International Journal of ChemTech Research
CODEN (USA): IJCRGG, ISSN: 0974-4290, ISSN(Online):2455-9555
Vol.10 No.5, pp 145-149, 2017

Synthesis and Characterization of Nickel Oxide Nano Particles

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Abstract : Nano particles are gaining importance in recent industrial applications. These nano particles are prepared by two major approaches top down and bottom up. Sol-gel method is one of the important method to prepare nano particles bottom up approach. In the present work, nano sized Nickel Oxide(NiO) powder was synthesized via sol-gel method using Nickel Oxide, as the precursors. The surface area and shape of the particles were characterized by a Scanning Electron Microscope (SEM).The characterized particles are dispersed in water to prepare nano fluid and the viscosity of this fluid is estimated by redwood viscometer; whereas thermal conductivity and specific heat are estimated using available models.
Key Words : Nano-particles; Nickel Oxide; Sol-Gel Method; Nano-fluid.

1. Introduction:

Nanoparticles (NP) are gaining prominent importance in recent industrial applications are defined as particles whose diameter is less than 100 nm (1nm = 10^{-9}m). These nanostructure materials may be single phase or multiphase polycrystalline solids with a typical average size of a few nanometers[1]. Though the name is 'Nano' it is not only limited to tiny science but often also employed in bulk materials and large surfaces. Nano Technology deals with these nano particles, mainly production and manipulation of these nano ranged materials[2]. Because of different physical and chemical properties like lower melting points, higher specific surface areas, specific optical properties, mechanical strengths, and specific magnetizations,nanometals are attractive in various industrial applications.These particles have great potential uses in the electronic, chemical and mechanical industries, as well as in the related technologies using catalysts, drug carriers, sensors, pigments, also as well as in magnetic and electronic materials.

Nano-materials are synthesized by two major approaches Top down and Bottom Up, large objects are modified to give nanoparticles in top down approach where as small building blocks are produced and assembled into larger structures in the later approach. High energy ball millingis the major technique in top-down approach for nanoparticle synthesis[3]. Low surface area, highly poly-disperse size distributions, and partially amorphous state of the as prepared powders are few disadvantages of milling.Inert gas condensation, solvo-thermal reaction[2], sol-get technique/process are the major methods in the bottom up synthesis approach.

A nanofluid is a dilute suspension of nanometer-size particles and fibers dispersed in a liquid. As a result, when compared to the base fluid, changes in physical properties of such mixtures occur, e.g., viscosity, density, and thermal conductivity. In the present work nano-sized nickel oxide powder is prepared by sol-gel method and the prepared particles are characterized by SEM, the physical properties of nano fluids viscosity, thermal conductivity and specific heat are calculated.

2. Materials and Methods:

2.1. Materials and Equipment:

All the Chemicals such as Nickel Oxide, Nitric Acid, Citric Acid, were of pure and analytical grade and were used without further purification. Citric acid was used as Chelating agent as it is experimentally proved to be simple and cost effective. Ethylene glycol was used as gelating agent for the formation of gel. Muffle furnace is used for sintering the powder, Scanning Electron Microscopy is used to characterize the nano powder, Sonicator is used to prepare nano fluids by dispersing the nano powder in the water. Redwood viscometer is used to estimate the viscosity of the fluids.

2.2. Synthesis of Nickel Oxide nanoparticles:

Nickel oxide nano powder was prepared by sol-gel process. The desired amount of nickel oxide was dissolved in nitric acid so as to form Nickel nitrate. This mixture is then subjected to continuous heating and stirring. As the continuous heating proceeds citric acid is added in the ratio of 1:0.66 moles. Further heating is carried out with constant stirring until the sol gets thick. At this stage ethylene glycol (approximately 3 times to the moles of NiO) is added. With the addition of ethylene glycol gel auto-ignition starts and completely burns. Figure-1 gives the picture of gel formation of nickel oxide sol, where as Figure -2 gives the powder ready for sintering.

Figure-1: Gel formation of nickel oxide sol

Figure-2: Nano powder ready for sintering

After complete combustion, the burnt powder is collected and subjected to sintering at three different temperatures. The whole sample was first sintered at 500°C and one-third part of the sample was weighed and taken aside from it. The remaining part is then sintered at 600°C after that half of the part is taken back; remaining part is further sintered at 700°C.

2.3. Characterization with Scanning Electron Microscopy (SEM):

The synthesized nano powders of nickel oxide were characterized to know the size and morphology of the prepared samples. The SEM generates a beam of incident electrons in an electron column above the sample chamber. The energy of the incident electrons can be as low as 100 eV or as high as 30 keV depending on the

evaluation objectives. The electrons are focused into a small beam by a series of electromagnetic lenses in the SEM column. Scanning coils near the end of the column direct and position the focused beam onto the sample surface. Scanning electron imaging was taken with a resolution of 3700 and a width of 13 mm on a 5µm scale.

