

Plant Based Reducing Agent for Nickel Nano Particles

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ABSTRACT: Nickel nano particles (NNPs) can be synthesized by physical, chemical, biological methods. The reduction of nickel nitrate salt to pure nickel nanoparticles using naturally occurring *Azadirachta indica* (neem) leaves extract as a reducing agent. In this work qualitative and economical NNPs are synthesized by using Green method. In aqueous solution of Nickel nitrate (1M) *Azadirachta indica* leaves extract is slowly added and ultra sonicated for 1Hr at 80°C. The solution is then directly heated and dried to get Nickel oxide powder. As obtained nickel oxide was then reduced to NNPs at different range temperature (700- 900°C) in hydrogen atmosphere. The effect of varying temperature for getting different quality and quantity of NNPs were studied. These NNPs was then characterized by (XRD) X-ray diffraction, SEM (Scanning Electron Microscope) and FTIR (Fourier Transform Infra-Red Spectroscopy). It was found that the quality and quantity of NNPs varies with different experimental conditions.

Keywords: Nickel nano particles; Green method; Ultra sonicated; *Azadirachta indica* and X-ray diffraction.

INTRODUCTION: Nanotechnology is the science at nanoscale objects which are in nanometer size i.e. 10^9 meter. The science which deals with the synthesis, characterization and development of various nanomaterials. In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties [1]. Different types of nanomaterials are being produced using different types of approaches i.e. top to down and bottom up[2]. These types of different nanoparticles are synthesized using different methods i.e. chemical methods, physical methods and biological methods.

Preparing nickel nitrate from nickel oxide in presence of inert gas, reduces it to metallic nickel. Growing of nickel nanoparticles using different bio organisms have been also reported [3]

Nickel oxide can be synthesized by different methods. Upon heating above 400°C, nickel powder reacts with oxygen to give NiO. In some commercial processes, nickel oxide is made by heating a mixture of nickel powder and water at 1000°C[4].

Synthesizing metallic nanoparticles using naturally occurring plant based reducing agent i.e. *Azadirachta indica* (neem) leaves extract. This type of synthesis is an emerging field because plant part acts as reducing agent as well as capping agent and free from toxic chemicals [5]. Some authors also reported the func-

tionization of nickel nano particles [6]. In the presented study, we applied green method for the synthesis of Nickel oxide nanoparticles. Green method is comparatively simpler, cost-effective synthesis method. Further the Nickel oxide nanoparticles were reduced to Nickel nanoparticles under the influence of hydrogen gas. The synthesized Nickel oxide nanoparticles and Nickel nanoparticles were characterized by key analytical techniques viz. Fourier Transform Infra-red Spectrometer (FTIR); X-ray diffraction technique (XRD); BET Surface area analyser (SAA) and Scanning Electron Microscopy (SEM).

MATERIALS AND METHODS:

Synthesis of Nickel Oxide Nanoparticles: Weighed and transferred about 20 g of nickel nitrate in glass beaker and added about 50 ml deionized water into it. To this aqueous solution of Nickel nitrate (1M) *Azadirachta indica* (neem) leaves extract is slowly added and ultra sonicated for 1Hr at 80°C. The solution is then directly heated and dried to get Nickel oxide powder.

Synthesis of Nickel Nanoparticles: Approximate quantity of powdered nickel oxide nanoparticles as obtained was taken into a quartz boat. The Lenton's Split Tube Furnace equipped with one meter long quartz tube was used for this synthesis. The hydrogen

gas was used in this experiment to maintain the inert atmosphere for the reduction of nickel oxide to nickel nanoparticle. Both end of quartz tube was closely packed in order to avoid the interference of atmospheric conditions while synthesis of nickel nanoparticles under the influence of hydrogen gas. Quartz boat containing nickel oxide is slowly moved into the quartz tube at the centre of the furnace and both the ends of tube were firmly sealed again allowed to pass hydrogen gas slowly and started heating the furnace upto 700°C throughout the experiment. After achieving the desired temperature the furnace the was kept at that temperature for 1Hr. under hydrogen gas purging continuously. After completion of reduction under hydrogen atmosphere, the quartz boat was kept as it was into the quartz tube and allowed to cool to room-temperature under the influence of hydrogen gas. Samples were collected and stored into the glass bottles.

The same experiment was carried out for 800°C and 900°C temperature for getting different morphologies of nickel nanoparticles.

