RESEARCH ARTICLE OPEN ACCESS

Synthesis of Nio Nanoparticles by Diffusion Flame Reactor

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ABSTRACT
Nanoparticles of NiO are synthesized by diffusion flame reactor using Nickel nitrate hexahydrate [$Ni(NO_3)_2.6H_2O$] as a precursor, dissolved in acetone. LPG and Oxygen is used as Fuel and oxidant in these experiments. Flowrate of 0.2slpm (Standard Liter per minute) of LPG, 0.7slpm of Oxygen and 10slpm of Nitrogen as career gas is used in flame reactor. The morphology and crystalline phase of the synthesized nickel oxide nanocrystals have been investigated by scanning electron microscopy (SEM), transition electron microscopy (TEM) and X-ray diffraction (XRD). The average particle diameter of the NiO particle is 40-50nm.
Keywords: Flame synthesis, Nano metal oxide, LPG, Oxygen, Diffusion flame

I. INTRODUCTION

Nano - metaloxide particles of Transition metals are gaining their importance in various fields such as catalysis, ceramic materials and electronic components [1]. Due to the nano scale range of the particles they exhibit novel material properties that are significantly different from those of their bulk counterparts. Nano-Nickel Oxide (NiO) is a very prosperous material widely used in catalysis [2], magnetic materials [3] battery cathodes [4] and gas sensors [5].The two mainstream methods to synthesize nano-particles are wet methods (sol-gel) and aerosol methods such as flame synthesis. Several researchers have prepared NiO nano particles by various methods sol-gel [6], thermal decomposition [7], surfactant-medicated synthesis [8], solvothermal method [9] and Flame sprayPyrolysis [10] .Wet methods offer fine control over particle size and particle size distributions. However in wet- process the final product often includes a mixture of amorphous and crystalline particles, resulting in costly and time consuming in post process steps. Flame aerosol methods can be operated continuously allowing for greater output with minimal post processing. These methods produce fewer byproducts, usually making them more economically viable then wet methods [11, 12].Gas phase combustion synthesis of inorganic particles is used routinely today to make a variety of commodities like SiO_2, TiO_2, Al_2O_3, NiO, MgO and ZnO etc.Y.Hou and H. kondoh [13] synthesized Nickel nanoparticles by chemical reduction method and reported hexadecylamine (HDA) as surfactant, is an effective approach to obtain smaller monodispersive metal nanoparticles. N.Dharmaraj and his group [14] synthesized Cubic nickel NiO nanoparticles with uniform size 40-50nm by dispersion method followed by heat treatment. Dae Jong Seo et al [10] synthesized ZnO, MgO and NiO nanoparticles of 30nm diameter range using nitrate salt and acetate salt precursors by spray Pyrolysis with Diffusion flame using propane-oxygen flame and reported flame spray pyrolysis of aqueous droplets is more versatile compared to conventional spray pyrolysis.

In this present paper, flame synthesis method is described to synthesize NiO nano particles using LPG-oxygen as fuel and oxidant, with nitrogen as carrier gas.

II. EXPERIMENT

2.1. Materials

The Following chemicals are commercially available and were used without pretreatment: Ni $(NO_3)_2.6H_2O$ (Finar Reagents, local made) as precursor, acetone (Finar Reagents) as solvent, Liquefied Petroleum gas (LPG) as Fuel (Praveena gas Agency, Hindustan petroleum), Oxygen (seven Hills Supplier, India) as oxidant.

2.2 Methodology

The flame reactor has been indigenously designed to produce nano materials. The detailed setup of the reactor has been discussed in our previous paper to synthesize carbon nano materials [15]. In this work only slight modifications have been done to inject the precursor solution as shown in fig-1 flow chat diagram of our reactor set up for synthesis of nano metal particles. The reactor operated under atmospheric pressure. The measured quantity of the LPG and the oxidant reaches the ignition chamber to ignite flame once the flame is ignited the flowrate of the fuel and oxidant has been changed to required flowrate. During the process we have observed the dark orange flame color which is perfectly in a spindle form. Along the entire length of the flame, its

temperature was recorded using a K-type thermocouple.

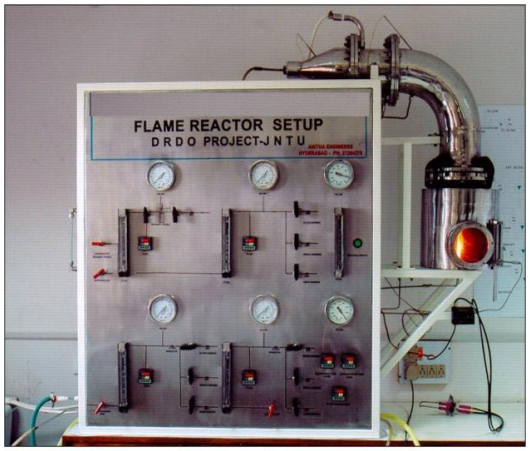

Figure 1 – Flame Reactor setup, with flow meters front view (Rotameters).