2.4. Dispersion of Synthesized Nano powders:

The nano powders that were synthesized and characterized were now subjected for dispersion in order to get nano fluids with nano particles dispersed completely. For the dispersion the base fluid taken was water of about 500ml and samples of NiO added to the water. These samples were stirred continuously for 30 minutes before moving forward to sonication. After the completion of stirring they were kept in sonicator for another 30 minutes so that complete dispersion can be possible under the ultrasonic waves that results from the sonicator.

2.5. Property Estimation of Nano fluids:

Estimation of Viscosity:

In order to compare the viscosity of the nano fluids with base fluids the nano fluid's viscosity was calculated using the Red wood viscometer. The cup of the viscometer was first cleaned and filled with the base fluid and then after with nano fluid. A 50 ml volumetric flask was placed below to collect the fluid. Now the ball rod was taken out and stop watch was on. Time taken to collect50ml of sample was noted. Two readings were taken randomly for all the fluids and the viscosity was calculated based on the formula

$$At - \frac{B}{t}$$

Where A and B are the constants and t is time taken to collect the sample.

Estimation of Thermal Conductivity:

Increase in thermal conductivity depends on nanoparticle material, size and concentration[4]. The specific heat of the nano fluids were calculated using classic models that were suggested by Maxwell. Maxwell's formula shows that the effective thermal conductivity of nano fluids relies on the thermal conductivity of the spherical particle, the base fluid and the volume fraction of the solid particles.

Estimation of Specific heat:

The accurate determination of specific heat is very important in evaluating the thermal performance of nano fluids. It is one of the key parameters for describing the nano fluids and the convective flow status[5]. Estimation of specific can help us in different studies of the heat flow and also different thermo physical properties.A correlation for nano fluids was presented by Pak and Cho, taking the idea from the liquid-particle mixture theory, subsequently Xuan and Roetzel[6] modified this correlation by assuming thermal equilibrium between the nano scale solid particles and the liquid phase by including the density.

3. Results and Discussion:

Nano powders of NiO were prepared by using Sol-gel method of Bottom-up technique and sintered at 500°C, 600°C, 700°C respectively. Repeated sintering showed an influence on the distribution of the nano particles which was observed in the SEM characterization.

3.1. Characterization of nano particles:

Size and distribution of the samples repeated sintered at 700°C and 600°C were similar when compared to T. Shanmugavel et.al,[7]. The results indicate that mono-dispersive and highly crystalline NiO nanoparticles are obtained. The particles were of spherical and rod shape. Figure -3 gives the SEM image of the sample sintered at 700°C. The particles were spherical, and the surface area is ranging from 0.194 μm^2 to 0.555 μm^2.

