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The Influence of Calcination Temperature on the Formation of Nickel Oxide Nanoparticles by Sol-gel Method

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Abstract *- In this study, NiO nanoparticles were prepared by the sol-gel method, which is one of the simplest and lowest-cost techniques. The synthesis was accomplished by using Troton X100 as the surfactant and Ni(NO3)2·6H2O as the inorganic precursor. The influence of experimental factors, such as optimization of calcination temperatures for the synthesis of NiO nanoparticles, was investigated. To confirm the formation of Nickel oxide, the calcined samples were examined by FT-IR spectra. Further, the structural characteristics of the calcined samples at temperature 800^{0}c were examined by X-ray diffraction, scanning electron microscopy, and transmission electron microscopy. The end result places the dynamic routes as a simple, low-cost contestant to the assembly of nickel oxide nanoparticles.*

Keywords - *TritonX100, solgel, NiO nanoparticles, calcination temperature, and deionized water*.

I. INTRODUCTION

Nanoparticles play an important role in many engineering, industrial and biological fields; in the current circumstances, several catalysts, electronic devices, supercapacitors, and ceramics applications. As a significant improvement of novel processing, synthesis and characterization of the nanoparticles have become necessary for existing and forthcoming applications [1]. Among the different nanoparticles, Metal oxide nanoparticles are at the top of rapid progress in nanotechnology. This versatility of metal oxide nanoparticles and applications mainly arises from their distinct properties related to their bulk materials [2-6]. Now a day metal oxides can be mentioned as active catalysts in heterogeneous catalysis, sensors, biochemical engineering, material science, and environmental remediation. For catalytic purposes, Nickel oxide (NiO), one of the most common transition metal oxide, was used [7-11].

Measured synthesis of metal oxide nanoparticles is needed for effective application. The sol-gel method is a definite and wet chemical multistep process that involves hydrolysis, polymerization, gelation, aging, drying, and calcination. Metal oxides derived from this method have better homogeneity and phase purity. The probable morphology and size of nanomaterial may be attained by means of monitoring the PH, temperature, concentration ratio of precursors and precipitating agent and also using different surfactant agents [12-20]. Sintering at low temperatures is the main advantage of the sol-gel method compare to the high-temperature conventional process.

One of the leading focuses of studies in the area of nanoparticles to define precise calcination temperature [21-23]. Calcination temperature is one of the important parameters to accomplish specific particle morphologies. Calcination must be done gradually with low heating rates in order to take away the remaining moisture and the gases from the decomposition of the organic and inorganic additives without detrimental of the end product.

In this work, we report a systematic investigation of calcination temperature for the synthesis of NiO nanoparticles. Further study Morphology and structure of synthesized material. The sol-gel method was used to synthesize nickel oxide nanoparticles from an aqueous solution containing only nickel nitrate, sodium hydroxide, Triton X100, and deionized water. This technique is a simple and cheap method that can produce pure metal oxide nanomaterial.

II. EXPERIMENTAL

Materials

Nickel nitrate ($Ni(NO_3)_2.6H_2O$, Sigma-Aldrich 99.99%), sodium hydroxide (NaOH, Merck 99%) and Triton X100 Merck 99%)

Preparation of nanocrystalline NiO

The NiO nanoparticles were synthesized by the sol-gel method using Nickel nitrate $Ni(NO_3)_2.6H_2O$ (1M) used as a precursor, and Triton X100 as capping agent was dissolved in 150 ml deionized water. The precipitating agent NaOH (1M) was slowly dropped under vigorous stirring. The green precipitated $Ni(OH)_2$ obtained was treated in a Teflon-lined autoclave at 120°C for the period of 24 hours. The subsequent gel was separated via filtration and repeatedly

washed by deionized water. Afterward, the washed precipitate was dried at 90 ℃ for 24 h. Finally, the precipitate was calcined at 450°C, 700°C, and 800°C for 3h.

III. Results and Discussion

Characterization

Fig.1 shows IR spectra of products forms at different calcination temperatures. IR spectrum of sample calcined at temperature 450°C shows the various stretching frequencies, intense peak at 3406.29 cm−1 corresponds to the O–H chemical bond signifying the existence of hydroxyl groups, another intense peak around 1633 cm−1 which can be attributed to H-bonding and the interaction of hydroxyl groups and broad peaks at 547.78 and 451.34 cm−1 indicates initiation of Ni-O bond formation. IR spectrum of sample calcined at temperature 700°C shows the various stretching frequencies at 3394.72, 1570.06, 1612.49, and 505.35 cm−1 indicates stretching vibration mode of the chemically bonded hydroxyl group as well as Ni-O stretching mode, respectively. Samples calcined at 450 and 700°C show frequencies from 2796.78 to 2873.94 cm−1 due to C–H stretching and anti-stretching vibration frequencies of Triton X100. IR spectrum of sample calcined at temperature 800°C shows the various stretching frequencies at 1473.62, 1041.56, and 501.49 cm−1. The stretching frequency, particularly at 501.49 cm−1, confirms the formation of NiO.