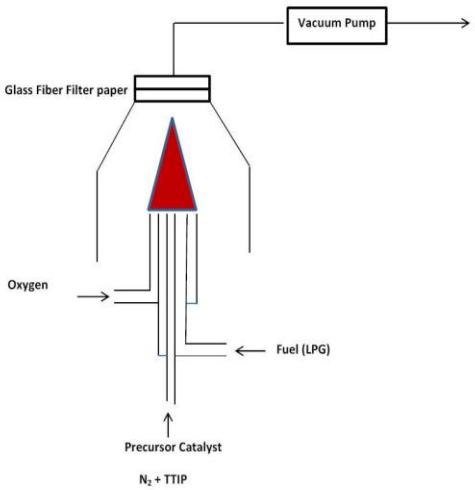

Figure 2 – Flow chart diagram of flame reactor unit

In this study, nano NiO particles were synthesized in diffusion flame by using Nickel nitrate hexahydrate [Ni $(NO_3)_2$ $6H_2O$] as precursor, Acetone as solvent. In this Experiment Flame is ignited in the burner with LPG and Oxygen, Nitrogen gas is bubbled through a Reagent vessel containing liquid precursor to deliver the precursor vapour to the burner, as shown in Fig-2.In order to prevent any condensation of the precursor, all gas line downstream the precursor reagent vessel and the burner were wrapped with heating tapes to maintain them at 60^oC.The NiO produced is captured on a glass fiber filter (Axiva GF/A) as shown in Fig-3, is scrapped carefully and weighed. Later heat treated at 350 ^OC in the presence of air for 60 minutes to remove any traces of amorphous carbon impurities then the samples were characterized by SEM, XRD, TEM and FT-IR to know the morphology and structure of the NiO.

Figure 3 – Sample collector with glass fiber filter paper holder

III. RESULTS AND DISCUSSION

XRD 3.1

The XRD (7000 Schimazu) analysis was carried out using Cu Kα1 type of radiation with a wavelength (λ) of 1.54060 A^o. XRD graph of NiO nano particles synthesized using LPG-Oxygen at flow rates of 0.2Slpm and 0.7 Slpm is shown in Fig-4.The step size was 0.02 degree/step and step time was 0.2 sec/step. The working range was 2ɵ = 20-80, the average crystal size NiO is calculated according the equation (1) scherer's equation [16] by selecting particle peak.

Where L is the length of the crystal in the direction of the d spacing, K is the constant of 0.9,λ is the wavelength of X-Ray, β is the full width at half- maximum (FWHM) of the selected peak and Ɵ is the Bragg's angle of diffraction peak.

$$L = \frac{K\lambda}{\beta Cos\theta} \quad \text{.....(1)}$$

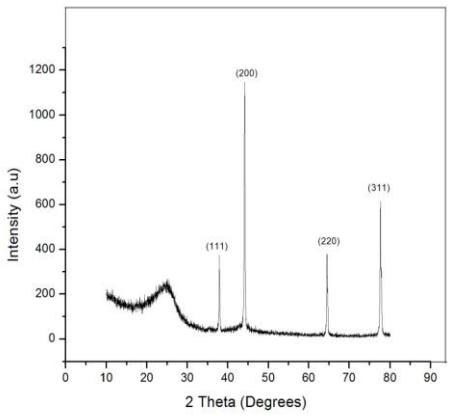

Figure 4 – XRD Pattern of

Flame synthesized NiO

The existence of strong and sharp diffraction peaks (Fig-4) at 2ɵ values 37.90, 44.14, 64.48 and 77.82^{o}Corresponding to (111) (200) (220) and (222) crystal planes indicated the formation of phase pure, cubic nickel oxide (bunsenite, NaCl type crystal) at flowrate of 0.2slpm of LPG and 0.7slpm of oxygen. The particle size of the synthesized nickel oxide calculated from XRD using the Debye-Sherrer equation is about 44.3 nm, in good agreement with the observed from SEM and TEM images. The nature and positions of the above diffraction peaks are characteristic of cubic nickel oxide phase as mentioned in the other reports [9, 17, and 18] and ICDD PDF-2 release 2003 file number 89-7131.

3.2 Scanning electron microscope (SEM) image

Fig. 5 shows the SEM image of NiO nanoparticles synthesized by Diffusion flame reactor usingNickel hexahydrate as precursor salt. From this picture it is clearly seen that NiO particles have uniform size and well dispersion in the bulk state with diameter around 45-55 nm. This indicates that the NiO nanoparticles synthesized with LPG as fuel and oxygen as oxidant is in uniform size and good agreement with XRD calculation of particle diameter with SEM image.