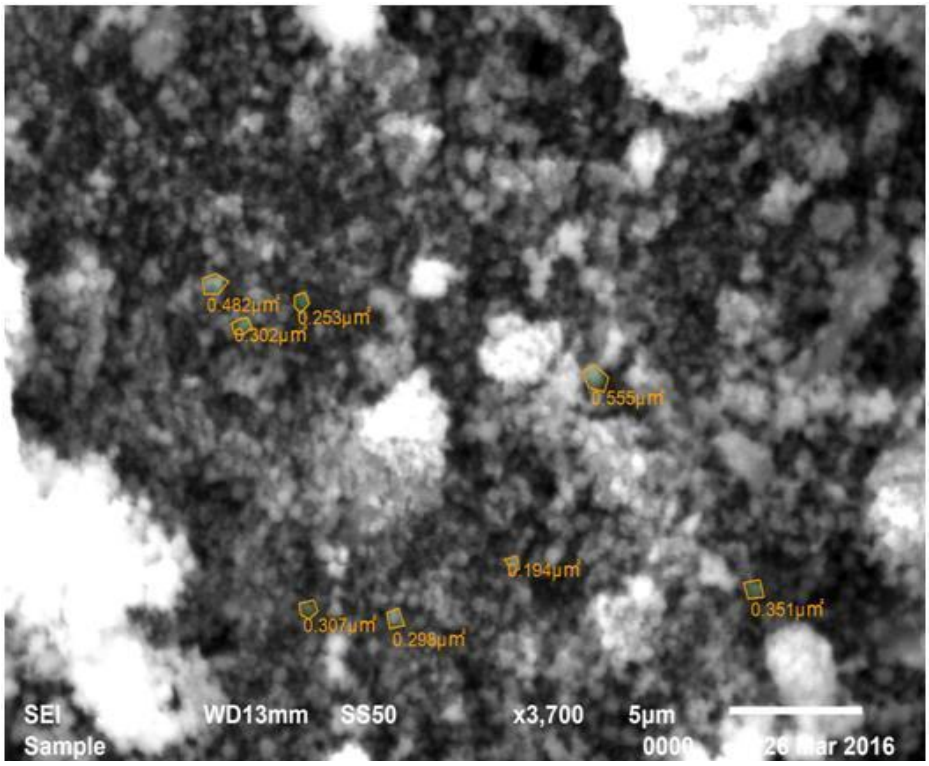

Figure-3: SEM image for the NiOsample sintered at 700°c

Figure – 4 is the SEM image of the NiO sample sintered at 600°C. in this case also the particles are spherical and the surface area is in the range of 0.253 μm^2 to 0.302 μm^2.

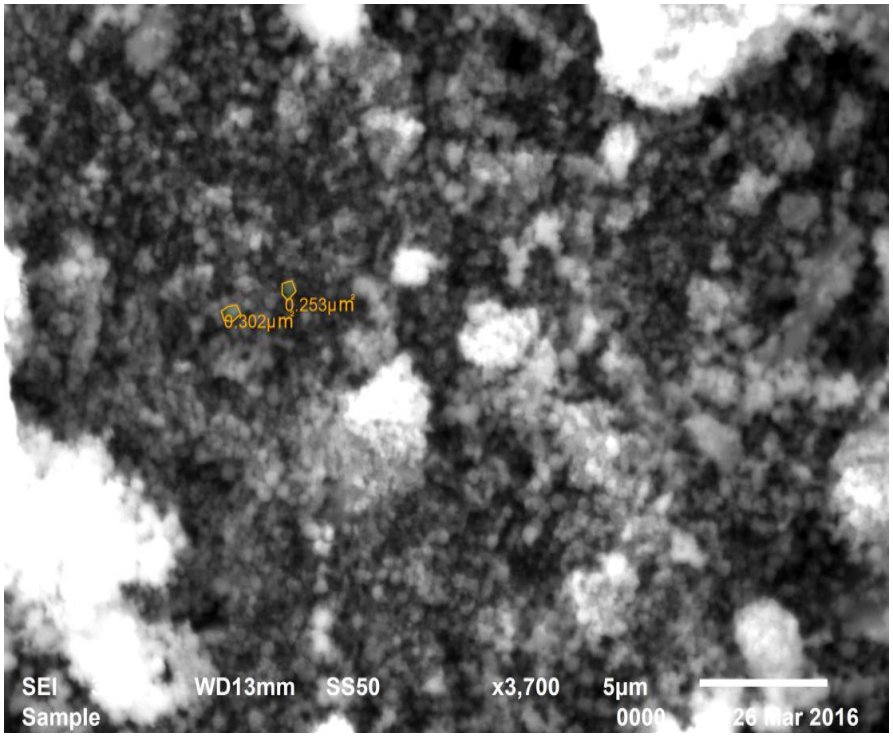

Figure-4: SEM image for the NiOsample sintered at 600°c

In both cases clusters were formed and observed due to the presence of moisture and also NiO has the tendency to form cluster due to its high surface area energy.

3.2. Properties of nanofluids:

Viscosity of NiO :

Viscosity of the base fluid was found to be 0.765cp at 30°C. For NiO it was increased to 8.0136cp at the same temperature. The increase in viscosity of the nano fluid makes it as a better choice than the base fluid as it supports in the improvement of heat transfer coefficient[8].

Thermal Conductivity of NiO :

When compared to the other metal oxide nano fluids nickel oxide nano fluid resulted in a low thermal conductivity. It decreased up to 0.023 times of the thermal conductivity of the base fluid. Thermal conductivity of base fluid water was 615.4 whereas that of NiO was calculated as 615.231.

Specific Heat of NiO :

Calculated value of the specific heat of the NiO fluid was 736.4188 J/ kg k. The decrease in the value should be noticed with reference to the base fluid which was observed with the Calculations performed according to the Xuan and Roetzel's model.

4. Conclusion:

NiOnano particles were prepared by following Sol-gel process of bottom up method. The characterized SEM results obtained showed that pure crystalline particles of spherical and rod shape were formed with some clusters and agglomerates. NiO sample's surface area was between the range of 0.1µm² to 0.5µm². The viscosity of the nano fluid prepared by dispersion of nano particles is increased but the thermal conductivity and specific heat of the fluid is low compared to base fluid i.e. water.

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