Characterizations: The Nickel nanoparticles as obtained in this experiment were characterized by FTIR, XRD, BET Surface Area analyzer and SEM. Following make of analytical instruments was used, FTIR Spectrometer with spectrum-one model from Perkin-Elmer using KBr palate method. X-ray diffraction (XRD) analysis was performed using Phillips analytical X-ray diffractometer with Cu K α radiation running at 45 KV/40 mA in the 2 θ range 2°–100° with step size of 0.02. Specific surface areas were measured using SmartSorb-92/93 model of Smart Instruments Co. Pvt Ltd by low temperature nitrogen adsorption using the Brunauer-Emmett-Teller (BET) single point method. The samples were degassed at 150°C for 2h prior to analysis. The SEM images were obtained from Phillips SEM 505 scanning electron microscope.

RESULTS AND DISCUSSION:

Nickel Oxide Nanoparticles: FTIR spectrum of Nickel oxide nanoparticle was recorded. The broad band at 3513cm⁻¹ has been attributed to O-H stretching vibrations. The band at 1630cm⁻¹ was attributed to the bending vibration of H-O-H bond. The broad absorption band in the region 500 to 570 cm is assigned to Ni-O stretching vibration mode [7]. The broadness of the band indicates the Nano crystalline nature of the samples.

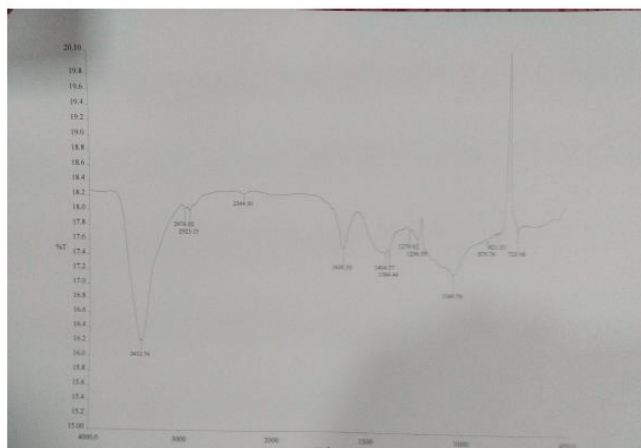


Figure 1: FTIR Spectrum of Nickel Oxide Nanoparticles.

The XRD pattern shows the crystalline pattern

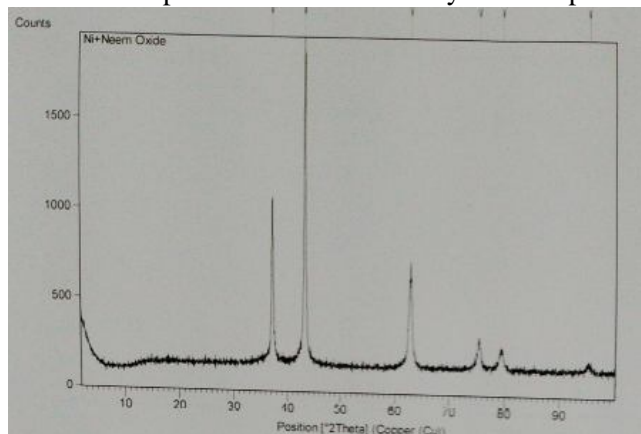


Figure 2: XRD Pattern of Nickel Oxide Nanoparticles.

The surface area of Nickel oxide nanoparticles was determined and surface area value was 1.45m²/g which indicates the formation of nanosized particles.

Nickel Nanoparticles: FTIR spectra of Nickel nanoparticles calcined at 700°C and 800°C and 900°C were recorded. The FTIR spectra of Nickel nanocatalysts collected at varying calcination temperatures (700°C, 800°C & 900°C) are comparable and indicates absence of characteristic bands which were present in FTIR spectrum of Nickel oxide nanoparticles. This confirms the changes at molecular level by means of loss of different oxides during calcination experiments. However there is an enhancement in the transmittance of bands obtained in the FTIR spectrum of Nickel catalyst calcined at 800°C in comparison with the catalyst obtained at 700°C. This affirms the development of pure Nickel catalyst at elevated temperature.

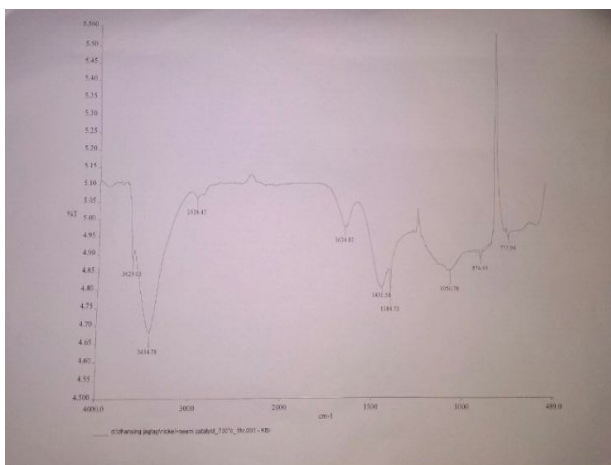


Figure 3: FTIR Spectra of Nickel Nano-particle calcined at 700°C.