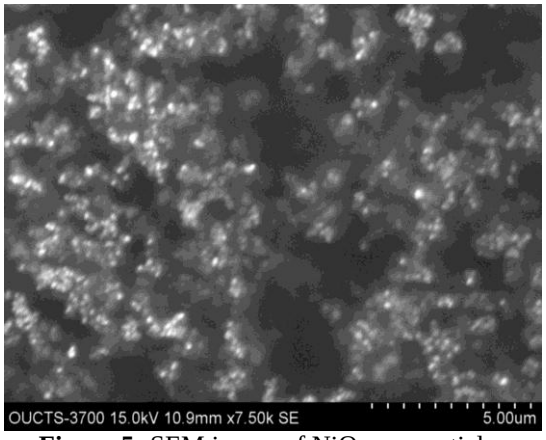

Figure 5- SEM image of NiO nanoparticles

3.3 Transmission electron microscopy (TEM)

TEM image Fig-.6 has been recorded using a copper grid dipped in a solution containing NiO nanoparticles dispersed in methanol by ultrasonifation. TEM images of NiO nanoparticles synthesized with Diffusion flame reactor reveal the presence of large number of NiO particles with uniform size diameter about 45-55nm and well dispersed in bulk material.

3.4 FT-IR

The FT-IR spectra of as synthesized NiO are shown in Fig-7. The absorption band centered

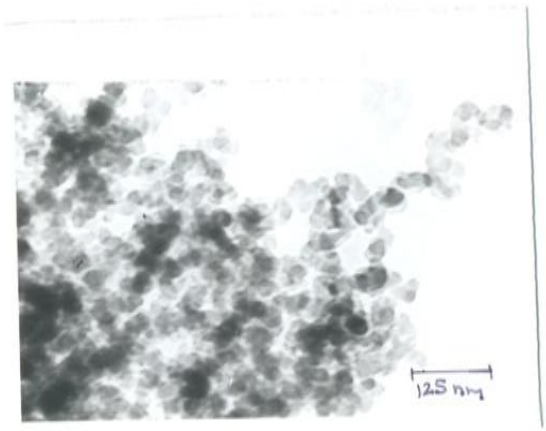

Figure 6- TEM image of NiO nanoparticles

at 3200 cm^{-1} was attributed to the O-H bond stretching vibrations and the band near 1400 cm-1 was assigned to H-O-H bending vibrations this is due to some moisture content in the sample. Furthermore the strong absorption band around 420 cm^{-1} was assigned to Ni-O stretching vibration. Indicating the presence of NiO nano particles in the flame synthesized compound.

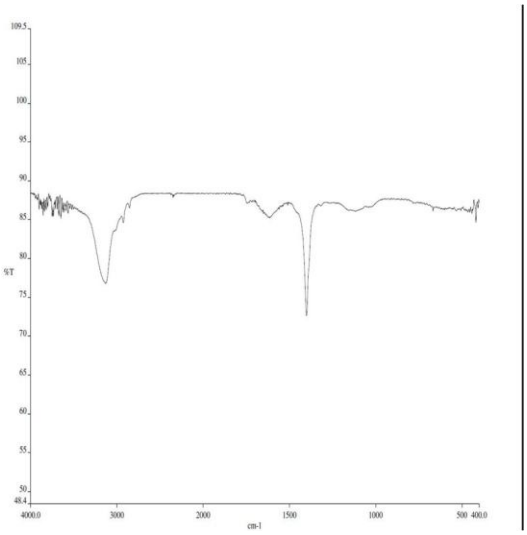

Figure 7- FT-IR Spectra of NiO nanoparticles

IV. CONCLUSION

In these present work NiO nanoparticles has been synthesized with diffusion flame reactor using LPG as fuel, oxygen as oxidant and Nitrogen as carrier gas with flowrates of 0.2, 0.7 and 10 slpm respectively. Nickel nitrate hexahydrate and Acetone is used as precursor and solvent in this work. Thus the synthesized NiO particle is characterized by SEM, TEM, XRD and FT-IR to know its characteristics. Hence concluded with is, the synthesized NiO particles is cubic in structure and uniform in size with 40-50 nm of average particle diameter.

ACKNOWLEDGEMENTS

The author acknowledge the financial support given by Mallareddy Institute of science and technology, which this work is carried out; I thank Jawarlalnehru Technological University Hyderabad for providing Facilities and National Centre for Compositional Characterization of Materials (NCCCM), Hyderabad for providing SEM data of all samples.

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