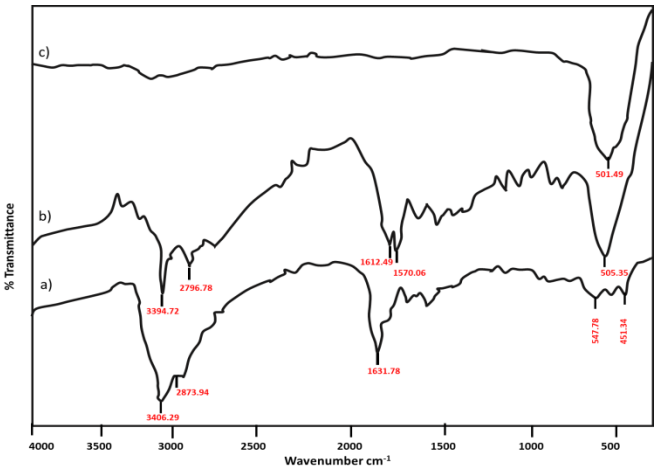

Fig 1: FT-IR spectra of NiO nanoparticles calcined at a) 450^{0}c b) 700^{0}c and c) 800^{0}c

XRD pattern of NiO sample calcined at 800^{0}C reveals diffraction peaks (Fig.2) with 2Θ value of 37.26, 43.24, 62.93, 76.34, and 79.20 originate from the crystal planes (111), (200), (220), (311), and (222) of NiO. All the reflections of the XRD pattern can be referring to the standard pattern of the pure cubic phase of NiO (JCPDS No.47-1049). This reveals that the NiO sample synthesized by the sol-gel method produced a face-centered cubic structure.

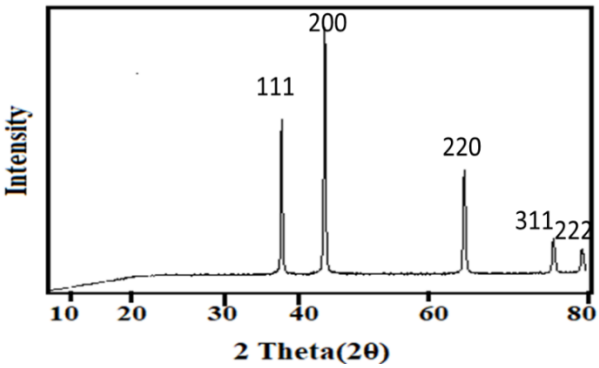

Fig 2: XRD pattern of NiO nanoparticles calcined at 800^{0}c

Morphology and elemental composition study of synthesized NiO were analyzed through SEM and EDS techniques, respectively(Fig. 3a and 3b). SEM analysis reveals nanoparticles are cubic-shaped with good uniformity and crystallinity. EDS confirms the elements of synthesized NiO.

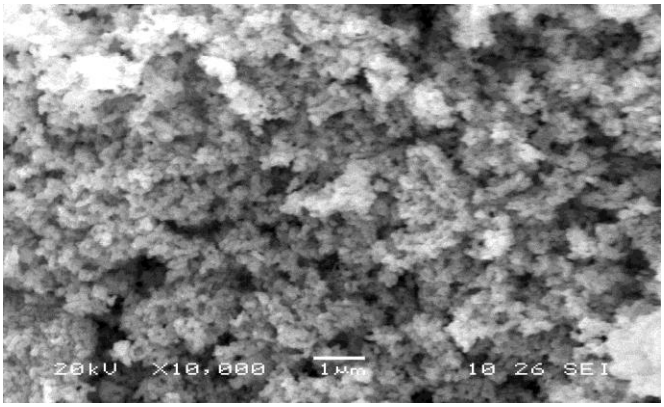

Fig 3a: SEM image of NiO nanoparticles calcined at 800^{0}c

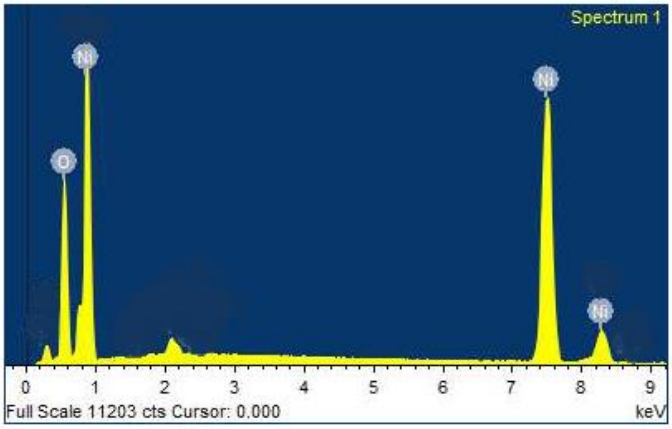

Fig 3b : EDS spectrum of NiO nanoparticles calcined at 800^{0}c

Particle size and morphology of synthesized NiO were analysed through TEM image and SAED pattern. TEM Analysis shows that the particles are cubic with average particle size 20nm. SAED pattern reveals that product obtained has pure crystalline phase (Fig. 4a and 4b).Fig.4c shows particle size distribution of NiO nanoparticles.