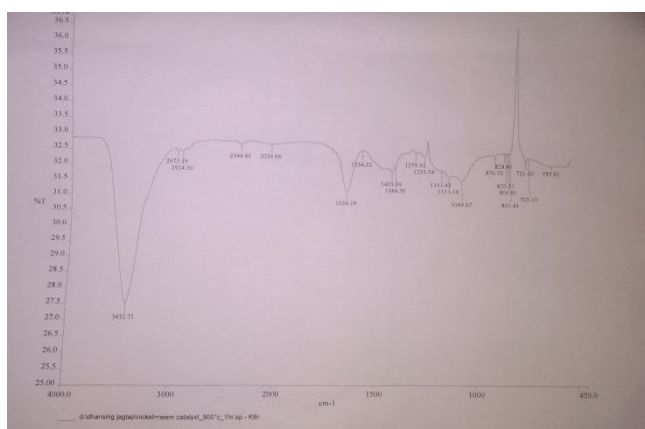


Figure 4: FTIR Spectra of Nickel Nano-particle calcined at 800°C

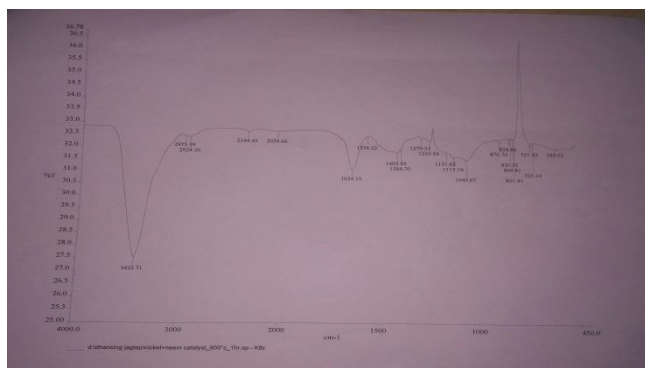


Figure 5: FTIR Spectra of Nickel Nano-particle calcined at 900°C.

The XRD patterns of Nickel oxide nanoparticles, NNP obtained at 700°C, 800°C and NNP obtained at 800°C showing the main diffraction peaks at 2-theta 37.28°, 43.34°, 62.94°, 75.42°, 79.58° and 95.18°. These 2-theta values are the characteristic of Nickel nanocatalysts. However, the intensity counts pertaining to 2-theta peaks were enhanced in the XRD pattern of nanocatalyst obtained at 900 °C compared with the in-

tensity counts of nanocatalyst obtained at 800°C & at 700°C respectively.

