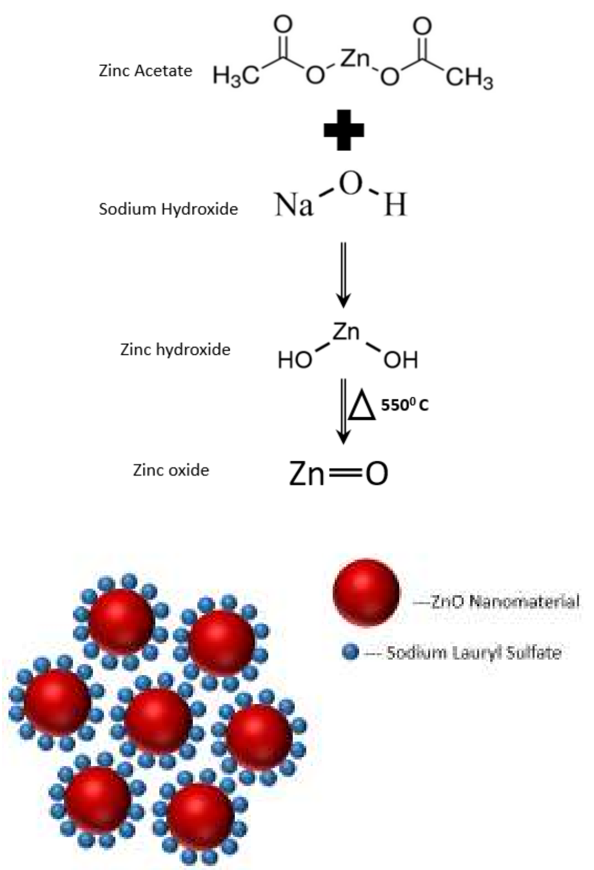

Figure 2: Net reaction of ZnO nanomaterial(top). Capping of SLS on ZnO nanomaterial(bottom)

Chun-Hong Kuo et al proposed SLS as a capping agent to control the growth and size of the nanoparticles [33]. Similarly, in this study SLS used as capping agent to control nucleation and growth of resultant ZnO nanomaterial by capping the surface.

UV- visible absorbance widely used to study optical properties of nanomaterials [34]. Absorption and emission spectra of synthesized material was measured by UV-Vis spectroscopy. (Thermo Scientific UV-10) Further ZnO dry powder was diluted in solvent to get absorption spectra. and its optical band gap was calculated accordingly. Absorption spectrum of ZnO powder is shown in fig.3. The maximum absorption was found at 369.8nm and band gap value has calculated to be 3.9eV due to quantization effect. W.-S. Chin et al found that the maximum absorption of ZnO nanomaterials was at 370nm [35].

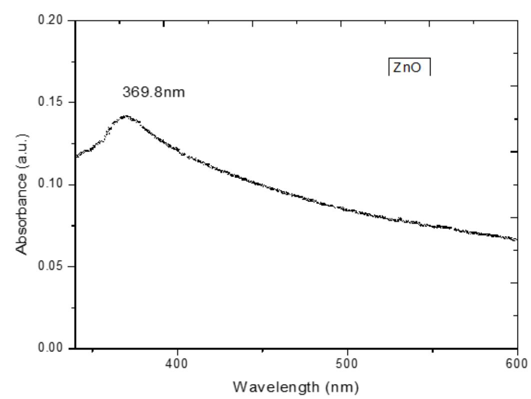

Figure 3: UV-Vis Spectroscopy analysis

SEM cum EDX analysis was conducted on synthesized material to understand the morphology and elemental analysis. The SEM images of the synthesized nanostructures are shown in fig.4. The low magnification image (right side) shows aggregation of nanostructured materials. The size of the nanostructures was found to be at ~1µm. However, as shown in higher magnification image at 100nm (Figure 3b), each nanostructures are made up of pebble like morphology with size of 100 nm.