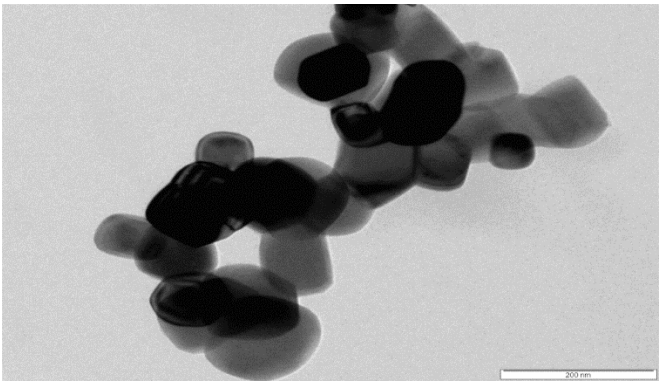

Fig 4a: TEM image of NiO nanoparticles calcined at 800^0c

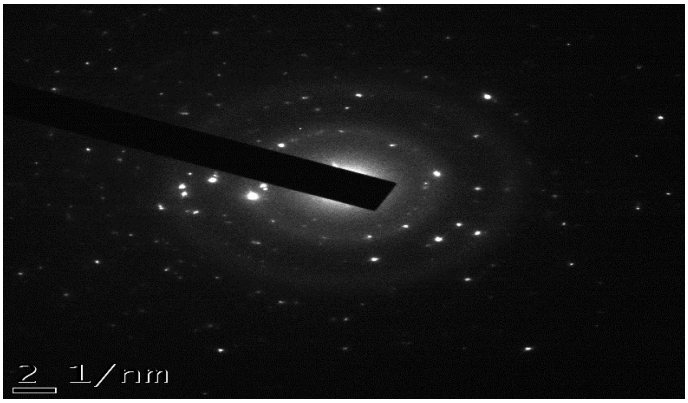

Fig 4b: SAED pattern of NiO nanoparticles calcined at 800^0c

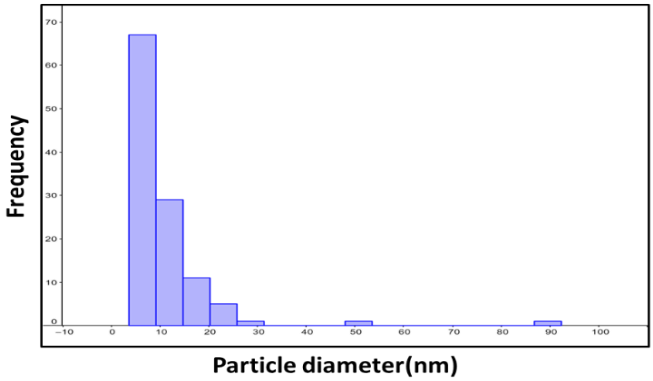

Fig 4c: Particle size distribution of NiO nanoparticles calcined at 800^0c

VI. CONCLUSIONS

NiO nanoparticles have been efficiently synthesized from nickel nitrate as the inorganic metal precursor and Triton X100 as surfactant using the sol-gel method. The product was characterized by FTIR, XRD, TEM, SEM, and EDS techniques. Pure cubic NiO nanoparticles were obtained at 800^0C calcination temperature. This further confirms sol-gel synthesis method was efficient for particle homogeneity. Overall this investigation clearly indicates the structural features and development of NiO nanoparticles were prominently influenced by calcination temperature.