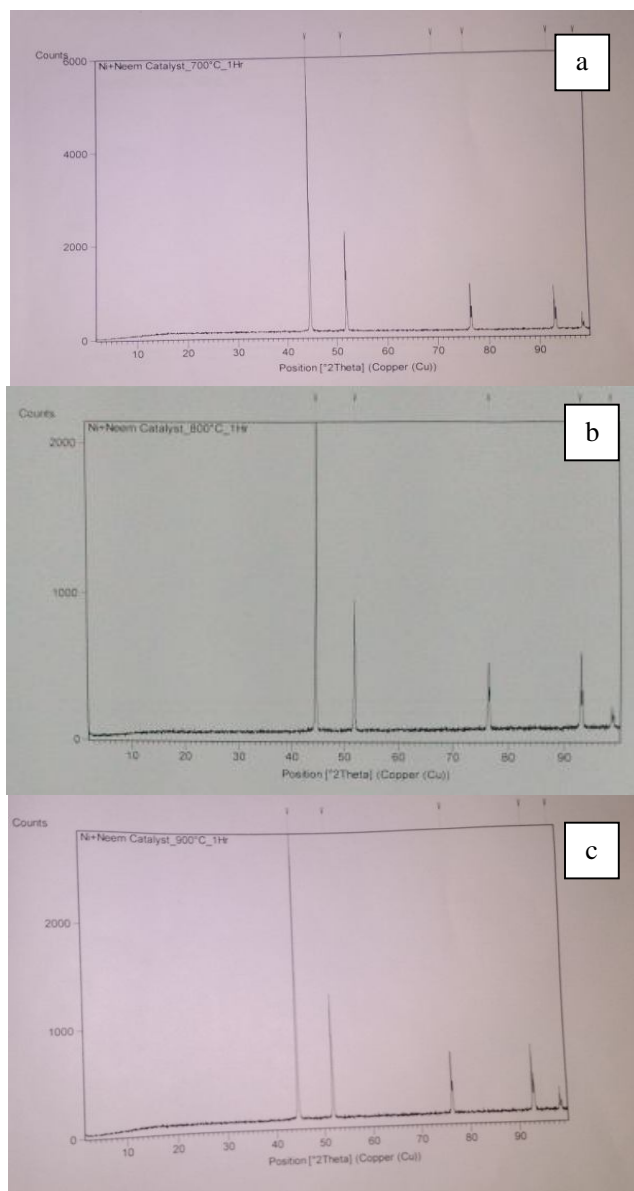


Figure 6: XRD Patterns of a) NNP calcined at 700°C b) NNP calcined at 800°C and c) NNP calcined at 900°C.

Table 1 presents Specific Surface area of Nickel nano particles obtained at 700°C, 800°C and 900°C temperatures respectively. The specific surface area of Nickel nanocatalysts were calculated as 5.75 m²/g for the catalyst obtained at 700°C whereas 7.05 m²/g for the catalyst obtained at 800°C and 9.4m²/g for the catalyst obtained at 900°C. The difference in the specific surface area at three temperatures clearly indicates the impact of calcination temperature on the surface area of Nickel nanocatalyst which is higher than that of Nickel oxide nanoparticles.

Table 1: BET Surface area comparison of three different nickel nanoparticles with that of nickel oxide nanoparticle.

Name	Nickel Oxide	NNP at 700°C	NNP at 800°C	NNP at 900°C
BET Surface Area (Sq.m/g)	1.45	5.75	7.05	9.04

SEM images of different Nickel nanoparticles obtained at different calcination temperature i.e at 800°C and 900°C were collected. It was concluded from SEM images that, the SEM image of Nickel catalyst obtained at 900°C are clearer than that of Nickel catalyst obtained at 800°C. The particle size of catalyst calcined at 800°C ranges between 21nm to 51nm and less uniform compared to that of the sample calcined at 900°C. Can be concluded that the elevated calcination temperature produces uniform and smaller particle size.

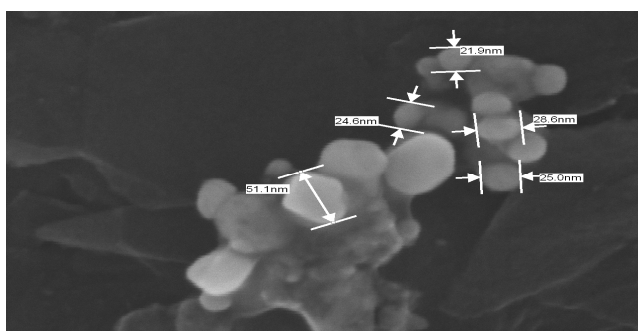


Figure 7: SEM Data of Nickel Nanoparticles obtained at 800°C.

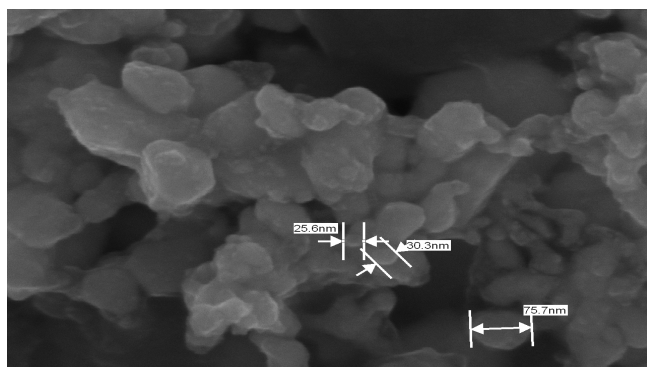


Figure 8: SEM Data of Nickel Nanoparticles obtained at 900°C.

CONCLUSION: Green method synthesis is practically simpler and cost-efficient method for the synthe-

sis of Nickel nanoparticles. The reduction of Nickel oxide nanoparticles to Nickel nanoparticles under the influence of hydrogen gas is an innovative approach. The FTIR bands of Nickel catalyst calcined at 900°C in comparison with catalyst obtained at 800°C confirms the purer Nickel catalyst at elevated temperature. The Surface area of Nickel nanoparticle obtained at 900°C is greater than surface area obtained at 800°C and 700°C, reveals the impact of increasing calcination temperatures on the crystallite size of Nickel nanoparticles.

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