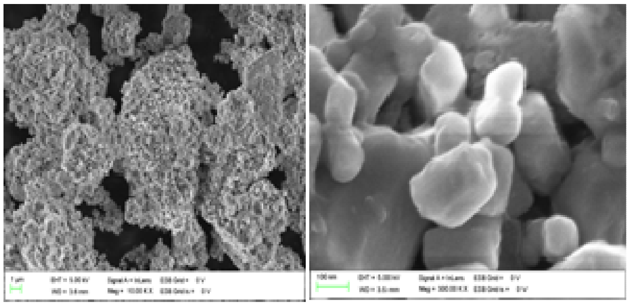

Figure 4: SEM images of ZnO nanomaterials (Right) Low magnification, (Left) High magnification.

The elemental composition of powder clearly studied by EDX (Fig.5), which shows that the major part of the synthesized material contains Zn^{2+} and O^{2-} with expected ratio for ZnO molecules (Table 1).

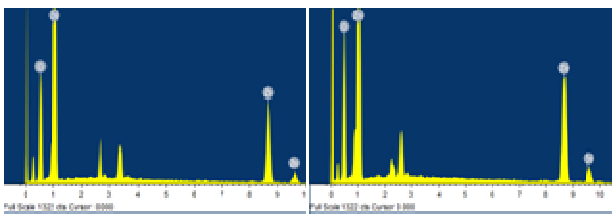

Figure 5: EDX results of ZnO Nanomaterial

Table 1: Composition of synthesized ZnO powder by EDX analysis

Elements	Weight[%]	Atomic[%]
O K	26.00	58.94
Zn K	74.00	41.06
Totals	100.00	100%

Obtained pure ZnO powder by sol-gel method of synthesis was further characterized by EDX suggested that the ZnO powder has high purity where Zinc content was found to be 55.38% and that of Oxygen 44.62%[36]. In present study the results obtained from the EDX characterization suggested that the ZnO powder has high purity (Zinc content – 41.06%; Oxygen content –58.94%), in which very little impurities can be seen. Tentatively, estimated stoichiometric mass percent of Zn and O are 80.3% and 19.7% [37].

CONCLUSION

ZnO nanomaterial has been synthesized by chemi-thermal method using zinc acetate and sodium hydroxide in presence of capping agent Sodium Lauryl Sulphate to control the size of ZnO nanomaterial. Characterization by UV-visible spectroscopy, SEM and EDX results confirms the band energy, morphology and elemental analysis belongs to ZnO nanomaterial. This method is robust and cost effective, can be used for industrial production of ZnO nanomaterials for various applications.

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REFERENCES

Hadis Morkoç and ÜmitÖzg€ur, in :Zinc Oxide: Fundamentals, Materials and Device Technology. Copyright #54 2009 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim ISBN: 978-3-527-40813-9.

Kumar, Narendra, and Sunita Kumbhat. Essentials in Nanoscience and Nanotechnology. Chapter:1 John Wiley & Sons, 2016.

Fernández□García M, Rodriguez JA. Metal oxide nanoparticles Nanomaterials: inorganic and bioinorganic perspective. Wiley 2008.

Özgür, Ü., Alivov, Y.I., Liu, C., Teke, A., Reshchikov, M., Doğan, S., Avrutin, V.C.S.J., Cho, S.J. and Morkoc, H.,. A comprehensive review of ZnO materials and devices. Journal of applied physics, 98(4), (2005), p.11.

Ersson, Kristin", "Materials Data on ZnO by Materials Project", https://materialsproject.org/docs/ calculations".

ISadraei R Simple Method for Preparation of Nano-sized ZnO Research & Reviews: Journal of Chemistry RRJCHEM, Volume 5, Issue 2, July, 2016.

Devaramani, Basavaraj S., Babu A. Manjasetty, and T. R. Nair. "The Novelty of Syntheses & Varied Applications of ZnO nano systems." arXiv preprint arXiv: 1002. 0199 (2010).