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REFERENCES

[1] G. Oskam, Metal oxide nanoparticles: synthesis, characterization and application, J Sol-Gel Sci Techn. 37(3) (2006) 161–164.
[2] H.Gleiter, Foreword, Nanostruct. Mater. 6(3)(1995) 5-6.
[3] M.Valden, X.Lai, and D.W.Goodman, Onset of catalytic activity of gold clusters on titania with the appearance of nonmetallic properties, Sci. 281(5383)(1998) 1647- 1650.
[4] J.A.Rodriguez, G.Liu, T.Jirsak, Hrbek, Z.Chang, J.Dvorak and A.Maiti, Activation of gold on titania: adsorption and reaction of SO(2) on Au/TiO(2)(110), J.Am. Chem. Soc. 124(18)(2002) 5247-5250.
[5] M.Baumer and H.Freund, Metal deposits on well-ordered oxide films, J. Progress in Surf. Sci., 61(8)(1999) 127-198.
[6] M.L.Trudeau and Y.Ying, Nanocrystalline materials in catalysis and electrocatalysis: Structure tailoring and surface reactivity, J.Nanostruct. Mater. 7(2)(1996) 245-258.
[7] S. Farhadi and S. Panahandehjoo, Spinel-type zinc aluminate ($ZnAl_2O_4$) nanoparticles prepared by the co-precipitation method: A novel, green and recyclable heterogeneous catalyst for the acetylation of amines, alcohols and phenols under solvent-free conditions, Appl. Catal. A: Gen., 382(2)(2010) 293-302.
[8] S. Deki, H. Yanagimoto, S. Hiraoka, K. Akamatsu and K. Gotoh, NH_2-Terminated poly (ethylene oxide) containing nanosized NiO particles: synthesis, characterization and structural considerations, Chem. Mater. 15(26)(2003) 4916-4922.
[9] Y.Wang, C.Ma, X.Sun and H.Li, Preparation of nanocrystalline metal oxide powders with the surfactant-mediated method, Inorg. Chem. Commun. 5(10)(2002) 751-755.
[10] X.Deng and Z. Chen, Preparation of nano-NiO by ammonia precipitation and reaction in solution and competitive balance, Mater. Lett. 58(4)(2004) 276-280.
[11] A.Dierstein, H. Natter, F. Meyer, H.O. Stephan, C. Kropf, and R. Hempelmann, Electrochemical deposition under oxidizing conditions (EDOC): a new synthesis for nanocrystalline metal oxides, Scripta Mater. 44(9)(2001) 2209-2212.
[12] I W. Sutapa, A. W. Wahab, P. Taba and N. L. Nafie, Synthesis and structural profile analysis of the MgO nanoparticles produced through the sol-gel method followed by an annealing process, Orient. J. Chem. 34(2) (2018) 1016-1025.
[13] L.L.Hench and J.K.West, The sol-gel process, Chem. Rev. 90(1) (1990) 33-72. ,
[14] L.J.Fu.H.Liu, C.Li, Y.P. Wu, E.Rahm and R.Holze. H.Q.Wu, Electrode materials for secondary lithium batteries prepared by sol-gel methods, Prog. Mater. Sci. 50(7)(2005) 881-928.
[15] H.Liu, Y.P.Wu, E.Rahm, R.Holze and H.Q.Wu, Cathode Materials for lithium-ion batteries prepared by sol-gel methods, J. Solid State Electrochem. 8(7) (2004) 450-466.

[16] L.Predoana, A.Barau, M.Zaharescu, H.Vassilchina, N. Velinova, B. Banov and A. Mornchilov, Electrochemical properties of the $LiCoO_2$ powder obtained by sol-gel method, J. Eur. Ceram. Soc. 27(2-3)(2007) 1137-1142.

[17] Q. Chena, J. Wang, Z. Tang, W. Hea, H. Shao, and J. Zhang, Electrochemical performance of the carbon-coated Li3V2 (PO4)3 cathode material synthesized by a sol-gel method, Electrochim. Acta. 52(16)(2007) 5251–5257.

[18] D.H.Chen and X.R.He, Synthesis of nickel ferrite nanoparticles by sol-gel method, Mater. Res. Bull. 36(7) (2001) 1369-1377.

[19] B.J.Hwang, R.Santhanam, and D.G.Liu, Characterization of nanoparticles of $LiMn_2O_4$ synthesized by citric acid sol-gel method, J.Power Sources 97(2001) 443-446.

[20] L. G.Teoh and K.D.Li, Synthesis and characterization of NiO nanoparticles by sol-gel method, Mater. Trans. 53(12)(2012) 2135-2140.

[21] N. M. Al-Hada, E. Saion, A. H. Shaari, M. A. Kamarudin and S. A. Gene, The influence of calcination temperature on the formation of zinc oxide nanoparticles by thermal-treatment, Appl. Mech. Mater., 446(2014) 181-184

[22] R. B. da Silva, R. A. Pinto, J. M. Soares, A. Franco Jr., M. A. Correa, F. Bohn, and J. A. P. da Costa, Effect of the synthesis method and calcination temperature on the formation of Ni–NiO nanocomposites, J. Solgel Sci. Technol. 291(2)(2019) 1-9.

[23] S. Satapathy, A. Ahlawat, A. Paliwal, R. Singh, M. K. Singh, P. K. Gupta, Effect of calcination temperature on nanoparticle morphology and its consequence on optical properties of Nd: Y_2O_3 transparent ceramics Cryst. Eng. Comm. 16(13)(2014) 2723-2731.