Pourrahimi, A. M., D. Liu, V. Ström, Mikael S. Hedenqvist, Richard T. Olsson, and Ulf W. Gedde. "Heat treatment of ZnO nanoparticles: new methods to achieve high-purity nanoparticles for high-voltage applications." Journal of Materials Chemistry A 3, no. 33 (2015), p. 17190-17200.

Wang, Zhong Lin. "Zinc oxide nanostructures: growth, properties and applications." Journal of Physics: Condensed Matter 16, no. 25 (2004), p.829–858.

R.K. Thareja *, Shobha Shukla. Synthesis and characterization of zinc oxide nanoparticles.by laser ablation of zinc in liquid. Applied Surface Science 253 (2007) 8889–8895

PRAUS, Petr, Jonáš TOKARSKÝ, and Ladislav SVOBODA. "Contribution to synthesis of zno nanoparticles by uv irradiation-assisted precipitation."

Wang, Zhong Lin. "Splendid one-dimensional nanostructures of zinc oxide: a new nanomaterial family for nanotechnology." Acs Nano 2, no. 10 (2008), p. 1987-1992.

HaiYan Xu,Hao Wang*, YongCai Zhang, WenLiang He, ManKang Zhu, Bo Wang, Hui Yan Hydrothermal synthesis of zinc oxide powders with controllable morphology. Ceramics International 30 (2004), p.93–97.

Dalia Ahmed Mohammed Osman , Mustafa Abbas Mustafa, Synthesis and Characterization of Zinc Oxide Nanoparticles using Zinc Acetate Dihydrate and Sodium Hydroxide. Journal of Nanoscience and Nanoengineering Vol. 1, No. 4, (2015), p. 248-251.

G. Amin, M. H. Asif, A. Zainelabdin, S. Zaman, O. Nur, and M. Willander Influence of pH, Precursor Concentration,Growth Time, and Temperature on the Morphology of ZnO Nanostructures Grown by the Hydrothermal Method Journal of Nanomaterials Volume (2011), p. 9.

Park, Seong Kyong, Jae Hyoung Park, Ki Young Ko, Sungho Yoon, Kyo Seon Chu, Woong Kim, and Young Rag Do. "Hydrothermal− electrochemical synthesis of ZnO nanorods." Crystal Growth and Design 9, no. 8 (2009), p.3615-3620

Tokumoto, M.S., Pulcinelli, S.H., Santilli, C.V. and Briois, V. Catalysis and Temperature Dependence on the Formation of ZnO Nanoparticles and of Zinc Acetate Derivatives Prepared by the Sol− Gel Route. The Journal of Physical Chemistry B, 107(2) , (2003), p.568-574.

Niederberger, M.. Nonaqueous sol–gel routes to metal oxide nanoparticles. Accounts of chemical research, 40(9) ,(2007) , p.793-800.

Briois, V., Ch Giorgetti, F. Baudelet, S. Blanchandin, M. S. Tokumoto, Sandra Helena Pulcinelli, and Celso Valentim Santilli. "Dynamical study of ZnO nanocrystal and Zn-HDS layered basic zinc acetate formation from sol− gel route." The Journal of Physical Chemistry C 111, no. 8 (2007), p. 3253-3258.

Barreto, G., G. Morales, A. Cañizo, and N. Eyler. "Microwave Assisted Synthesis of ZnO Tridimensional Nanostructures." Procedia Materials Science 8 (2015), p. 535-540.

Wojnarowicz, Jacek, Roman Mukhovskyi, Elzbieta Pietrzykowska, Sylwia Kusnieruk, Jan Mizeracki, and Witold Lojkowski. "Microwave solvothermal synthesis and characterization of manganese-doped ZnO nanoparticles." Beilstein Journal of Nanotechnology 7, no. 1 (2016), p.721-732.

Kumar, Sanjay. "Synthesis of ZnO nanostructures by microwave irradiation using albumen as a template." Journal of Nanoparticles 2013.

XianweiMeng. Solvothermal Synthesis of ZnO Nanoparticles and Anti-Infection Application in Vivo ACS Appl. Mater. Interfaces, 2015, 7 (2), p. 1308–1317

Rai, Prabhakar, Woon-Ki Kwak, and Yeon-Tae Yu. "Solvothermal synthesis of ZnO nanostructures and their morphology-dependent gas-sensing properties." ACS applied materials & interfaces 5, no. 8 (2013), p. 3026-3032.

Tonto, Parawee, Okorn Mekasuwandumrong, Suphot Phatanasri, Varong Pavarajarn, and Piyasan Praserthdam. "Preparation of ZnO nanorod by solvothermal reaction of zinc acetate in various alcohols." Ceramics International 34, no. 1 (2008), p. 57-62.

Anand, Vikky, and Vimal Chandra Srivastava. "Zinc oxide nanoparticles synthesis by electrochemical method: Optimization of parameters for maximization of productivity and characterization." Journal of Alloys and Compounds 636 (2015), p. 288-292.

Agnieszka Kołodziejczak-Radzimska andTeofil Jesionowski, "Zinc Oxide—From Synthesis to Application: A Review" in Materials (2014), 7, p.2833-2881.

Lv, Wei, Bo Wei, Lingling Xu, Yan Zhao, Hong Gao, and Jia Liu. "Photocatalytic properties of hierarchical ZnO flowers synthesized by a sucrose-assisted hydrothermal method." Applied Surface Science 259 (2012), p. 557-561.

Jamal, Raied K., Mohammed A. Hameed, and Kadhim A. Adem. "Optical properties of nanostructured ZnO prepared by a pulsed laser deposition technique." Materials Letters 132 (2014), p.31-33.

Omri, K., I. Najeh, R. Dhahri, J. El Ghoul, and L. El Mir. "Effects of temperature on the optical and electrical properties of ZnO nanoparticles synthesized by sol–gel method." Microelectronic Engineering 128 (2014), p.53-58.

Kathirvelu S, D'souza L, Dhurai B. UV protection finishing of textiles using ZnO nanoparticles. Indian Journal of Fibre & Textile Research.34(9), (2009), p.267-73.

Xie Y, He Y, Irwin PL, Jin T, Shi X. Antibacterial activity and mechanism of action of zinc oxide nanoparticles against Campylobacter jejuni. Applied and environmental microbiology.77(7):23, (2011), p. 25-31.

Kuo, Chun-Hong, Tian-Fu Chiang, Lih-Juann Chen, and Michael H. Huang. "Synthesis of highly faceted pentagonal-and hexagonal-shaped gold nanoparticles with controlled sizes by sodium dodecyl sulfate." Langmuir 20, no. 18 (2004): 7820-7824.

Lu PJ, Huang SC, Chen YP, Chiueh LC, Shih DY. Analysis of titanium dioxide and zinc oxide nanoparticles in cosmetics. Journal of food and drug analysis.;23(3), (2015) p.587-94.

Zhang, Zhihua, Meihua Lu, Hairuo Xu, and Wee□Shong Chin."Shape□Controlled Synthesis of Zinc Oxide: A Simple Method for the Preparation of Metal Oxide Nanocrystals in Non□aqueous Medium." Chemistry-a European Journal 13, no. 2 (2007): 632-638.

Talam S, Karumuri SR, Gunnam N. Synthesis, characterization, and spectroscopic properties of ZnO nanoparticles. ISRN Nanotechnology. (2012)

Hasnidawani JN, Azlina HN, Norita H, Bonnia NN, Ratim S, Ali ES. Synthesis of ZnO nanostructures using sol-gel method. Procedia Chemistry.;19: (2016), p. 211-6.